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Precision Measurement and Calibration

Optics, Metrology, and Radiation

Handbook 77 — Volume III



United States Department of Commerce
National Bureau of Standards

Precision Measurement and Calibration

Selected Papers on Optics, Metrology, and Radiation

A compilation by Sherman F. Booth of
previously published technical papers
by the staff of the National Bureau of
Standards.

Issued in three volumes*

- I. Electricity and Electronics.
- II. Heat and Mechanics.
- III. Optics, Metrology, and Radiation.



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Abstract

This Handbook is a three-volume compilation of approximately 150 selected papers previously published by the staff of the National Bureau of Standards on precision measurement, calibration, and related subjects. It was prepared to meet the urgent need of newly established standards laboratories for a "textbook" and reference source in these fields. Volume I contains papers in electricity and electronics; Volume II, heat and mechanics; and Volume III, optics, metrology, and radiation. Each volume contains a complete index of the entire Handbook by author, subject, and title.

Foreword

The National Bureau of Standards is charged with the responsibility of establishing and maintaining the national standards of physical measurement, and of providing means for their effective utilization. This responsibility carries with it the mission of providing the central basis for a complete, consistent system of physical measurement, adequate for national growth in research and technology.

The recent tremendous increase in industrial activity, particularly in the missile and satellite fields, has led to an unprecedented demand for precision measurement, which, in turn, is bringing about the establishment of hundreds of new standards laboratories. Many of these new laboratories must cover the entire field of measurement, and must do so with a staff not previously trained in work on standards of precision measurement.

To aid these laboratories in transmitting the accuracies of the national standards to the shops of industry, the Bureau has prepared this three-volume Handbook. It is a compilation of publications by the Bureau staff that have been found of value to those who are establishing and operating new standards laboratories. Omitted are some extended works, as well as a few shorter papers that are otherwise readily available.

It is hoped that this compilation will serve both as a "textbook" and a reference source for the many scientists and engineers who must be trained in the shortest possible time to fill responsible positions in this critical area.

A. V. ASTIN, *Director.*

Preface

Because of the urgent need for this Handbook, it has been reproduced by a photoduplication process. As a result, the individual publications that make up the compilation will be found to vary in such details as style, size of type, and method of pagination.

Each paper reproduced for the compilation is essentially complete as originally published and retains its original page numbering. All pages have also been numbered in regular sequence throughout the three volumes. Thus, the volume page number and the original page number are combined, for example 100/10.

Because of the short time available, a complete review of each paper included was not possible. However, some efforts were made to bring the older papers up to date, and only those papers have been included that are of current value. Nevertheless, users should be cautioned that the state of the art may have advanced beyond that represented in some of the older papers.

These three volumes, extensive as they are, include only a fraction of the published work of the National Bureau of Standards relating to standards. However, many of the reprinted papers contain extensive bibliographies that will enable the user who is confronted with a special problem to locate additional information.

The papers that appear or are cited in this three-volume Handbook were originally published over a period of several years as circulars, research papers, chapters of books, and as articles in scientific and technical periodicals. Thus individual copies of many papers are no longer readily available. More recent Bureau publications, and in some cases the older papers for which prices are given, may still be obtainable by purchase from the Superintendent of Documents, U.S. Government Printing Office, Washington 25, D.C. Other papers may often be obtained directly from the authors or from the publishers of the Journals in which the papers appeared. The papers referred to in the various lists, if not generally available as stated above, are usually available for reference in technical, university, Government depository, and public libraries.

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Selected Papers on
Optics and Metrology

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SOME FUNDAMENTALS OF MODERN DIMENSIONAL METROLOGY

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ABSTRACT

Progress in technology now requires that for some purposes the ultimate in precision and accuracy be very closely approached in the measurement of primary dimensional standards that are defined by material surfaces. A research and development program at NBS is aimed toward careful evaluation or measurement of many factors that are sources of error and the devising of means and methods for eliminating, or at least reducing, sources of error wherever possible. Some principles are reviewed, and progress toward complying with them is reported.

Units of measurement are discussed in relation to increase in accuracy, particularly the proposed redefinition of the meter in terms of light wavelengths and the recent redefinition of the inch in terms of the meter. Other subjects treated are: progress toward increased stability of materials of standards, the relation of both macro- and micro-geometry to accuracy, control and accurate measurement of temperature, methods of attaining high amplification, improvements in interferometry, and possible future developments. It is shown that knowledge gained in these respects has practical application not only in measurements to highest degrees of accuracy but also in facilitating commercial measurements.

Presented at the twenty-eighth annual meeting of the American Society of Tool and Manufacturing Engineers in Detroit, Michigan, April 21-28, 1960. All papers presented at such meetings are the property of the ASTM. Permission must be secured in writing from the Society to reprint or publish this paper

INTRODUCTION

This paper discusses some of the latest developments in attaining extremely high precision and accuracy in the measurement of lengths and the dimensions of master gages. The demands for high reliability in functioning of modern mechanisms of many types require control of two main factors: (1) the uniformity and stability in the composition and structure of materials, and (2) uniformity of dimensions of components. The control of dimensions of mechanical parts has reached such a state that we at the top of the pyramid of dimensional standards must seek the ultimate in precision and accuracy as a practical engineering matter relative to dimensional standards that are defined by surfaces.

There always has been concern for highest accuracy in length measurements, primarily as a matter of scientific accomplishment, but also as related to line standards of length such as tapes and scales used in civil engineering, and for establishing highly accurate values for the wavelengths of light of certain spectrum lines. The Bureau of Standards' custody of the national standards in many fields of technology makes it necessary for NBS to provide calibration services in order to make such standards available to the public. Naturally most of this work is conducted at the highest practical levels of accuracy, as otherwise the transfer of the units represented in the standards from the national standard to the working levels in industry would be seriously impaired. Moreover, it is incumbent on the Bureau to strive to keep a step ahead of the industrial public's actual requirements for accuracy so that such requirements may be adequately fulfilled. This means that there must be continuing programs of research and development relative to measuring methods and equipment as well as in the maintenance of standards.

The Engineering Metrology Section is the

branch of the Bureau that specializes in measurements of dimensions that are defined by surfaces, such as master gages and end standards other than gage blocks, which are calibrated by the Length Section. In its program, more especially in recent years, the emphasis has been on careful evaluation of those factors which are sources of error in measurement and on devising ways and means to eliminate or at least reduce sources of error wherever possible. An important phase of this program has been popularly known as the research program on measurement to the ten-millionth inch.³

We had known in a general way that certain factors were affecting the accuracy of our measurements. And we had applied corrections for them to the extent that our knowledge of them permitted, in the full realization that we were not attaining the ultimate in precision and accuracy in routine calibrations, but realizing at the same time that there was no particular need for such ultimate refinement. In recent years such a need has developed, and it has arisen from the activities of practical engineers. Accordingly, this paper presents, not merely information of an interesting and academic nature, but a general review of recently attained knowledge that has been gleaned by laborious effort largely within the Bureau and also by some other investigators. The field is too broad to cover fully within the scope of this paper, but the material presented is applicable by metrologists not only in achieving the ultimate in accuracy but frequently in solving the everyday problems of measurement in the numerous metrology laboratories that are springing up throughout the country.

The paper deals with some phases of units of measurement, dimensional standards, both macro- and micro-geometry, in relation to design of standards and to measurement, methods of amplification, and control and measurement of environmental factors. It reviews some of the principles relating to achieving the ultimate in accuracy and some of the more recent developments in equipment and methods for implementing those principles.

REDEFINITIONS OF LENGTH UNITS

In this discussion it is appropriate to start with fundamental units of length, particularly since one is shortly to be redefined and the other has recently been redefined. The unit on which all of our length measurement is based is the

meter. It is to be redefined in terms of wavelengths of light. Certain spectrum lines have in the past been considered for use as length standards, notably the red line of cadmium, the green line of the mercury isotope 198, and certain lines of krypton isotopes. For example, nine different determinations of the wavelength of the red cadmium line were made in the interval from 1892 to 1940. The average difference of these determinations¹⁷ from their mean is 0.14 parts per million, and they exhibit no trend with time, thus indicating the consistency of the procedures employed for relating the wavelength of the cadmium red line to the meter as defined by the International Prototype Meter.

Once a value is assigned to a wavelength, it can be used as a standard of length, when applied with suitably accurate equipment, to a higher degree of accuracy than any material standard, especially for lengths shorter than one meter. This situation led to the adoption in 1927 of the wavelength of the red cadmium line as a provisional international standard of length by the International Conference on Weights and Measures. This action had the further effect of leading toward a redefinition of our fundamental unit of length in terms of a light wavelength instead of in terms of a material standard, namely the distance between the centers of two ruled lines on a platinum-iridium bar that is in the custody of the International Bureau of Weights and Measures at Sevres, France.

It is expected that the International Conference on Weights and Measures at its meeting in 1960 will define the meter as 1,650,763.73 times the wavelength in vacuo of the orange-red radiation, corresponding to the transition between the energy levels $2p_{10}$ and $5d_5$ of the krypton 86 atom. This redefinition will not change the length of the meter in the least, but it is done with the intention of defining it more accurately than it can be defined by two ruled lines on a metal-alloy bar. Another important reason for this is that such a material standard cannot be expected to remain unchanged for all time or forever survive all possible accidents and catastrophes. Various national standards laboratories, including NBS, are developing equipment and techniques for calibrating material standards in terms of this

¹⁷Superior numbers refer to References at end of paper.

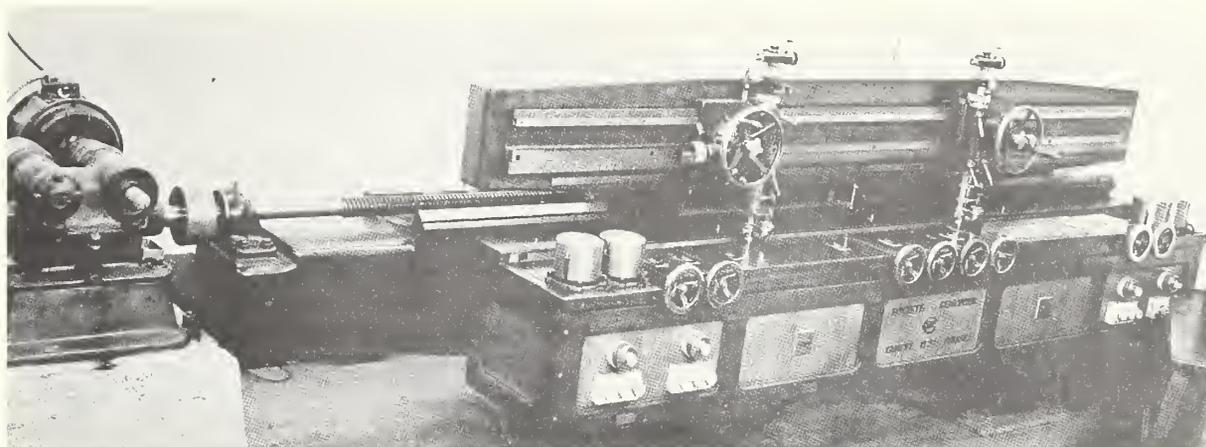


Fig. 1—Longitudinal comparator for line standards.

wavelength standard. There is inherent in the new procedure the possibility of upgrading the accuracy of lengths shorter than a meter to approximately 1 part in 10 million.

At present comparisons of line standards are made at NBS by means of a longitudinal comparator shown in Fig. 1. In a series of inter-comparisons in 1952-53 of 9 meter bars among themselves and with Prototype Meter 27, the primary standard of length of the United States, the values obtained are believed to be not in error by more than 0.2μ (8 microinches) and in most cases not in error by more than 0.1μ (4 microinches); that is, within 1 to 2 parts in 10,000,000.⁹

In selecting the orange-red line of Kr_{86} as being one of the most suitable lines for the primary length standard, a great many factors related to atomic properties were taken into account. Most of these are enumerated in Engelhard's paper.² Looking toward future improvements in wavelength standards, the NBS has already developed even better wavelength standards using atomic beam techniques.

The other important redefinition is an event that has already happened, namely the redefinition of the yard and correspondingly the inch

effective on July 1, 1959. In science and technology worldwide agreement has been achieved by this redefinition of these units, and the discrepancy between our values and those of the United Kingdom of nearly 4 parts in a million has been obliterated. The present inch is 2 microinches shorter than the one previously used in the U. S. and about $1\frac{3}{4}$ microinches longer than the earlier British inch. The change has practical effect only when measurements to six significant digits are involved; that is, for tool engineers, normally only when measurements are to microinches. Tabulated values of the change in value from the old to the new inch for standard lengths of gage blocks are shown in Table I with a diagram illustrating the different inch units in Fig. 2.

Anyone having a report showing the lengths of gage blocks expressed in U. S. inches may convert to lengths in international inches by *adding* amounts shown in Table I to the deviation from nominal length given in the original report.

Conversely, anyone desiring to convert the lengths of gage blocks expressed in international inches to lengths in U. S. inches may do so by subtracting the amount shown from the values in the report.

The manufacturers most affected by the change are those of gage blocks and other highly accurate length standards, measuring machines, jig borers, and jig grinders.¹⁴ The transition has proceeded very smoothly and the change has been hailed as very desirable because now we have only one inch or yard unit. Domestically these are now known simply as "the inch" and "the yard."

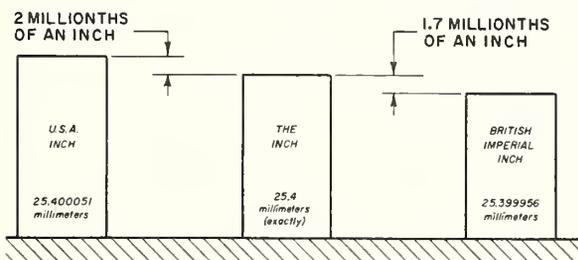


Fig. 2—Length changes resulting from the redefinition of the inch.

Table 1

Nominal Length of Gage Block	Change in Microinches
0.010 to 0.250 inch inclusive	none
0.300 to 0.750 inch inclusive	1
0.800 to 1.000 inch inclusive	2
2.000 inch	4
3.000 inch	6
4.000 inch	8
5.000 inch	10
6.000 inch	12
7.000 inch	14
8.000 inch	16
10.000 inch	20
12.000 inch	24
16.000 inch	32
20.000 inch	40

MATERIALS FOR CONTACT LENGTH STANDARDS

In the case of the meter bar, the choice of an alloy of 90 percent platinum and 10 percent iridium has proved to have been an excellent one for primary length standards from the standpoint of stability. However such an alloy is not economically available for widespread use in length standards. Practically all contact standards are made of steel or one of the hard carbides. The Bureau is engaged in an extensive investigation with the object of achieving the production of gage blocks of dimensional stability, surface quality, and other physical properties necessary to permit reliability of measurements to from 0.1 to 0.2 microinch per inch. The program has thus far been successful in developing three groups of gage blocks, each with a different material or treatment, that have met the requirements and have shown a dimensional stability of 0.2 microinch/in./yr. A voluminous progress report of this investigation is available.⁷ In this report optimum hardening and stabilization treatments developed for 52100 steel are described as follows:

“With hardened steels, the difficulty of completely balancing the dimensional changes occurring from structural transformation against residual stresses can be minimized by keeping these factors at low levels. As a starting point, it was decided to eliminate almost all of the retained austenite, which would not only reduce the possibility of growth but would possibly give a higher hardness and allow a higher tempering temperature or longer time at temperature to obtain a final hardness of 65 Rc. The higher tempering temperature was con-

sidered beneficial in the reduction of residual stresses. The standard procedure used for hardening by direct quench was to austenitize at 1550°F for 15 minutes and quench to room temperature in a quenching oil having an accelerated cooling rate.”

“The residual stresses resulting from thermal gradients on cooling from the hardening temperature are often reduced by martempering. The martempering treatment adopted consisted of austenitizing in a chloride bath at 1550°F for 15 minutes, quenching in a sodium nitrate-nitride bath at 300°F for 30 seconds, and then quenching to room temperature in oil”

“Both of the above hardening treatments were followed by a stabilization treatment designed to remove retained austenite, temper the martensite, and reduce residual stresses. This treatment for blocks hardened to 65 Rc consisted of an immediate refrigeration at -140°F for an overnight period, and a final temper at 250°F for nine hours. The interval between steps was kept as short as possible.”

The most stable of the nitrided 410 stainless steel gage blocks are blocks in the annealed condition and nitrided by the conventional two-stage process, and whose nongaging surfaces were not ground or otherwise machined after nitriding. It was not possible to observe the behavior of these blocks during the first year after heat-treatment. However, thereafter the overall change in length computed on the basis of one year is a consistent +0.2 microinch/in./yr.

Another set of annealed 410 stainless steel gage blocks was nitrided by the conventional single-stage process. Also, the white layer on the nongaging surfaces was ground off so as to leave a hard, bright, nitrided case. The stability of these blocks ranged from +0.1 to +0.3 microinch/in./yr. with an average of +0.2.

ACCURACY OF STANDARDS RELATED TO GEOMETRY

A contact standard is a solid body of which its geometry is one of its most important attributes. Webster’s Dictionary defines “geometry” as “that branch of mathematics which investigates the relations, properties, and measurement of solids, surfaces, lines, and angles.” For the purposes of this discussion it is convenient to develop principles relating to macro-geometry and others relating to micro-geometry.

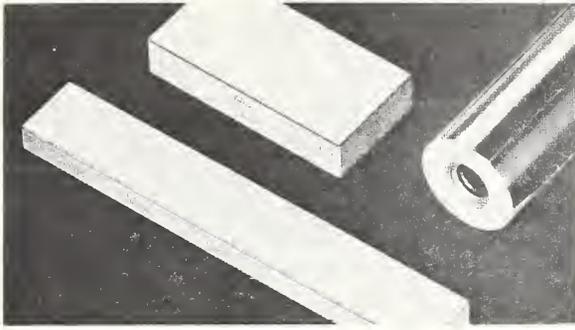


Fig. 3—Earlier and improved (top) angle gage block designs, and improved combination length bar (right).

Macro-Geometry

The macro-geometry or design of standards usually has an important bearing on the accuracy to which they may be made and used. The principles to be applied in optimum design of a standard for a given purpose will probably be evident to an experienced designer who gives the matter some thoughtful consideration, but they are not always obvious. I am not prepared to set forth a complete set of applicable principles, if indeed it is possible to compile such a set. A very important principle is that the design of a standard should generally be such that elastic distortion under the measuring loads normally applied is kept to a minimum. This can be illustrated by two recent examples of improvements in design that have produced greater accuracy by minimizing distortion.

The first example is that of two angle gage blocks, the first or older design at the left in Fig. 3, having long, relatively narrow wringing surfaces that are supported by an insufficient amount of metal. In an angle block the deviation from planeness of the wringing surface has an important effect on the accuracy of the block as an angle standard. If over a 2-in. length the midpoint is one microinch higher or lower than the ends of the gaging surface and the surface is curved, the variation in angle from end to end is 0.8 second. At the right of the figure is shown the more modern design of shorter and wider wringing surfaces and more adequate supporting metal. Needless to say, by the use of such blocks a higher degree of accuracy is attainable.

The second example is the design of a thread in the end of a combination length bar. To build up a standard of a desired length, bars of suitable lengths are selected from an assortment and screwed together with a torque in

the range from 10 to 20 lb.-in., using connecting studs. It was found that the design of this threaded connection⁸ has an appreciable effect on the reduction in length resulting from the torque applied. In Fig. 4 the older design of connection is shown at the left and the improved design at the right. The latter has short lengths of engagement and chamfers in the end of the threaded hole. The design is so proportioned that the decrease in length caused by the axial compressive force balances the increase in length caused by the radial bursting force and the moment associated with the axial compressive force. It was found by L. W. Nickols of the National Physical Laboratory that, using connecting studs of uniform diameter, the average reduction in length per threaded connection of the earlier type was about 6 microinches at 10 lb.-in. and 13 microinches at 20 lb.-in. torque. With the improved design the change in length per joint amounted to about 1 microinch and was independent of the assembly torque. This improved design permits substituting threaded connections for simply wrung connections in assemblies requiring highest accuracy, with much greater convenience in handling.

The force of gravity affects distortion. A familiar example is the need to support a bar standard, when measured horizontally, at the Airy points, in order that the ends of the standard may remain parallel to each other and the flexure of the bar held to a minimum. The distance between the Airy points is 0.577 times the length of the bar.

Another such example is that if a gage block is measured vertically and supported at its lower end it is shorter than when supported horizontally. The amount of shortening is proportional to the square of the length so that for long blocks this factor becomes significant. The applicable formula is

$$\delta = \frac{wL^2}{2AE}$$

where

δ = amount of shortening

w = weight per unit of length of the block

L = total length of block

A = cross-sectional area

E = Young's modulus of elasticity

For a steel block

$$\delta = 4.55 L^2 \times 10^{-9} \text{ in.}$$

and

$$\delta = 1 \text{ microinch when } L = 14.85 \text{ in.}$$

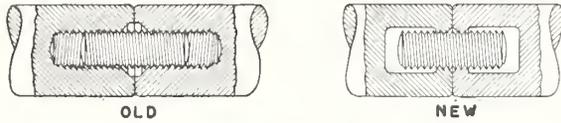


Fig. 4—Earlier and improved designs of threaded connections of combination length bars.

A distortion factor that is often overlooked is the penetration of a measuring contact point into the surface of a standard and a test piece. If these are of different materials it is necessary to evaluate the difference between the penetrations and apply it as a correction to the measurement. The chart presented in Fig. 5 is a quick means for determining such amounts. The chart also shows some equivalent compressive loads. It is necessary to be certain that the compressive strengths of the materials involved are not exceeded.

Another principle relating to macro-geometry of standards is the principle of averaging by

design to attain a single, highly accurate measurement in the place of taking several measurements of a given dimension and then averaging the results. The following are some examples:

1. About the year 1840 Sir Joseph Whitworth produced plane surface plates by grinding a group of three plates, pairing them off successively during the process.

2. Approximately 40 years ago the principle of averaging was applied and is still used in the Hoke process of mechanical lapping of precision gage blocks to produce a group of blocks of uniform length. The averaging is accomplished by systematic interchange of short and long blocks in their positions between the laps. Major Hoke also applied this principle in the lapping of gears to produce uniformity of tooth spacing and thickness.

3. A gage indexing fixture, Fig. 6a and 6b, similar to a rotary table, in which the indexing

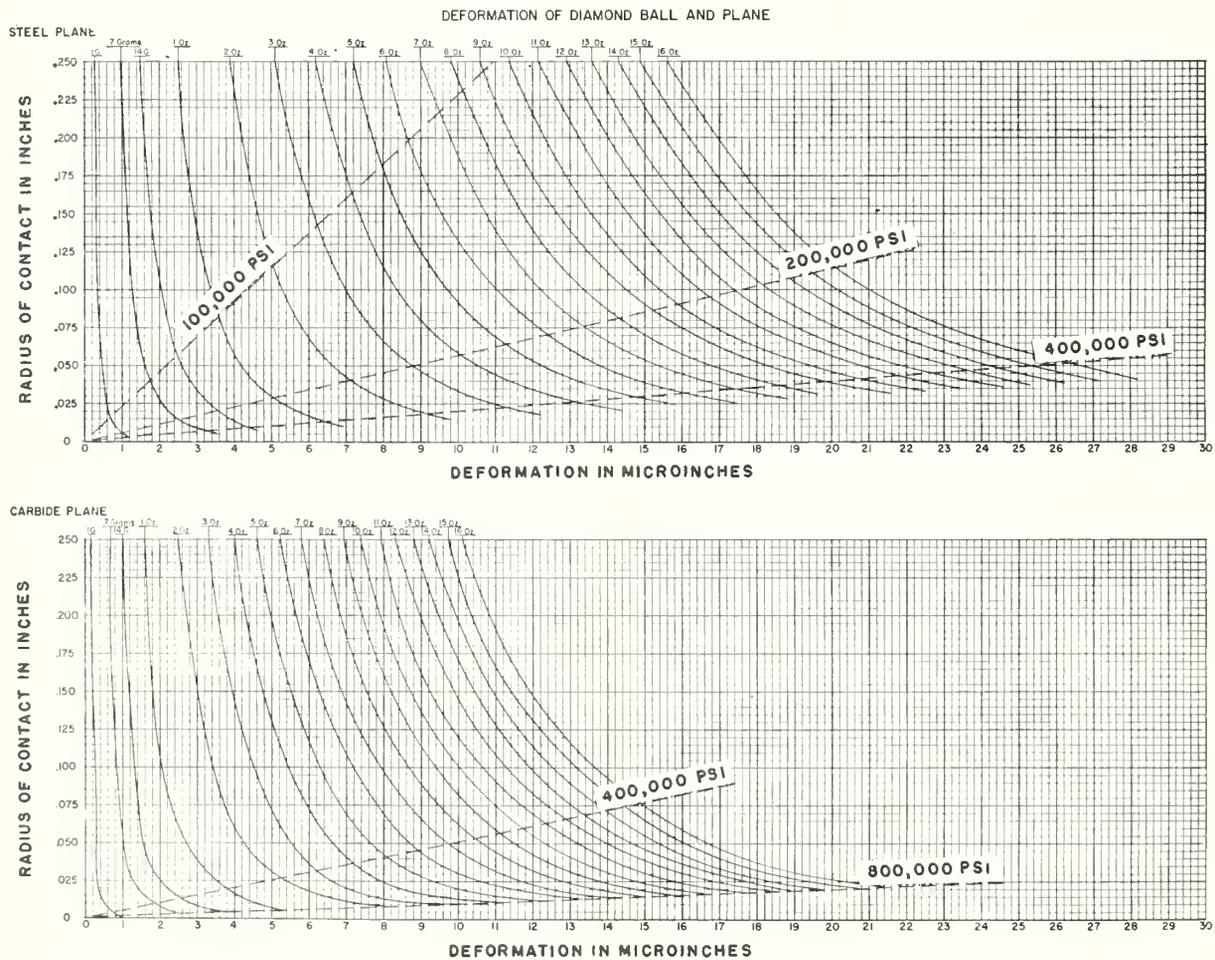


Fig. 5—Chart showing relationships between materials, radius of contact, and elastic deformation; also compressive loads for mechanical comparators.

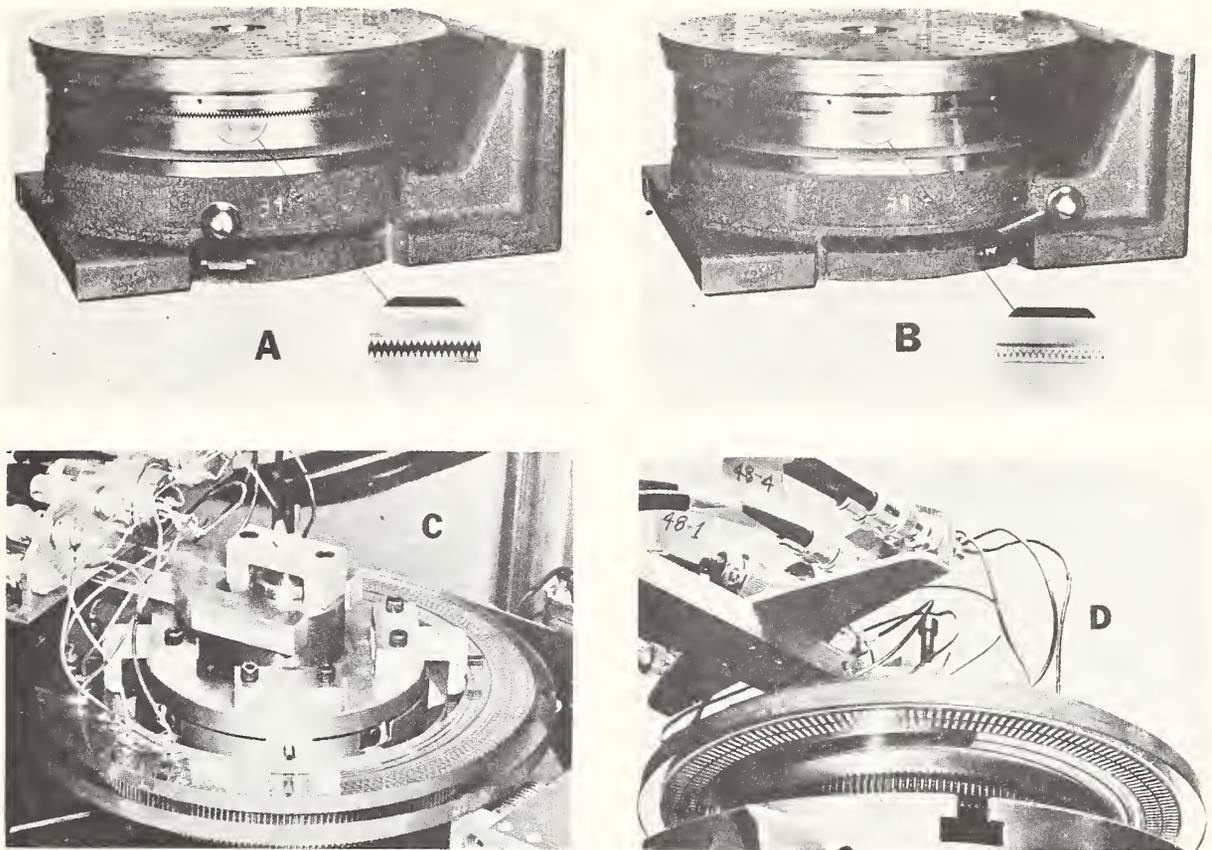


Fig. 6—Averaging principle applied to angle measurement. Mechanical fixture shown open for free rotation (A) and locked in position (B). (Courtesy of AA Gage Co.) Transducer discs showing driver (C) and coupler (D). (Courtesy of Telecomputing Services, Inc.)

of the table with respect to the base is controlled mechanically by the meshing of 360 serrations around the periphery. Thus all of the 360 serrations enter into the positioning of the table at each degree interval. All deviations of the positions of the respective serrations from the nominally correct positions are averaged, resulting effectively in very nearly zero deviation for each position.

4. A transducer¹⁰ for indicating the angular position of a shaft to an accuracy approximating 1 part per million that is essentially a highly accurate electrostatic phase shifter. A pair of dimensionally stable glass discs, Fig. 6c and 6d, each carrying a pattern, photoetched from a copper film deposited on the glass, are rotatable with respect to each other. These patterns (one, a driver, consists of 200 sinusoidal elements, and the other, a coupler, of segments of circles concentric with the center of rotation) face each other and are closely spaced. The coupler pattern picks up the signal contributions of all 200 driver elements simultaneously, thus averaging out the effects of random pattern

deviations in the driver and coupler elements.

Similarly, an Inductosyn system of circular form has been adapted¹ to control rotary positioning tables to an accuracy of 2 seconds.

5. The Societe Genevoise of Geneva, Switzerland, as long as ten years ago applied the averaging principle to photoelectric scanning of lines on a ruled bar to produce another ruled bar of higher accuracy.*

Micro-Geometry

Under the heading of micro-geometry we would consider small deviations from nominally correct form, including surface roughness and deviations from planeness, parallelism, roundness, etc. For length or dimensional measurements to 0.1 microinch accuracy such deviations must not exceed a fraction of a microinch, and even then the length measurement must be confined to a specific and readily located position on each surface. An example of the importance of flatness and parallelism deviations in measurements of highest accuracy is afforded

*No published description has been found.

by the Bureau's experience in measuring a 16-in. and an 18-in. gage block to better than 1 part in 5 million, the first time in its history that the Bureau has certified blocks to such high accuracy.⁶ In these measurements results obtained by two completely independent methods agreed to the nearest microinch or better for both gage blocks. The Bureau's measurements also agreed to 2 microinches with independent measurements of the same gage blocks made by the National Physical Laboratory (England). Of considerable importance in this accomplishment was the accuracy of the Bureau's master blocks with which these blocks were compared. The Bureau had the opportunity to select from among 17 sets those master blocks of a given make that had the least errors in flatness and parallelism of gaging surfaces. Good flatness made possible close and repeatable wringing of the blocks to an optical flat, and the combination of good flatness and parallelism reduced observational errors in making the length comparisons.

Surface roughness is a factor in the accuracy of all measurements, but it is particularly so in high accuracy measurements made by optical interference methods.¹³ Such measurements determine an optical length whereas what is normally required is the mechanical length. This problem is dealt with later in a discussion of interferometry.

For the measurement of fine finishes such as are required on good dimensional standards the most generally applicable means is the microinterferometer, also called the interference microscope. The earliest of these was developed by Linnik,⁵ a Russian, and was manufactured 15 or more years ago by Carl Zeiss of Jena (Fig. 7). A newer design is made by Zeiss of Oberkochen, Fig. 8. Others are the Hilger and Watts, London, Fig. 9a; the "Multimi" of C. E. Johansson, Eskilstuna, Fig. 9b; the "Rugometre" of La Precision Mecanique, Paris; one by Hahn and Kolb, Stuttgart; one by Askania, Berlin; and one by Cook, Troughton and Simms, York, England. Detailed descriptions of these are available in the references.¹²

The Johansson instrument and some others apply the principle of multiple-beam interference in which very narrow, sharp, dark bands are produced. Under certain conditions this permits a more accurate evaluation of scratch depth than two-beam interference. In general the roughness depth measuring range for two-

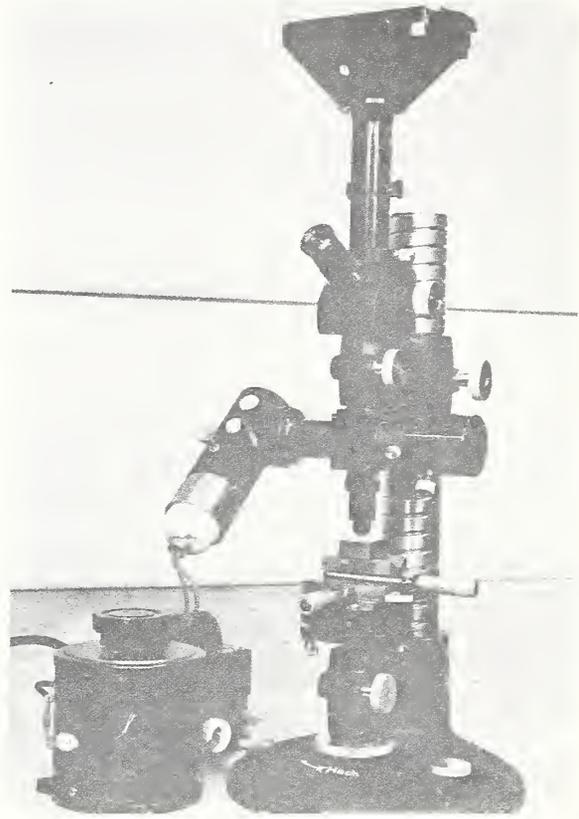


Fig. 7—Linnik microinterferometer.

beam interference starts at about 1 microinch and for multiple-beam from 0.04 to 0.2 microinch. Multiple-beam interference is particularly useful in evaluating small irregularities other than scratches, as for example the thickness of laminae of crystals as exhibited by steps on the surface.

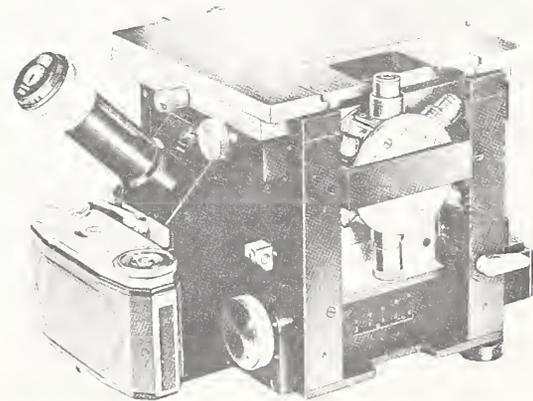


Fig. 8—Zeiss microinterferometer.
(Courtesy of Carl Zeiss, Inc.)

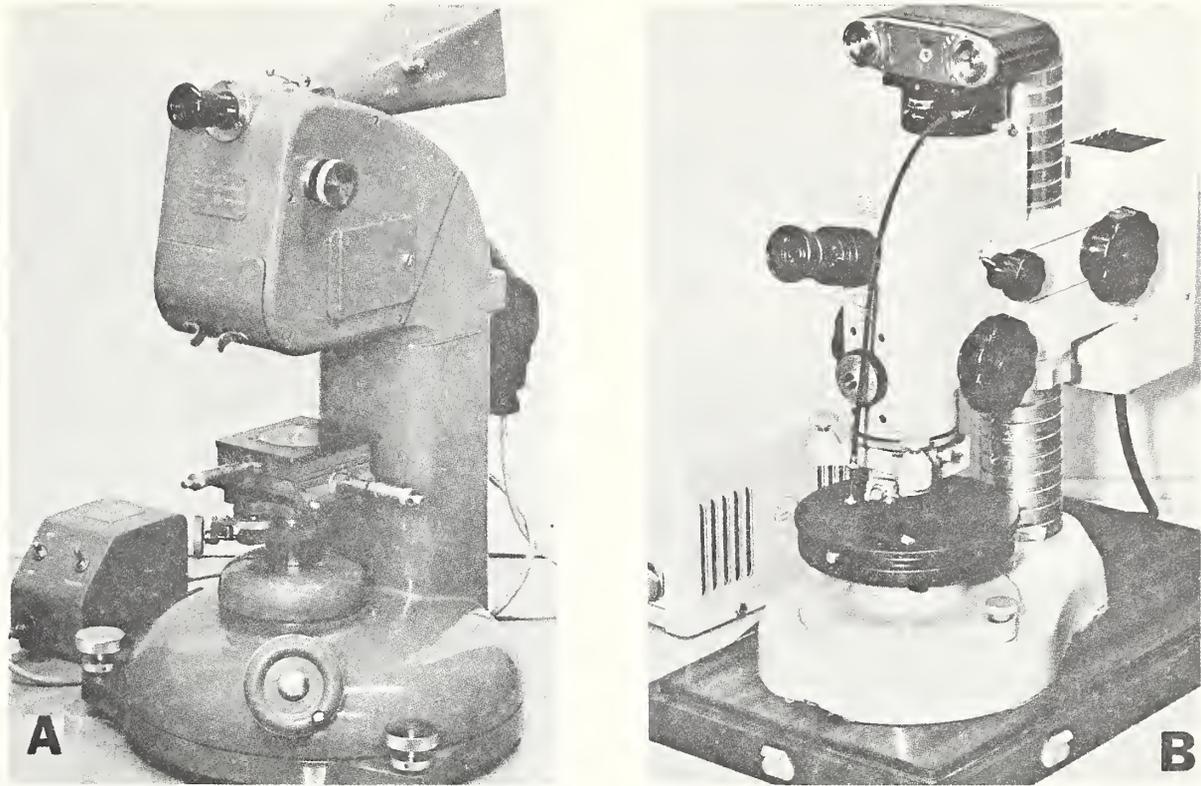


Fig. 9—A. Hilger and Watts microinterferometer.
B. Johansson microinterferometer.

TEMPERATURE CONTROL AND MEASUREMENT

In high-accuracy determinations of lengths or sizes, the accurate measurement of temperature is fully as important as the measurement of dimension. By international accord the nominal dimensions of gages and product shall be correct at a temperature of 68°F (20°C). Thus the most accurate calibration of dimensional standards requires that their actual sizes be determined at 68°F. For extreme accuracy in calibration this involves:

1. Good temperature control at or near 68°F of the ambient air and shielding against heat sources and voids.
2. Accurate measurement of actual temperature of both the dimensional standard and the test piece.
3. Knowledge of the coefficient of thermal expansion of both items in the room temperature range. This may require measurement of this coefficient.

Accurate temperature measurement presents a most serious aspect because there are at least four temperature-dependent variables directly involved in a measurement—two of tempera-

ture measurement and two of thermal expansion measurement. Let us assume that, using an electrical resistance thermometer, we are able to read the thermometer with an uncertainty of 0.001°C and that this reading represents the temperature of a thermocouple on the surface of the steel gage block used as a standard. This uncertainty is about the least that we can attain in temperature measurement. If the coefficient of expansion of the block is 0.000115 in. per deg. C, then in 1 inch of length the uncertainty in the length of the block is $(11.5 \times 10^{-6}) \times (1 \times 10^{-3}) = 11.5 \times 10^{-9}$ or 0.0115 microinch. If there is a like uncertainty in the temperature of the test piece, the total uncertainty for both is 0.023 microinch. Thus, if we are trying to measure to an accuracy of 0.1 microinch, practically one-fourth of this may be lost in temperature measurement alone. Superimposed on this are any uncertainties in the values of the coefficients of thermal expansion of the standard and test piece. Unless these attributes are measured for both the specific standard and test piece involved, the discrepancy in coefficients of the two pieces may be 1 μ in./in. Thus, if the temperature

of measurement of the piece differs from 20°C by 0.1°, the uncertainty in measurement of 1 inch would be 0.1 microinch from this cause alone.

If, on the other hand, the thermal coefficients of the standard and test piece have been measured to within an uncertainty of 0.0000002 in., the maximum uncertainty for both is 0.0000004 in., and to correct for a 0.1° temperature difference the uncertainty would be 0.04 microinch per inch.

Laboratories in which temperature, humidity, and dust are controlled are now very much a matter-of-course for dimensional measurement purposes. The degree of control of temperature varies usually from about 0.5 to about 1°F for the variation at a single position. Accurate control of temperature may not always be required to obtain a sufficient degree of accuracy for a given test when the uncertainty in measurement may be several microinches. Close control pays off, however, in reducing the cycling change of temperature within the measuring instrument and thus reducing the consequent relatively large variations in the readings obtained. In other words, the temperature of the measuring machine remains constant within narrow limits, the necessary number of readings for a given accuracy is reduced with consequent economy in labor. For work of exceptional accuracy the best available control is not sufficient. It is necessary to perform the measurement inside a cabinet insulated against both conducted and radiant heat; also arrangements inside the cabinet should be such as to minimize convection currents. Then, to approach the ultimate in accuracy, thermocouples shielded from radiant energy and convection currents should be applied at various points to both the workpiece and the standard to determine the temperature of each.

To facilitate such temperature measurements D. B. Spangenberg of our staff has assembled the necessary means in a mobile unit, shown in Fig. 10a. At the base of the unit is an insulated box, Fig. 10b, containing a large copper block which provides the reference temperature. Ten thermocouple circuits are provided, and the cold junctions of the thermocouples are inserted in a cavity inside the copper block together with a sensitive mercury thermometer reading to 0.01°F. For higher accuracy a platinum resistance thermometer

may be substituted or used as an auxiliary. Circuits are so arranged that the temperature

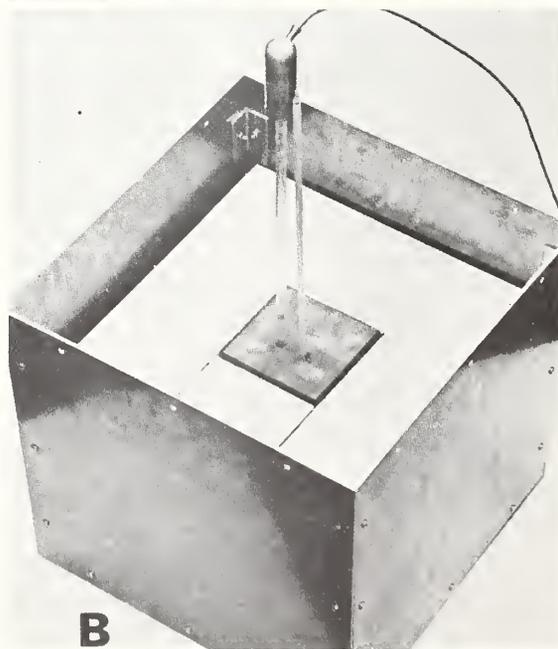
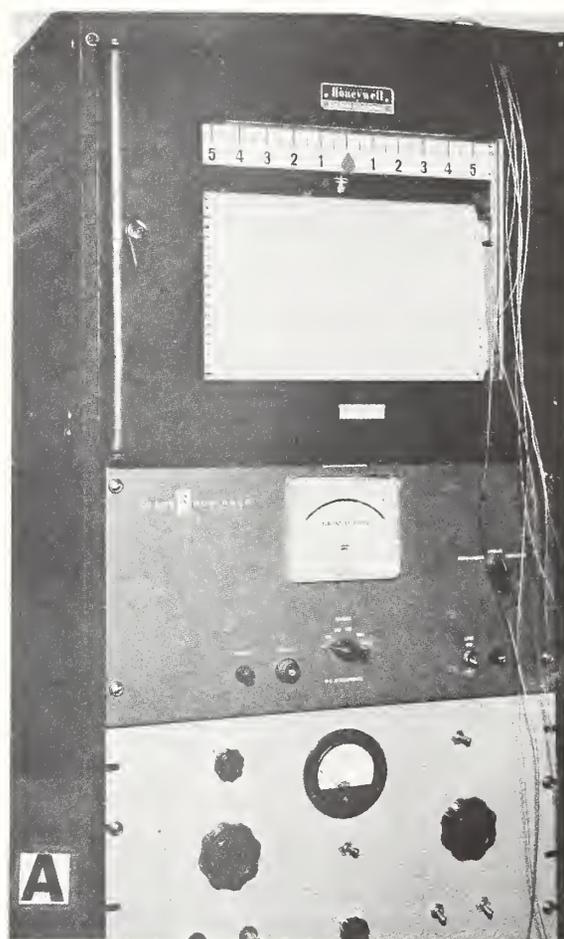


Fig. 10—A. Mobile temperature measuring unit.
B. Reference block and thermometers.

difference between thermocouple junctions may be read directly on a millimeter. A switching arrangement provides for quick successive readings of the thermocouples, which are disposed at various selected positions on the standard and test piece. Temperature differences may be plotted by a recorder located at the top of the unit.

Another disturbing factor is the temperature gradient within the test piece and in the standard from the center to the outside surface of each. This gradient does exist in significant amount even when all precautions are taken to provide a constant ambient temperature. To measure this gradient would require that thermocouples be embedded inside the test piece and standard as well as on the surface of each. We cite as an example the following experience⁷: The temperature of a copper block in an insulated box, related to that of the air in the room, showed a seasonal variation and was generally colder than the air in the winter and warmer in summer. Such a cyclic effect would likewise exist in a test piece and account for the fact that variations in 33 absolute measurements during 5 years of a 2-in. master gage block, although constant within 0.000001 in., were best fitted by a cyclic function with a yearly period.

It is generally not practicable to insert a thermocouple inside of a standard, but in our new absolute interferometer, designed by T. R. Young, the construction of which is nearly completed, this measurement will be accomplished by means of a dummy test block having thermocouples both inside and on the surface.

HIGH AMPLIFICATION

In all high-accuracy measurements, high amplifications in the order of 20,000 times or more are required. Linearity over a range of at least 50 micrometers is desirable. Optical interference fringes of normal fringe spacing give amplifications, when observed directly, of from about 12,000 to 25,000 depending on the fringe spacing used. This amplification has been multiplied by a factor of 60 by applying a micrometer for reading fringe fractions, to produce an amplification of nearly 1 million times in an achromatic interference comparator designed by Young and Saunders.¹⁵ The limiting factor relative to amplification in such an instrument is the accuracy to which a cross hair can be centered on the boundary between two

colors of a polychromatic interference fringe. Interference fringes have the advantage that the amplification is essentially linear throughout the coherence length of interference, which has now attained a maximum of about 33 in. for ordinary discharge-tube light sources.

For mechanical comparisons of dimensions, satisfactory high amplifications are available in electronic circuits applied in various well-known commercial comparators.

INTERFEROMETRY

A major part of the research and development work of the Engineering Metrology Section is in the field of interferometry. In the application of optical interference methods it must be remembered that wavelengths of light are fixed and stable standards only when the light is transmitted in a vacuum. When transmitted through air, the wavelength is shortened in proportion to the refractive index of the portion of air used in the particular experiment. The refractive index is affected by the air temperature, barometric pressure, humidity, carbon dioxide content, and pollution of the air. It is computed from measurements of these individual factors, each of which is a potential source of error. The most serious source of error is the barometric reading, which is affected by the accuracy of setting of the level of mercury in the well of the barometer, the measurement of the height of the mercury column from this level, the correction of this height for the deviation of the value of the acceleration of gravity from the standard value in the locality where the measurement is made, and the temperature of the mercury column. The overall error in determining the actual wavelength may be greatly reduced by direct measurement of the refractive index of air. J. B. Saunders of our staff has designed an interferometer in which a given wavelength of light in air is directly compared with the corresponding wavelength in vacuum. The interferometer is under construction at the present time. This is an essential development toward achieving an accuracy of 0.1 micrometer per in. in the measurement of gage blocks.

In applying optical interference methods to determine, with ultimate accuracy, the length of a contact standard (such as a gage block) by applying light wavelengths as standards, it becomes necessary to evaluate and apply the following additional corrections, particularly in

measuring lengths longer than one inch:

1. A correction based on the dimensions of the slits through which light enters into an interferometer and emerges to the eye. The optical path lengths of rays originating at the edges of the slit are longer than for rays at the center of the slit. This correction can be computed.¹⁶ It is minimized by using as narrow a slit as will provide sufficient light to permit seeing the interference bands. Mr. Strang of our staff has developed data (Table II) relative to slit dimensions of a Zeiss-Koester's interferometer with collimating lens of 200 mm focal length for light sources commonly used.

Table II

	Light Source	
	Cadmium Red and Mercury 198 Green and Violet	Mercury 198 or Krypton 86
Limiting aperture, width, in.	0.010	0.003
Limiting aperture, length, in.	0.010	0.009
Slit correction, microinches/in.	0.13	0.06

2. An obliquity correction based on the viewing angle. This depends on the design of the interferometer and needs to be evaluated for the particular instrument used.

In addition to the above it is always necessary to apply a correction for change of phase of the waveform of light on reflection from surfaces of different materials and/or different surface roughnesses. This is the correction which converts the optical length to the mechanical length of a gage block. This phase shift is a combination of two factors, one related to the con-

ductivity of the reflecting material, being zero for a nonconductor such as quartz, and equivalent to about one microinch for steel. Koester⁴ measured the amount that it varies with the wavelength and found that for an aluminum coating the change in terms of length is about twice as much in the violet as it is in the red spectrum lines.

The factor corresponding to surface roughness is very difficult to measure absolutely, as pointed out by Engelhard² and Saunders¹¹ but, once evaluated for a given sample surface, such surface can be used as a secondary standard for comparison purposes. An instrument for making such a comparison, shown in Fig. 11 and described in reference,² is an Ulbricht's sphere, by means of which is measured the ratio of scattered light reflected from the surface to total reflected light. The Bureau has acquired this instrument and is in the process of evaluating it for practical application in the measurement of gage blocks.

FUTURE DEVELOPMENTS

Researchers in physical science tend to gravitate to the more glamorous fields of investigation such as electronics and nucleonics. However, even these fields of engineering and science are finding application in metrology, and thus the age-old and seemingly prosaic quest for increased accuracy in dimensional measurement is not lacking in excitement in these days. Interferometry is by no means fully developed. Work is proceeding on the problem of narrowing spectrum lines, that is, producing more homogeneous wavelengths and thus extending the coherence length of interference to twice and possibly several times the 85 cm (33 in.) that represents the present limit of coherence using krypton 86. Thus it is hoped that it will be possible to measure a one-meter length absolutely in a single step.

A good beginning has been made by C. E. Haven and A. G. Strang of our staff in the application of interferometry to the measurement of diameters of balls and cylinders. A publication covering this development is in prospect.

In the electronics field the photocell is beginning to be substituted for the eye in reading telescopes and microscopes. This leads to the automatic calibration of the subdivisions of line standards, a process that is now being made effective at NBS. It also leads toward

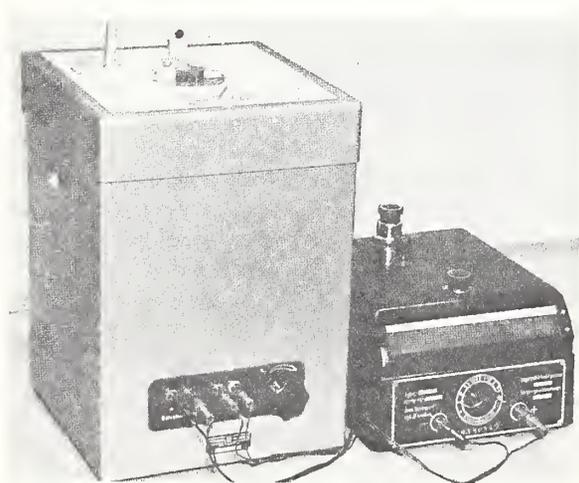


Fig. 11—Photometer for measuring ratio of scattered to total light reflected by a surface.

the automatic calibration of polygons used as high accuracy angle standards, with an increase in accuracy over that attainable by visual readings of autocollimators. We are making a beginning on this at NBS.

In applying mathematics to measurement, statistical methods and the use of high-speed computers are finding increasing application in the reduction of observed data. It is also possible that we shall go beyond the conventional branches of algebra, geometry, trigonometry, involutometry, etc., and begin to apply the differential calculus, particularly to facilitate the solution of problems of measuring deviations from geometric regularity.

Automation already has limited application in metrology, and the next few years will see some interesting further developments. Present examples are: (1) In the calibration of gage blocks at NBS by mechanical comparison with standards, a meter, showing displacement of the comparator stylus, is zeroed on the standard, and the reading on the test piece is punched on a card. Two such independent comparisons are made. A set of cards for the set of blocks is processed by a computer which prints out the deviations of the blocks from nominal size. (2) The electronic averaging device for measuring angles illustrated in Fig. 6c has been utilized in connection with a digitalizer so that the relative angular positions of the two discs is instantaneously shown, in decimal parts of the total circle, to five or six decimal places. Under current development at NBS are: (1) the automatic calibration of all the subintervals of a meter or yard standard, (2) the extension of the automatic calibration of gage blocks to absolute interferometric readings in two light wavelengths, and (3) the automatic calibration of all angles of a polygon using photoelectric autocollimators.

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The Basis of Our Measuring System*

A. G. McNISH†

Summary—The measuring system used for scientific work affords a means of making physical measurements with great precision and accuracy. The best measurements can be made of the quantities taken for the basis of the system. A decrease in both accuracy and precision arises in measuring quantities which are related to them in a complicated way. The standards which fix the magnitudes of the units on which the system is based appear to be very constant. Some improvement in the system may be obtained by substituting physical constants for these standards. This has already been done for the standard of temperature, and it can be done advantageously for the standards of length and time; but there seems to be no way to replace advantageously the standard for mass.

INTRODUCTION

THE most satisfactory kind of information that we can secure about phenomena is developed through measurement. If the phenomena are not susceptible to measurement, the information we can obtain of them is rather unsatisfactory and incomplete. It is the high measurability of the phenomena involved which sets the physical sciences apart from the others and causes them to be called the exact sciences.

Corollary to this, the more accurate the measurements, the better is the information which we obtain. To a considerable extent, the spectacular advances we have achieved in the physical sciences during the past few decades are the results of more accurate measurements. Rough descriptions of phenomena are no longer satisfactory. A few centuries ago, it was adequate to know that the rate of free fall for a body is independent of its mass. Later, the knowledge that the path of a freely falling body with some initial motion describes an approximate parabola sufficed. Today we recognize that the ideal path is actually an ellipse; and, if the gravitational field is not completely specified by the inverse square law, perturbations of the elliptical path must be allowed for, as in the case of an artificial satellite.

In order to carry out accurate measurements, we have established a system of units and a system of standards to fix and preserve the sizes of the units. What are the requirements of such a system? How many units and how many standards do we need? And how many of them are what we call "basic"?

Let us consider several possible systems. For example, we might take for the unit of length, the length of some arbitrarily selected bar; for the unit of mass, the mass of some specified object; the unit of electromotive force, the open circuit EMF of a particular Clark cell; the unit of current, the short-circuit current of a particular grav-

ity cell; the unit of resistance, the resistance of a particular piece of wire, etc. For each quantity in physics, we would have an independent unit for measuring that quantity and the magnitude of each unit would be fixed by the standard embodying it.

What are the faults of such a system? One of its disagreeable aspects is that, with units so chosen, it would be a rare case of good fortune if we found that the equation $E=IR$ holds. We would find, of course, $E \propto IR$. Each physical law expressed in the form of an equation would require a different factor of proportionality to relate the sizes of the units involved. However, if we have selected our standards for such a system with good judgment, we would have an excellent set of standards and we could measure physical quantities with great accuracy in terms of the unit embodied by the standards. Also, we could select the standards so that all units would be of convenient size for practical use.

We can exercise a little parsimony in our choice of independent standards by selecting only a few to fix the units of several quantities and have the units for other quantities fixed by physical equations. Thus, for example, having independent units for electromotive force and resistance, we can write $I=kE/R$ to define the unit of current. We can simplify things by setting k equal to unity. How far can we go in our parsimony? What is the smallest number of arbitrary, independent units and standards which are required for a measuring system?

The founders of the metric system sought to reduce the number of independent standards by defining the gram as the mass of one cubic centimeter of water, leaving the meter as the one independent unit and adopting a constant of nature, the density of water, to fix the unit of mass. For reasons to be discussed later, they found this unsatisfactory. Had it worked we would have found it convenient for it would be necessary for measuring laboratories to compare only meter bars. Each could then set up consistent units of area, volume, and mass, depending only on the meter bar.

We can see that with a system such as this an entire system of measuring units can be built up based upon a single arbitrary, independent standard. The unit of time could be the time of swing for a one-meter pendulum, or the time light takes to travel one meter. Other units could be defined by various equations as we did before for the unit of electric current.

If we set all of the constants in our defining equations equal to unity, we will find this system very convenient for theoretical work, for most equations are in their simplest form. But we will not find the system good for experimental work because many of the quantities most frequently measured in physics would not be measurable

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with sufficient accuracy in terms of the units prescribed for them.

Thus we see that we can have a multi-unit multi-standard system in which all units and standards must be regarded as "basic." Also, we have seen that we can have a single-standard system in which only one quantity is taken as basic. Is this the limit to which our parsimony can go? Can we have a consistent system of measurement with *no standard at all*? The answer to this is, yes, and a moment's reflection will show that it can be done in a number of ways.

To illustrate one of the many ways this can be done, suppose, we rewrite the electromagnetic wave equation $\partial^2\phi/\partial t^2 = \partial^2\phi/\partial x^2$ instead of $\partial^2\phi/\partial t^2 = c^2\partial^2\phi/\partial x^2$, the quantum equation $E = \nu$ instead of $E = h\nu$, the gravity equation $F = m_1m_2/r^2$ instead of $F = Gm_1m_2/r^2$, and the molecular energy equation $E = (3/2)T$ instead of $E = (3/2)kT$. Now with provisional measuring units of arbitrary size we can perform various experiments involving these equations, assuming that other equations of physics are written in their conventional forms. We then solve the results obtained and find what values we must assign to our provisional units. If these sets of experiments are performed by different people, they will be in agreement on the sizes of their units.

The necessary experiments already have been performed, using our conventional units of measurement, so that we can write down the equivalence of these in our new units as shown in Table I. To put it another

TABLE I
APPROXIMATE VALUES OF CONVENTIONAL UNITS IN TERMS
OF UNITS DERIVED BY SETTING c , G , h , k AND
 ϵ_0 EQUAL TO UNITY

One meter	2×10^{34} length units
One second	8×10^{42} time units
One kilogram	2×10^7 mass units
One degree Kelvin	2×10^{-33} temperature unit
One watt	2×10^{-53} power unit
One coulomb	2×10^{17} charge units

way, what we have done is to assign the value unity to the speed of light c , Planck's constant h , the universal gravity constant, G , and Boltzmann's constant k . If we also set μ_0 and ϵ_0 , which we call the permeability and permittivity of space equal to unity, as we may do consistently since c is unity, further interesting results are obtained. We now find that the units of the electrostatic system and the electromagnetic system as defined by Coulomb's equations are of identical magnitude; the unit of electric charge is about 4.8×10^{-18} coulombs, which is approximately equal to the nuclear charge for the elements where the percentage mass defect is a maximum; the electronic charge is given exactly by $e = \sqrt{\alpha/2\pi}$, where α is the well-known Sommerfeld fine-structure constant, approximately equal to $1/137$, Eddington's magic number! Also the unit of energy is exactly the energy released when one unit of mass is annihilated in accordance with Einstein's equation $E = mc^2$.

In spite of a number of appealing features of this system we cannot recommend it for practical use. Because of the uncertainty involved at the present time in relating experimentally the gravitational constant to other quantities of physics, the magnitudes of the units so defined would have an uncertainty in their practical realization of about 1 part in 1000. We can, of course, use some other constant instead of G and thus reduce the uncertainties, but they would still be much greater than we can afford to have them.

We now ask ourselves if we have really eliminated standards from our system by this procedure. Have we not made c , h , G , k , and ϵ_0 our new standards? Are these not our new "basic" quantities of physics? This is all a matter of point of view.

Some of these concepts may seem heterodox to those who have been indoctrinated in the trinity of mass, length, and time as fundamental units. They have been touched on here because some smattering of them is necessary to understand the system of units and standards currently used by physical scientists of all nations of the world. They serve to illustrate the requirements of a system of units and standards for an adequate measuring system, requirements which are very well met by our present system.

OUR PRESENT UNITS AND STANDARDS

The authors of the metric system intended that the meter should be one ten-millionth of the length of the north polar quadrant of the Paris meridian, demonstrating a desire that a measuring system be based on some natural magnitude which would remain constant, a desire which still exists with metrologists. But they soon found that they could compare two meter bars with each other with greater precision than they could relate them to the earth's quadrant.

Similarly, they found that the masses of two metal cylinders weighing about one kilogram each could be compared with each other more precisely than either could be related to the mass of 1000 cubic centimeters of water which was supposed to define them. Thus, the pioneers in precise metrology found that greater precision in measurement could be achieved if they adopted some readily measurable artificial things for standards than if they adopted less readily measurable things of nature.

Accordingly, the International Commission of the Meter, meeting in 1872, resolved to take the meter in the Archives of Paris "as is" (*dans l'état où il se trouve*) for the standard of length. Similarly, they adopted the platinum-iridium kilogram of the Archives as the standard of mass, "considering that the simple relationship, established by the authors of the metric system between the unit of weight and unit of volume, is represented by the actual kilogram in a sufficiently exact manner for the ordinary uses of industry and science . . . and that the exact sciences do not have the self-same need of a simple numerical relationship, but only

of a determination as perfect as possible of this relationship." Three years later many of the leading nations of the world signed the treaty of the meter which created a procedure for coordinating the standards of measurement for the scientific world through an International Bureau of Weights and Measures and a General Conference of Weights and Measures.

For many years no new standard for time was adopted, the ancient definition of the second as $1/86,400$ part of a mean solar day being retained. A separate and independent standard for temperature measurements was adopted and changed a number of times until, in 1954, the thermodynamic temperature scale was based on the triple-point temperature of water as 273.16°K . Other units were defined in terms of the units embodied by these standards by agreed-on equations of physics.

Thus, our system of units and standards does not follow either of the extreme types of systems described in our introduction. Four, and four only, independent standards have been adopted to which we attach the name prototypes. The reasons for this are clear. They are standards for quantities which can be measured very accurately and they are standards which we believe can be preserved or *re*-produced, as in the case for the standard of temperature, very accurately. Furthermore, standards for other quantities can be constructed from them with adequate accuracy in accordance with the defining physical equations.

FROM PROTOTYPES TO ELECTRICITY STANDARDS

As pointed out before, we could establish our standards of electricity and magnetism with the same degree of arbitrariness that we used for the prototypes. However, it is not desirable that we do this, for then the units of electrical and mechanical power would not be the same unless we cluttered up the equations of electricity and magnetism with an unnecessary number of numerical constants. Furthermore, standards for the more useful electric quantities may be established in terms of the prototype standards with great accuracy, as shown by Silsbee.¹

In the early days of research in electricity and magnetism, there was no way to measure these phenomena accurately. The strength of a current was measured by the deflection of a compass needle which the current produced, but, since the deflection depended on the strength of the earth's magnetic field, two observers in different places could not compare their results. Variations in the strength of the geomagnetic field from place were measured by timing the oscillations of a "permanent" magnet which, even in those days, was known to be far from permanent.

C. F. Gauss first showed how magnetic and electric quantities could be measured accurately in terms of the units used for mechanical quantities which are embodied in relatively invariant standards. This is the well-known magnetometer experiment which every student

of physics and electrical engineering must have performed. That Gauss thoroughly understood what he was doing we must presume. That he anticipated the reverence and mysticism with which future generations of scientists would regard the units of mass, length, and time is not likely. It is clear from the writings of those who followed him that they recognized a duplicity in electric units (esu) and magnetic units (emu) and that they could not simultaneously set ϵ_0 and μ_0 in these systems equal to a dimensionless unity and still retain the generally agreed-on unit of time as well as the metric units of length and of mass.

Thus, in the early days of electromagnetic science, the systems of units were not on a satisfactory basis since electric and magnetic units were conflictingly defined. Nor was the situation remedied by combining the esu and emu systems into that bifurcated system which falsely is called Gaussian. A further disagreeable feature is that some of the units in both systems are of inconvenient size, and are not even approximately equal to the units for voltage, current, and resistance which had been adopted by the communications engineers of that day.

To resolve these difficulties, Giorgi proposed that one more arbitrary unit and a standard for it, this time for an electric quantity, be added to the system. By suitably choosing this new unit we can make the MKS units and the practical electrical units form a consistent system. What we mean by this is that equations of the type $W = I^2R$ would be valid if I and R were expressed in the electrical units and W is expressed in MKS units. Of course, Coulomb's equations for the force between unit electric charges and between "unit magnetic poles" would contain constants of proportionality other than unity just as does the equation for the gravitational attraction between two unit masses, as it is ordinarily written.

Giorgi's proposal found such increasing favor among scientists that in 1946 the International Commission on Weights and Measures adopted it in principle. But instead of adopting a new independent unit and a standard for it, they redefined the ampere¹ in accordance with another suggestion of Giorgi in such a way as was equivalent to rewriting the equation for the force between two infinitely long current-carrying conductors. The old emu equation for force per unit length for unit separation of the conductors was $F = 2I^2$, where F is in dynes and I in abamperes. The new form of the equation became $F = 2\mu_0 I^2 / 4\pi$, where F is in newtons, I in amperes, and μ_0 has the value $4\pi \times 10^{-7}$, if rationalized equations are used. It follows from this that the equation for the force between two charges is $F = Q_1 Q_2 / 4\pi \epsilon_0 R^2$ and, if F is in newtons, Q_1 and Q_2 in coulombs, and R in meters, the equation is correct if ϵ_0 is given by $1/\mu_0 c^2$, where c is the speed of light in meters per second.

The present-day system of electric units is known as the MKSA system. It is important to recognize that the unit for electric current, the ampere, does not oc-

¹ F. B. Silsbee, "The ampere," this issue.

copy quite the same position in it as the meter, kilogram, and second, for they are prototype units and their values are fixed by their independent, proper standards. On the other hand, the value of the ampere is fixed in terms of them as given by an equation involving an arbitrarily adopted constant of proportionality. Thus the position of the ampere today is somewhat like that of the kilogram in the scheme proposed by the founders of the metric system.

We can measure all physical quantities directly in terms of the prototype standards, but following this procedure in all measurements is inconvenient, inaccurate, and imprecise. For this reason we construct standards for various derived physical quantities and assign values to them in terms of the corresponding unit as derived by experiment from defining equations involved and the units fixed by the prototype standards. It is clear that, however carefully we perform the experiments in deriving these standards, they can never have the exactness inherent in the prototype standards. How these errors enter our derived standards is discussed in detail by Silsbee¹ and Engen.²

Among the most accurate of the derived standards are those for some of the electric quantities. Because of this high accuracy and the convenience of making electric measurements, these standards are often used for measuring other quantities. We measure the heat of combustion of fuels, for example, by comparing the heat evolved when they are burned in a calorimeter with the known heat evolved by the passage of an electric current through a resistor. The results are given directly in joules, the internationally agreed-on unit for heat, but many chemists foolishly divide the results by 4.1840 to convert them to calories.

We can see how well our measuring system works in practice by examining how well various physical quantities can be measured in terms of the prototype standards. Several typical quantities are listed in Table II with estimates of the uncertainties involved in the best measurements of standards for these quantities at the National Bureau of Standards. The uncertainties are expressed in two ways, those of accuracy and those of precision. Under precision are placed the uncertainties in comparing two nominally identical standards for the quantity involved. Under accuracy are placed estimates of the uncertainties in relating the derived standards to the prototype standards. The uncertainties correspond approximately to "probable errors." No entries under accuracy are given for the meter, the kilogram, or the triple-point temperature of water since they are "accurate" by definition. The quantity, time, is not included in the table because it involves some considerations which will be treated at length later.

The table illustrates a number of interesting characteristics of our measuring system. The greatest precision is attained in measuring the quantities which

have been selected as the basis of our system. In all cases illustrated except one, maximum precision is achieved when the magnitude of the quantity is the unit for the quantity. In all cases there is a decrease in accuracy for standards which embody a multiple or submultiple of the selected unit. Derived standards are subject to considerable inaccuracy, and this inaccuracy increases with the experimental complexity involved in relating them to the prototype standards. However, derived standards may be compared with similar ones with a precision far greater than the accuracy of the particular standard involved.

TABLE II
ESTIMATES OF ACCURACY AND PRECISION IN
MEASURING PHYSICAL QUANTITIES

Physical quantity	Device	Magnitude	Uncertainty in parts per Million	
			Accuracy	Precision
Length	Meter bar	1 meter	—	0.03
	Gage block	0.1 meter	0.1	0.01
	Geodetic tape	50 meters	0.3	0.10
Mass	Cylinder	1 kilogram	—	0.005
	Cylinder	1 gram	1	.03
	Cylinder	20 kilogram	0.5	0.1
Temperature	Triple-point cell	273.16°K	—	0.3
	Gas thermometer	90.18°K	100	20
	Optical pyrometer	3000°K	1300	300
Resistance	Resistor	1 ohm	5	0.1
	Resistor	1000 ohms	7	1
	Resistor	0.001 ohm	7	1
Voltage	Standard cell	1 volt	7	0.1
	Volt box-standard cell	1000 volts	25	10
Power DC	Standard cell-resistor	1 watt	11	1.5
	Wattmeter	10-1000 watts	100	50
	Microcalorimeter	0.01 watt	1000	100

The maintenance and establishment of standards for all kinds of physical measurements is the basic responsibility of National Bureau of Standards. Fulfillment of this task requires development of precise measurement techniques and prosecution of basic research in most fields of the physical sciences. For a standards program is not a static program. Growing technology requires more and more standards, extension of the range of existing standards, and improvement in accuracy. For example, the rather poor accuracy associated with measurement of microwave power is not due to difficulties in the experiment alone, but partly to the fact that accurate measurement of microwave power is a recent requirement of our technology. It is interesting to note that the old "International" Ohm as embodied in the standard specified for it by the International Conference on Electrical Units and Standards in 1908, and used until 1948, differed from its theoretical value by about 500 parts in 1,000,000.

² G. F. Engen, "A refined X-band microwave microcalorimeter," to be published.

Since wavelengths emitted depend on conditions of excitation, experiments have been conducted with lamps containing krypton 86 held at constant pressure by immersion in liquid nitrogen at its triple point. Various national laboratories have found this wavelength standard so satisfactory that the International Committee on Weights and Measures in October last year recommended that the General Conference, which meets in 1960, redefine the meter in terms of wavelength of a specified krypton line, and suggested a number for this equivalence based on the measurements of the cadmium wavelengths and comparisons of the wavelengths of the cadmium and krypton lines.

The effect of this redefinition, if it is adopted by the General Conference, will not be marked. The meter has never been related directly to the chosen krypton line, but the interrelations are sufficiently well known that any differences will probably be within the uncertainties of measurement now existing. It will have the effect of embodying the unit of length in what we believe to be an immutable standard and thus fulfilling an old aspiration. Meter bars, gage blocks, etc., will continue to be used as standards for the kinds of measurements for which they are suited. Every once in a while the length of meter bars will be redetermined in terms of the wavelength of light, instead of determining the wavelength of light in terms of the international meter bar.

The possibility of embodying our unit for time in a physical constant is even more attractive. We can measure time, and its reciprocal, frequency, with the greatest precision of any physical quantity. For example, we may compare the ratio of the average frequencies of two oscillators over concurrent time intervals with as great a precision as we choose. The limit is set by how long the oscillators will operate and how many cycles we wish to count, but such comparisons are pointless if the frequencies of the oscillators are not relatively stable over the interval involved. Conversely, we may measure time with equal precision by counting cycles of a particular oscillator, assuming its frequency is constant.

There is a serious problem in measuring time accurately. We can lay two meter bars side-by-side and compare their lengths. If, by subsequent comparisons, we find that their lengths have not changed relatively we have confidence that our length standards have not changed. But there is no way to lay two time intervals side-by-side; we must rely on the stability of an oscillator to compare time intervals. Man-made oscillators show drifts in frequency with respect to each other, and since oscillators are not passive things like meter bars and kilogram weights, we expect them to drift.

To obtain a good standard for time and frequency we adopted first an astronomical constant, the rotational frequency of the earth which had been regarded as constant since the days of Joshua. Man-made oscillators were used to interpolate for shorter intervals of time and the second was defined as $1/86400$ of a mean solar day. Since the apparent solar day varies throughout the

year due to eccentricity of the earth's orbit, astronomers kept track of time by observing star transits, in relation to which earth's rotation is much more uniform.

Precise astronomical observations revealed that this standard was not good enough. The frequency of rotation of the earth is changing with respect to the revolutions of the moon about the earth and the earth about the sun, when allowance is made for perturbations of the revolution time. All planetary motions are in substantial accord. In addition to a gradual slowing down, which is to be expected from tidal friction, there are erratic fluctuations in rotational speed. For this reason astronomers carry out their more precise calculations in ephemeris time, which is based on planetary motions.

With the improvement of quartz-crystal oscillators, seasonal fluctuations in the earth's rotation with respect to the stars have appeared. Though the oscillator frequencies drift, they drift monotonically, allowing us to measure these seasonal fluctuations which amount to 1 part in 10^8 . Correcting for this seasonal fluctuation, astronomers⁵ have established a more uniform time scale, called UT2, good to 1 part in 10^9 , tied in with the earth's rotation, and hence subject to effects of long term changes.

So we see that even the smoothed rotation frequency of the earth is not good enough for a standard. Accordingly the second was redefined in 1956 by the International Committee on Weights and Measures as $1/31556925.9747$ of the tropical year 1900 at 12 hours ephemeris time. Why this strange definition? Why not take the sidereal year or the anomalistic year? The lengths of all these years change in known, highly regular ways, so that specification of any epoch was necessary. We chose the tropical year, which is the time between two successive passages of the center of the sun across the celestial equator in the same sense, because accurate tables were already available for its variation, based on the epoch 1900.

The need for a better standard of time became urgent during the past decade with the improvement in microwave techniques. Microwave terms in the spectra of molecules and atoms were being measured with increased precision. The Bureau began to explore these phenomena as the basis for constructing more stable oscillators.⁶ Before 1952 Lyons and his co-workers at the Bureau⁷ had measured the microwave resonance in the ground state of the cesium atom with a precision of 1 part in 10^7 . Essen and his co-workers⁸ at the National Physical Laboratory a few years later increased this precision to a few parts in 10^{10} . It is likely that greater

⁵ U. S. Naval Observatory, "The Naval Observatory Time Service," Circular No. 49; 1954.

⁶ B. F. Husten and H. Lyons, "Microwave frequency measurements and standards," *Trans. AIEE*, vol. 67, pp. 321-328; 1948.

⁷ J. E. Sherwood, H. Lyons, R. H. McCracken, and P. Kusch, "High frequency lines in the hfs spectrum of cesium," *Phys. Rev.*, vol. 86, p. 618; 1952.

⁸ L. Essen and J. V. L. Parry, "The caesium resonator as a standard of frequency and time," *Phil. Trans. R. Soc., London*, vol. 250, pp. 45-69; 1957.

precision can be attained in measuring the cesium frequency and also other atomic frequencies such as those of rubidium, as was indicated by recent work of Bender and Beaty of the Bureau and Chi of the Naval Research Laboratory.⁹ Since the frequencies of these resonances depend on energy levels of the atoms involved, and since, in the case of the cesium, independent experiments have agreed to within the limits of precision, we may presume that they can serve well as standards for time and frequency.

How can we relate these resonance frequencies to the defined unit of time, the ephemeris second? Since the second is defined by an event which occurred over 50 years ago we must measure the resonance frequencies in terms of current values of the UT2 second, and then, through observations on the moon, relate the UT2 second to the ephemeris second. This was done¹⁰ recently, based on four years of observation of the moon, showing that the ephemeris second corresponds to $9,192,632,770 \pm 20$ cycles of the cesium frequency. This is much less precise than the defined value of the second or the precision with which the cesium resonance can be observed.

We have no hope of relating the atomic resonances to the ephemeris second with much greater precision in the near future. We thus are faced with the fact that atomic constants are much better standards for time and frequency than astronomical constants. Furthermore, as standards they are much more accessible than the astronomical constants which require long years of observation to compare them precisely with other quantities. Clearly, we are able today to improve our standard for time by selecting one of the atomic resonances and defining the second in terms of it, making the definition such that the new definition will agree as closely as feasible with the present one.

The frequencies broadcast by the Bureau's stations WWV and WWVH are now monitored and kept as constant as possible by reference to the cesium resonance. The intervals between the seconds pulses are maintained in the same way. Therefore the seconds pulses gradually get out of step with mean solar time. When the difference becomes great enough the pulses are shifted by exactly 20 milliseconds to bring them back in. Thus we are already using two kinds of time, atomic time—that's for the scientists—and mean solar time—that's for the birds and other diurnal creatures.

These attempts to improve time and frequency measurement may seem a quest for precision for precision's own sake, a futile pushing of the decimal point. But this is not correct. It is an attempt to establish standards so that we may learn what physics lies beyond the decimal point. For such things have Nobel Prizes been awarded.

⁹ P. L. Bender, E. C. Beaty, and A. R. Chi, "Optical detection of narrow Rb⁸⁷ hyperfine absorption lines," *Phys. Rev. (letter)*, vol. 1, pp. 311-313; 1958.

¹⁰ W. Markowitz, R. G. Hall, L. Essen, and J. V. L. Parry, "Frequency of cesium in terms of ephemeris time," *Phys. Rev. (letter)*, vol. 1, pp. 105-107; 1958.

We have already learned that the earth turns irregularly on its axis. Now we ask, do time scales based on astronomical, atomic, and molecular processes change with respect to each other as some think they might? (The ammonia maser depends on molecular processes for its frequency stability.) Perhaps from these newly achieved precisions and those soon to be achieved we may even be able to resolve experimentally the famous clock paradox of relativity!

We see that, of the four prototype standards, one is already embodied in a physical constant. Another seems about to be, and a third one is ready to be. What about the standard of mass? We can see no way of embodying that in a physical constant at present without detracting from the accuracy of our system. We seem to be "struck" with the platinum-iridium kilogram. And is that bad?

Table II showed that the kilogram is the most precisely measurable of our prototype standards, that uncertainty in comparing it with other masses is about as small as the uncertainty in comparing the cesium second with the ephemeris second. What could cause the kilogram to change? Any damage to it which would remove 1 in 10^9 of its mass by a scratch or nick would be perceived readily by the naked eye. The oxidation of platinum at normal temperatures is so slow it has never been measured and no sign of an oxide coating on the metal has ever been noticed. We know of one calculable change which will take place in it. One of the isotopes of platinum, Pt 190, is radioactive. It undergoes α -decay with a half-life of about 10^{12} years. Since the abundance of this isotope is only 0.012 per cent it will take about 10^8 years for it to produce a change as great as the imprecision of measurement. We do know that the Arago kilogram, produced during the first half of the last century, has exhibited a loss of mass of the order of a few milligrams. This kilogram was forged from sponge platinum and must have had inclusions of gas which escaped. The kilograms of today are of fused metal and free from changes of this nature. There appears to be no good practical reason for replacing the kilogram with a physical constant.

Physical constants are extensively used for derived standards and for standards embodying magnitudes differing greatly from the defined unit. The International Temperature Scale, which is an approximation to the Thermodynamic Scale, is based on the equilibrium temperatures of various substances. Higher temperatures are measured in terms of the constants in Planck's radiation law. The speed of light is employed in the measurement of large distances in optical and radio surveying. The gyromagnetic precession frequency of the proton,¹ which has been measured with great accuracy at the Bureau, affords a means of comparing two different magnetic fields with high precision. Many other physical constants, too numerous to mention, find employment in extending the range of physical measurement. The determination of the values of these many

constants with the greatest possible accuracy in terms of our prototype standards is one of the main tasks of the Bureau.

CONCLUSION

Our measuring system has developed over the years in such a way as to provide the maximum of precision and accuracy in measuring the many quantities of physics. The standards chosen from time to time to preserve the magnitudes of the units were sufficiently measurable and durable to meet the needs of their days. As scientific advancement required better standards, the persons responsible for preservation of the

system have been ready to adopt new standards for the old ones. Thus we see a transition taking place now from a system based on man-made standards to a system based mainly on physical constants, as far as this is possible.

The prototype standards seem to need little improvement. The advances which we must make in our metrology are in extension of the range over which accurate measurements can be accomplished. Particularly, we need to develop techniques to measure accurately new quantities and new aspects of old quantities to meet the requirements of our expanding technology.

Units and Systems of Weights and Measures

Their Origin, Development, and Present Status

Lewis V. Judson



National Bureau of Standards Circular 570

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Preface

The subject of weights and measures is of universal interest. Millions of daily industrial operations and commercial transactions depend on a uniform and convenient system of weights and measures.

In its broadest sense the subject of weights and measures covers much more than the units used in the sale and purchase of commodities. Our high standard of living depends in large part on our ability to measure accurately everything from a loaded railroad car to the diameter of a submicroscopic particle. In fact, almost everyone makes daily use of an accepted system of measurement—from the school child who studies arithmetic to the machinist who measures to a ten-thousandth of an inch, from the housewife who purchases a pound of butter to the manufacturer of automotive engines.

The National Bureau of Standards receives many requests for information on both the customary and metric systems of weights and measures. It is to serve this need—for teachers, students, and the general public—that this Circular has been prepared. It brings together much of the information that was previously available in separate mimeographed leaflets. For scientists and industrialists who want more extensive information on the subject, the Bureau has published *Units and Systems of Weights and Measures*, Miscellaneous Publication 214.

A. V. ASTIN, *Director*.

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Units and Systems of Weights and Measures

Their Origin, Development, and Present Status

Lewis V. Judson

This Circular brings together in a convenient form information about weights and measures that experience has shown is of interest to the general public. Much of it has been issued previously by the National Bureau of Standards in temporary and scattered form. The Circular discusses the origin and early history of units and standards, gives general information about the metric system, and states and explains the present status of standards of length, mass, time, and capacity in the United States and in Great Britain. It discusses for the benefit of the general reader such matters as the distinction between units and standards, and that between mass and weight. Two items of everyday life relating to weights and measures are considered in some detail: The weight of coal, and the definitions and usages of the terms "ton" and "tonnage." The Circular concludes with tables of weights and measures, prepared for the benefit of those requiring such tables for occasional ready reference. School teachers will find in this Circular considerable material to supplement their textbooks.

1. Introduction

The National Bureau of Standards was established by act of Congress in 1901 to serve as a National scientific laboratory in the physical sciences and to provide fundamental measurement standards for science and industry. In carrying out these related functions the Bureau conducts research and development in many fields of physics, mathematics, chemistry, and engineering. At the time of its founding, the Bureau had custody of two primary standards—the meter bar for length and the kilogram cylinder for mass (or weight). With the phenomenal growth of science and technology over the past half century, the Bureau has become a major research institution concerned not only with everyday weights and measures but also with hundreds of other scientific and engineering standards that have become necessary to the industrial progress of the Nation. Nevertheless, the country still looks to the Bureau for information on the units of weights and measures, particularly their definitions and equivalents.

The subject of weights and measures can be treated from several different standpoints. Scientists and engineers are interested in the methods by which precision measurements are made; State weights and measures officials are interested in laws and regulations on the subject and in methods of verifying commercial weighing and measuring devices. But a vastly larger group of people are interested in some general knowledge of the origin and development of weights and measures, of the present status of units and standards, and of miscellaneous facts that will be useful in everyday life. This Circular has been prepared to supply that information on weights and measures that experience has shown to be the common subject of inquiry.

2. Units and Systems of Weights and Measures

The expression "weights and measures" is used in this Circular in its basic sense of referring to measurements such as length, mass, and capacity, thus excluding such topics as electrical measurements and thermometry. This section on units and systems of weights and measures presents some fundamental information to clarify thinking on this subject and to eliminate erroneous and misleading use of terms.

2.1. Origin and Early History of Units and Standards

a. Units and Standards

It is essential that there be established and kept in mind the distinction between the terms "units" and "standards."

A *unit* is a value, quantity, or magnitude in terms of which other values, quantities, or magnitudes are expressed. In general, a unit is fixed by definition and is independent of such physical conditions as temperature. Examples: The yard, the pound, the gallon, the meter, the liter, the gram.

A *standard* is a physical embodiment of a unit. In general it is not independent of physical conditions, and it is a true embodiment of the unit only under specified conditions. For example, a yard standard has a length of one yard when at some definite temperature and supported in a certain manner. If supported in a different manner, it might have to be at a different temperature in order to have a length of 1 yard.

b. General Survey of Early History of Weights and Measures

The beginnings of the development of weights and measures go back to primitive man in prehistoric times. Hence, there is a great deal of uncertainty about the origin and early history of weights and measures. Many believe that the units first used by primitive man were those of length and weight and that units of area, volume, and capacity were of much later origin. Units of length may have been the earliest. These were derived from the limbs of the human body, and included the length of the foot, the width of the palm, the length of the forearm, etc. Units of weight included weights of kernels of grain and weights of shells.

At first these units were not very definitely defined. Later they became somewhat more definite when, for example, the foot became the length of the foot of a tribal chief or other ruler. At a much later date physical standards were made and deposited for safekeeping in a temple or other place of security. These early physical standards were usually very crude; it is generally considered, however, that they were as satisfactory for the needs of the people at that time as our most modern standards are for our own needs.

Our present knowledge of early weights and measures comes from many sources. Some rather early standards have been recovered by archeologists and preserved in museums. The comparison of the dimensions of buildings with the descriptions of contemporary writers is another source of information. An interesting example of this is the comparison of the dimensions of the Greek Parthenon with the description given by Plutarch from which a fairly accurate idea of the size of the Attic foot is obtained. In some cases we have only plausible theories and we must sometimes decide on the interpretation to be given to the evidence. For example, does the fact that the length of the double-cubit of early Babylonia was equal (within two parts in a thousand) to the length of the seconds pendulum at Babylon indicate a scientific knowledge of the pendulum at a very early date, or do we merely have a curious coincidence? By studying the evidence given by all available sources, and by correlating the relevant facts, we obtain some idea of the origin and development of the units. We find that they have changed more or less gradually with the passing of time in a complex manner because of a great variety of modifying influences. We find the units modified and grouped into systems of weights and measures: The Babylonian system, the Philetarian system of the Ptolemaic age, the Olympic system of Greece, the Roman system, and the British system, to mention only a few.

c. Origin and Development of Some Common Units

The origin and development of units of weights and measures has been investigated in considerable detail and a number of books have been written on the subject. It is only possible to give here somewhat sketchily the story about a few units.

One of the earliest units was the *foot*. This was first the length of the human foot without further specification or modification, then the length of the foot of various rulers of tribes and groups of people. Later, by gradual evolution, it was the foot as used in succession by the

Egyptians, Greeks, and Romans, brought to Britain by the Romans, modified with the passing of time, and finally defined in Great Britain as 1/3 of the British-Imperial Yard and in this country as 1/3 of the U. S. yard.

A very interesting and important unit of length used by many ancient peoples was the *cubit*, originally defined as the distance from the point of the elbow to the end of the middle finger. This unit was about 18 inches long, but there were important variations in the length of a cubit.

The *inch* was originally a thumb's breadth. In the Roman duodecimal system it was defined as 1/2 foot, and was introduced into Britain during Roman occupation, where it became a part of the English system of weights and measures.

The *mile* was defined by the Romans as 1 000 paces* or double steps, the pace being equal to 5 Roman feet. This Roman mile of 5 000 Roman feet was introduced into Britain, became 5 000 English feet, and in Tudor times (probably in the reign of Henry VII, 1485 to 1509, but definitely by a statute of Queen Elizabeth, who reigned 1558 to 1603) was changed to 5 280 feet in order to make the furlong of 1/8 mile equal to the rood of 660 feet, or 220 yards (40 rods of 16 1/2 feet, or 5 1/2 yards each).

The *yard* as a unit of length is apparently of much later origin than the units previously discussed. It appears to have had a double origin: (1) as the length of an Anglo-Saxon gird or girdle, and (2) as the length of the double cubit. There is an old tradition, often stated as a fact, that Henry I decreed that the yard should thenceforth be the distance from the tip of his nose to the end of his thumb.

The *point* is the basic unit for measuring type. This unit originated with Pierre Simon Fournier in 1737. It was modified and developed by the Didot brothers, Francois Ambroise and Pierre Francois, in 1755. It was first put into effect in the United States by a Chicago type foundry (Marder, Luse, and Company) in 1878. As adopted in 1886 by the American Type Founders' Association and now defined in the United States, Canada, and Great Britain, it is 0.013 837 inch, a value only slightly less than 1/72 inch.

Of units of weight, one of the earliest is the *grain*, which was originally the weight of a grain of wheat or of some specified seed native to some particular locality.

The Roman *pound* (libra) was the hundredth part of an older weight, the talent, which is believed to have been originally the weight of an Egyptian royal cubic foot of water. The Roman pound was divided into 12 ounces (unciae, meaning twelfth parts) of 437 grains each. This system was introduced into Britain where the pound was increased so as to have 16 of the original ounces. This pound became known as the avoirdupois pound, the word avoirdupois meaning "goods of weight." The idea of a pound divided into 16 parts was not a new one, as the Greeks had divided their pound into 16 parts, as well as into 12 parts. The pound, which in England had long been used for mint purposes and called the troy pound, consisted of 5 760 grains (12 ounces of 480 grains each). The origin of this troy pound and troy ounce is very uncertain. One theory is that the troy pound came from Troyes, France, but there seems to be a serious question whether even the name had its origin in that place. Sometime prior to 1600 A. D., the avoirdupois pound was increased by 8 grains so that it would consist of 7 000 grains instead of 6 992 grains and thus the number of grains in the avoirdupois pound would have a more simple ratio to the number of grains in the troy pound, which, being used for mint purposes, it was considered advisable to keep unchanged.

That the *ton* was the weight of a certain volume of some material is highly probable. Among the Anglo-Saxons it may have been the weight of a quantity of wheat in 32 bushels, that is, in 1 chaldron.

The *stone* was an early unit of weight in the British Isles. At one time it appears to have been 16 pounds in the system: 16 pounds=1 stone, 16 stones=1 wey, 16 weys=1 last, and 1/2 last=1 ton (not the present ton). The stone is still used to a considerable extent in Great Britain, being now equal to 14 pounds except in special cases. (8 stones=1 hundredweight=112 lb; 20 hundredweights=1 ton=2 240 lb. This ton is commonly referred to as the long ton in the United States.)

*It should be noted that a space has been inserted instead of commas in all of the numerical values given in this Circular, following a growing practice originating in tabular work to use the space to separate large numbers into groups of three digits.

A unit of antiquity which has survived without change is the *degree of arc*. The early Babylonians reckoned the year as 360 days. They therefore divided the circle into 360 parts, or degrees. They knew that a chord equal to the radius subtends an arc of 60° . The number 60 became the basis of their sexagesimal number system and is an explanation of the division of the degree into 60 minutes and of the minute into 60 seconds. This is also the basis of the relation between longitude and time. Since the earth makes one complete rotation (360°) on its axis in 24 hours, a time change of 1 hour is represented by each 15° of longitude ($360/24=15$).

2.2. The Metric System

a. The Metric System: Definition, Origin, and Development

The metric system is the international decimal system of weights and measures based on the meter and the kilogram. The essential features of the system were embodied in a report made to the French National Assembly by the Paris Academy of Sciences in 1791. The definitive action taken in 1791 was the outgrowth of recommendations along similar lines dating back to 1670. The adoption of the system in France was slow, but its desirability as an international system was recognized by geodesists and others. On May 20, 1875, an international treaty known as the International Metric Convention was signed providing for an International Bureau of Weights and Measures, thus insuring "the international unification and improvement of the metric system." The metric system is now either obligatory or permissive in every civilized country of the world.

Although the metric system is a decimal system, the words "metric" and "decimal" are not synonymous, and care should be taken not to confuse the two terms.

b. Units and Standards of the Metric System

In the metric system the fundamental units are the meter and the kilogram. The other units of length and mass, as well as all units of area, volume, and capacity, also compound units, such as pressure, are derived from these two fundamental units.

The meter was originally intended to be 1 ten-millionth part of a meridional quadrant of the earth. The Meter of the Archives, the platinum end-standard which was the standard for most of the 19th century, at first was supposed to be exactly this fractional part of the quadrant. More refined measurements over the earth's surface showed that this supposition was not correct. The present international metric standard of length, the International Prototype Meter, a graduated line standard of platinum-iridium, was selected from a group of bars because it was found by precise measurements to have the same length as the Meter of the Archives. The meter is now defined as the distance under specified conditions between the lines on the International Prototype Meter without reference to any measurements of the earth or to the Meter of the Archives, which it superseded. The kilogram was originally intended to be the mass of one cubic decimeter of water at its maximum density, but it is now defined as the mass of the International Prototype Kilogram without reference to the mass of a cubic decimeter of water or to the Kilogram of the Archives. Each of the countries which subscribed to the International Metric Convention was assigned one or more copies of the international standards; these are known as National Prototype Meters and Kilograms. The liter is a unit of capacity based on the mass standard and is defined as the volume occupied, under standard conditions, by a quantity of pure water having a mass of 1 kilogram. This volume is very nearly equal to 1 000 cubic centimeters or 1 cubic decimeter; the actual metric equivalent is, 1 liter=1 000.028 cubic centimeters. (The change in this equivalent from the previously published value of 1 000.027 is based on a recomputation of earlier data carried out at the International Bureau of Weights and Measures.) Thus the milliliter and the liter are larger than the cubic centimeter and the cubic decimeter, respectively, by 28 parts in 1 000 000; except for determinations of high precision, this difference is so small as to be of no consequence.

The metric system, by itself, is not a complete system covering all physical measurements. A complete system requires certain additional units such, for example, as units of temperature and time.

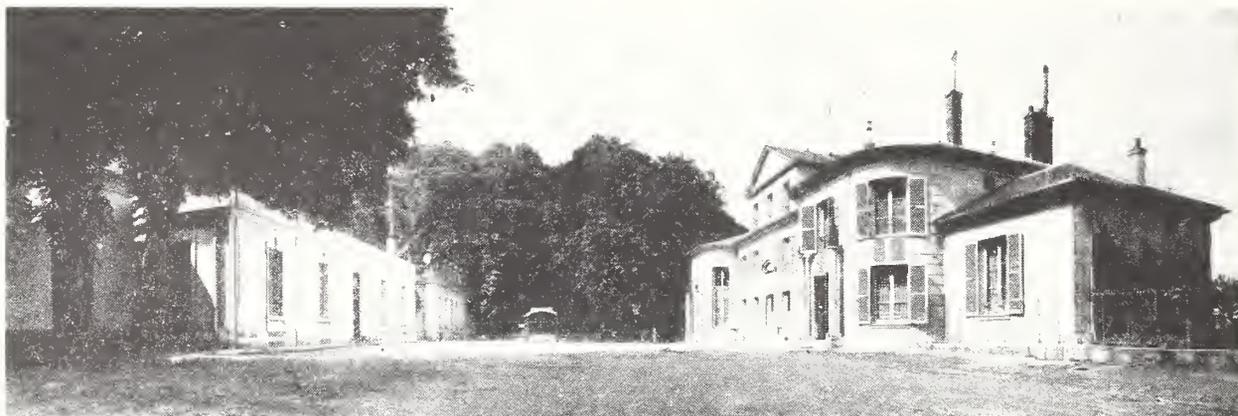


FIGURE 1. *International Bureau of Weights and Measures.*

Founded in accordance with Treaty of May 20, 1875. Located on international territory in Sèvres near Paris, France

c. The International Bureau of Weights and Measures

The International Bureau of Weights and Measures (fig. 1) was established at Sèvres, a suburb of Paris, France, in accordance with the International Metric Convention of May 20, 1875. At the Bureau there are kept the International Prototype Meter and the International Prototype Kilogram, many secondary standards of all sorts, and equipment for comparing standards and making precision measurements. The Bureau, maintained by assessed contributions of the signatory governments, is truly international.

In recent years the scope of the work at the International Bureau has been considerably broadened. It now carries on researches in the fields of electricity and photometry in addition to its former work in weights and measures with which were included such allied fields as thermometry and the measurement of barometric pressures.

d. Present Status of the Metric System in the United States

The use of the metric system in this country was legalized by Act of Congress in 1866, but was not made obligatory.

The United States Prototype Meter No. 27 and United States Prototype Kilogram No. 20 are recognized as the primary standards of length and mass for both the metric and the customary systems of measurement in this country because these standards are the most precise and reliable standards available. Obviously it is not possible to accept both a meter and a yard, and both a kilogram and a pound as "primary" standards, unless there is willingness to accept the possibility of continually changing the ratio between the corresponding units. In each case one must be accepted as the primary standard and the other derived therefrom by means of an accepted relation. In the United States the yard is defined in terms of the meter, and the pound in terms of the kilogram. There is in the United States no primary standard either of length or mass in the customary system.

The use of metric units in certain athletic events in this country is undoubtedly of considerable interest to many people. Initial action by the Amateur Athletic Union was taken in November 1932, when it adopted metric distances for track events to be run in athletic meets held under the jurisdiction of that organization. Metric units for track and field events were adopted by various athletic organizations but this movement soon began to lose ground. In 1951 the use of metric distances in track and field events on national championship programs held under AAU auspices was restricted to Olympic years.

e. Arguments For and Against the Metric System

That there are arguments for and against the metric system is evidenced by the rather voluminous literature on the subject.

The National Bureau of Standards neither advocates nor opposes the compulsory adoption of the metric system. Those desiring arguments in favor of its more general adoption are referred to the Metric Association, J. T. Johnson, President, 2025 Silver Tip Lane, Long Beach, Michigan City, Ind. Those desiring arguments against it are referred to The American Institute of Weights and Measures, W. R. Ingalls, President, Georgetown, Mass.

The book compiled by Julia Emily Johnson entitled "Metric System" and published in 1926 by The H. W. Wilson Co., New York, N. Y., contains a bibliography and other material useful for debates.

2.3. British and United States Systems of Weights and Measures

The implication is sometimes made that the systems of weights and measures in general use in the British Empire and those in general use in the United States are identical. It is true that the difference between the U. S. and the British inch is not significant except in a few cases of the most refined measurements, that the British and the U. S. pound may be considered practically identical, and that many tables such as 12 inches=1 foot, 3 feet=1 yard, and 1 760 yards=1 mile are the same in both countries; but there are some very important differences.

In the first place, the U. S. bushel and the U. S. gallon, and their subdivisions differ from the corresponding British units. Also the British ton is 2 240 pounds, whereas the ton generally used in the United States is the short ton of 2 000 pounds. The American colonists adopted the English wine gallon of 231 cubic inches. The English of that period used this wine gallon and they also had another gallon, the ale gallon of 282 cubic inches. In 1824 these two gallons were abandoned by the British when they adopted the British Imperial gallon, which is defined as the volume of 10 pounds of water, at a temperature of 62° F which, by calculation, is equivalent to 277.42 cubic inches. At the same time, the bushel was redefined as 8 gallons. In the British system the units of dry measure are the same as those of liquid measure. In the United States these two are not the same, the gallon and its subdivisions being used in the measurement of liquids, while the bushel, with its subdivisions, is used in the measurement of certain dry commodities. The U. S. gallon is divided into 4 liquid quarts and the U. S. bushel into 32 dry quarts. All the units of capacity mentioned thus far are larger in the British system than in the U. S. system. But the British fluid ounce is smaller than the U. S. fluid ounce, because the British quart is divided into 40 fluid ounces whereas the U. S. quart is divided into 32 fluid ounces.

From the foregoing it is seen that in the British system an avoirdupois ounce of water at 62° F has a volume of 1 fluid ounce, because 10 pounds is equivalent to 160 avoirdupois ounces, and 1 gallon is equivalent to 4 quarts, or 160 fluid ounces. This convenient relation does not exist in the U. S. system because a U. S. gallon of water at 62° F weighs about 8½ pounds, or 133½ avoirdupois ounces, and the U. S. gallon is equivalent to 4×32, or 128 fluid ounces.

1 U. S. fluid ounce = 1.040 8 British fluid ounces.

1 British fluid ounce = 0.960 8 U. S. fluid ounce.

Among other differences between the British and the American systems of weights and measures it should be noted that the use of the troy pound was abolished in England January 6, 1879, only the troy ounce and its subdivisions being retained, whereas the troy pound is still legal in the United States, although it is not now greatly used. The common use in England of the stone of 14 pounds should be mentioned, this being a unit now unused in the United States, although its influence was shown in the practice until World War II of selling flour by the barrel of 196 pounds (14 stones). In the apothecaries system of liquid measure the British insert a unit, the fluid scruple, equal to one third of a fluid drachm (spelled *dram* in the United States) between their minim and their fluid drachm. In the United States the general practice now is to sell dry commodities, such as fruits and vegetables, by weight.

2.4. Subdivision of Units

In general, units are subdivided by one of three systems: (a) decimal, that is into tenths; (b) duodecimal, into twelfths; or (c) binary, into halves. Usually the subdivision is continued by the use of the same system. Each method has its advantages for certain purposes and it cannot properly be said that any one method is "best" unless the use to which the unit and its subdivisions are to be put is known.

For example, if we are concerned only with measurements of length to moderate precision, it is convenient to measure and to express these lengths in feet, inches, and binary fractions of an inch, thus 9 feet 4 $\frac{3}{8}$ inches. If, however, these measured lengths are to be subsequently used in calculations of area or volume, that method of subdivision at once becomes extremely inconvenient. For that reason civil engineers, who are concerned with areas of land, volumes of cuts, fills, excavations, etc., instead of dividing the foot into inches and binary subdivisions of the inch, divide it decimally, that is, into tenths, hundredths, and thousandths of a foot.

The method of subdivision of a unit is thus largely made on the basis of convenience to the user. The fact that units have commonly been subdivided into certain subunits for centuries does not preclude these units also having another mode of subdivision in some frequently used cases where convenience indicates the value of such other method. Thus the gallon is usually subdivided into quarts and pints, but the majority of gasoline-measuring pumps of the price-computing type are graduated to show tenths of a gallon. Although the mile has for centuries been divided into rods, yards, feet, and inches, the odometer part of an automobile speedometer indicates tenths of a mile. Although our dollar is divided into 100 parts, we habitually use and speak of halves and quarters. An illustration of rather complex subdividing is found on the scales used by draftsmen. These scales are of two types: (a) architects, which are commonly graduated with scales in which $\frac{3}{32}$, $\frac{3}{16}$, $\frac{1}{8}$, $\frac{1}{4}$, $\frac{3}{8}$, $\frac{1}{2}$, $\frac{3}{4}$, 1, 1 $\frac{1}{2}$, and 3 inches, respectively, represent 1 foot full scale, as well as having a scale graduated in the usual manner to $\frac{1}{16}$ inch; and (b) engineers, which are commonly subdivided to 10, 20, 30, 40, 50, and 60 parts to the inch.

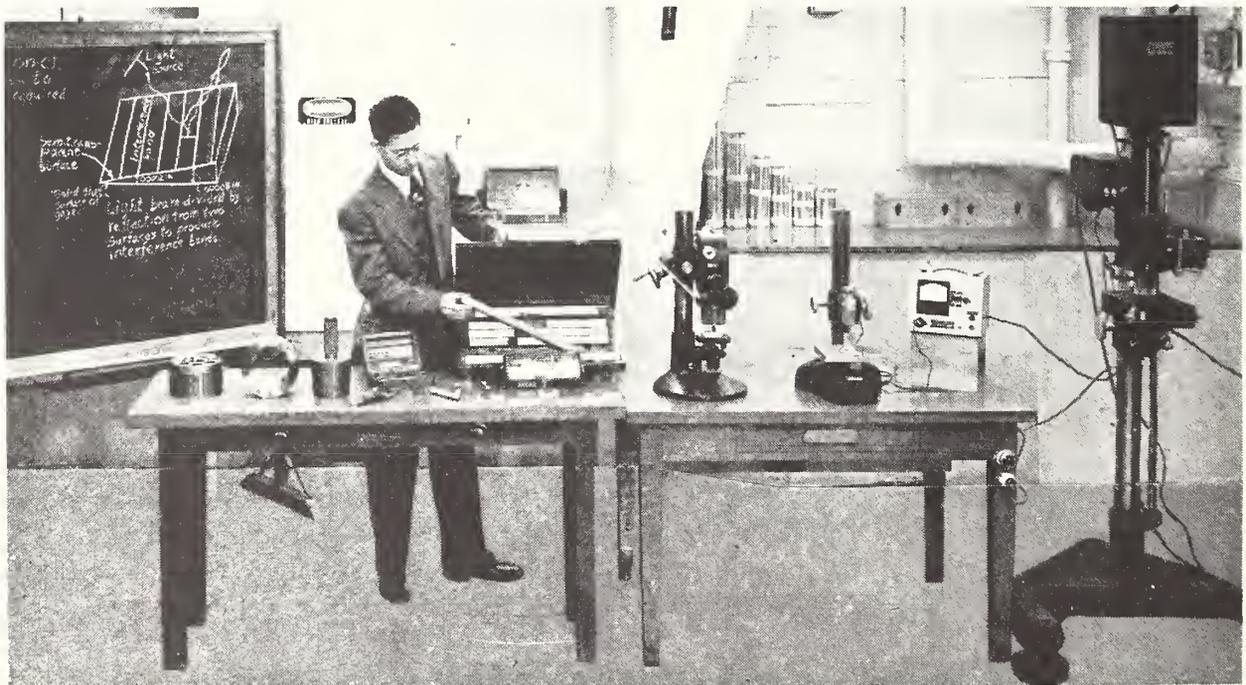


FIGURE 2. *Gage block testing*

Proper fitting and functioning of interchangeable mechanical parts depend upon precisely calibrated gage blocks. The NBS checks master gage blocks of industry. Shown on the table are various gage blocks and ring gages used by industry along with some of the equipment used by NBS for this calibration (see page 8).

The dictum of convenience applies not only to subdivisions of a unit but also to multiples of a unit. Elevations of land above sea level are given in feet even though the height may be several miles; the height of aircraft above sea level as given by an altimeter is likewise given in feet, no matter how high it may be.

On the other hand, machinists, toolmakers, gage makers, scientists, and others who are engaged in precision measurements of relatively small distances, even though concerned with measurements of length only, find it convenient to use the inch, instead of the tenth of a foot, but to divide the inch decimally to tenths, hundredths, thousandths, etc., even down to millionths of an inch. Verniers, micrometers, and other precision measuring instruments are usually graduated in this manner. Machinist scales are commonly graduated decimally along one edge and are also graduated along another edge to binary fractions as small as $\frac{1}{64}$ inch. The scales with binary fractions are used only for relatively rough measurements.

It is seldom convenient or advisable to use binary subdivisions of the inch that are smaller than $\frac{1}{64}$. In fact, $\frac{1}{32}$ -, $\frac{1}{16}$ -, or $\frac{1}{8}$ -inch subdivisions are usually preferable for use on a scale to be read with the unaided eye.

2.5. Arithmetical Systems of Numbers

The subdivision of units of measurement is closely associated with arithmetical systems of numbers. The systems of weights and measures used in this country for commercial and scientific work, having many origins as has already been shown, naturally show traces of the various number systems associated with their origins and developments. Thus (a) the binary subdivision has come down to us from the Hindus, (b) the duodecimal system of fractions from the Romans, (c) the decimal system from the Chinese and Egyptians, some developments having been made by the Hindus, and (d) the sexagesimal system (division by 60), now illustrated in the subdivision of units of angle and of time, from the ancient Babylonians.

The suggestion is made from time to time that we should adopt a duodecimal number system and a duodecimal system of weights and measures. Another suggestion is for an octonary number system (a system with 8 as the basis instead of 10 in our present system or 12 in the duodecimal) and an octonary system of weights and measures. Such suggestions have certain theoretical merits, but are very impractical because it is now too late to modify our number system and unwise to have arbitrary enforcement of any single system of weights and measures. It is far better for each branch of science, industry, and commerce to be free to use whatever system has been found by experience best to suit its needs. The prime requisite of any system of weights and measures is that the units be definite. It is also important that the relations of these units to the units of other systems be definite, convenient, and known, in order that conversion from one system to another may be accurately and conveniently made.

3. Standards of Length, Mass, Time, and Capacity

3.1. Standards of Length

The primary standard of length in the United States is the United States Prototype Meter 27, a platinum-iridium (90% platinum, 10% iridium) line standard having an X-shaped cross section. The length of this bar, which is deposited at the National Bureau of Standards in Washington, is known in terms of the International Prototype Meter at the International Bureau of Weights and Measures at Sèvres, near Paris, France.

The U. S. yard is defined, following the policy stated in the Mendenhall Order¹ of April 5, 1893, as follows:

$$1 \text{ U. S. yard} = \frac{3}{3937} \frac{600}{937} \text{ meter.}$$

¹ This order stated that the Office of Weights and Measures, with the approval of the Secretary of the Treasury, would in the future regard the International Prototype Meter and Kilogram as fundamental standards, and that the customary units would be derived therefrom in accordance with the Act of July 28, 1866.

The relation 1 U. S. yard = $\frac{3\ 600}{3\ 937}$ meter, derived from the Law of 1866 that made the use of the metric system legal in the United States, was confirmed by later comparisons of copies of the British yard with the U. S. national copies of the meter. Since the Mendenhall Order it has been used as an exact relation. From this it follows that 1 U. S. inch is slightly larger than 0.025 400 05 meter, or 25.400 05 millimeters.

For industrial purposes a relation between the yard and the meter has been adopted by the American Standards Association (ASA B48.1-1933), and by similar organizations in 15 other countries. This relation is

$$1\ \text{inch} = 25.4\ \text{millimeters (exactly), that is, } 0.025\ 4\ \text{meter (exactly),}$$

from which 1 yard = 0.914 4 meter (exactly), or 914.4 millimeters (exactly).

The adoption of this relation by industry, for use in making conversions between inches and millimeters, did not change the official definition of the yard or of the meter. Its legal adoption in the United States and in Great Britain would be a desirable step in the direction of international uniformity in precision length measurements.

In Great Britain the Imperial Yard is represented by a yard bar made of bronze in 1844. The relation between that yard and the meter according to the most recent published determinations is

$$1\ \text{British Imperial Yard} = \frac{3\ 600\ 000}{3\ 937\ 014}\ \text{meter.}$$

Although opinions about the British yard standard are conflicting, there is substantial evidence that the British yard has shortened by a few parts in a million during the past century. Uncertainties in some of the measurements and in the thermometric scale of the early compari-

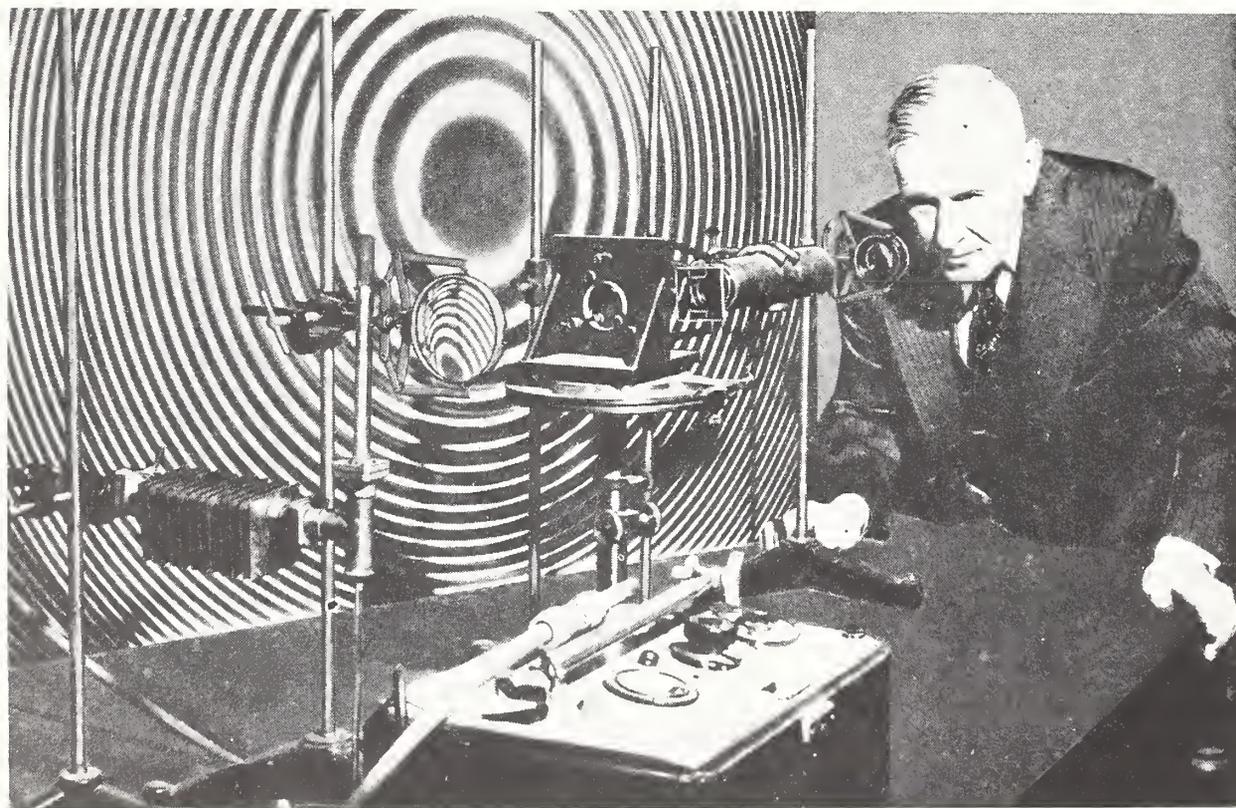


FIGURE 3. Mercury 198 lamp.

William F. Meggers positions the eyepiece of an optical train prior to observation of the circular interference fringes of the green light from a mercury 198 lamp. This electrodeless lamp contains about 1 milligram of mercury of atomic weight 198. The lamp is excited in a radiofrequency field (left foreground). The interference pattern portrayed in the background enables researchers to make accurate length measurements. The NBS-Meggers Mercury 198 lamp is one of the light sources proposed for use in defining length by reference to a specified wavelength of light.

sons, however, make it impossible to state the difference between the U. S. and the British yards a century ago or to be certain of the amount that the British Imperial Yard has changed since that date.

In 1927 the Seventh General (International) Conference on Weights and Measures approved a resolution stating the wavelength of the red cadmium radiation under standard conditions of temperature, pressure, and humidity to be 0.000 643 846 96 millimeter. From this the length of the meter in terms of the wavelength of light was provisionally expressed as equal to 1 553 164.13 wavelengths of cadmium light under the specified conditions.

With the advances made in physics in recent years better sources of monochromatic radiation for use as wavelength standards of length have been developed. The National Bureau of Standards has proposed that the wavelength of the green radiation of the 198 isotope of mercury (fig. 3) be adopted as the fundamental standard of length instead of the International Prototype Meter. The change proposed is in the standard; the unit would not be altered. Other laboratories have proposed other radiations, and investigations are now in progress in a number of national laboratories in various countries to determine what length standard will be the most stable and the most suitable for the purpose. It is believed that a natural phenomenon such as monochromatic radiation will be more unchangeable than a metal standard, even though no change in length of the international platinum-iridium bar seems to be detectable.

a. Tests and Calibrations of Length Standards

The National Bureau of Standards tests standards of length including meter bars, yard bars, miscellaneous precision line standards, steel tapes, invar geodetic tapes (see fig. 4), precision gage blocks, micrometers, and limit gages. It also measures the linear dimensions of miscellaneous apparatus such as penetration needles, cement sieves, and haemocytometer chambers. In general the Bureau accepts for test only apparatus of such material, design, and construction as to ensure accuracy and permanence sufficient to justify test by the Bureau. Tests are made in accordance with test-fee schedules, copies of which may be obtained by application to the Bureau.

The Bureau does not test carpenters rules, machinists scales, draftsmans scales, and the like. Such apparatus, if test is required, should be submitted to State or local weights and measures officials. NBS Circular 572, Calibration of Line Standards of Length and Measur-

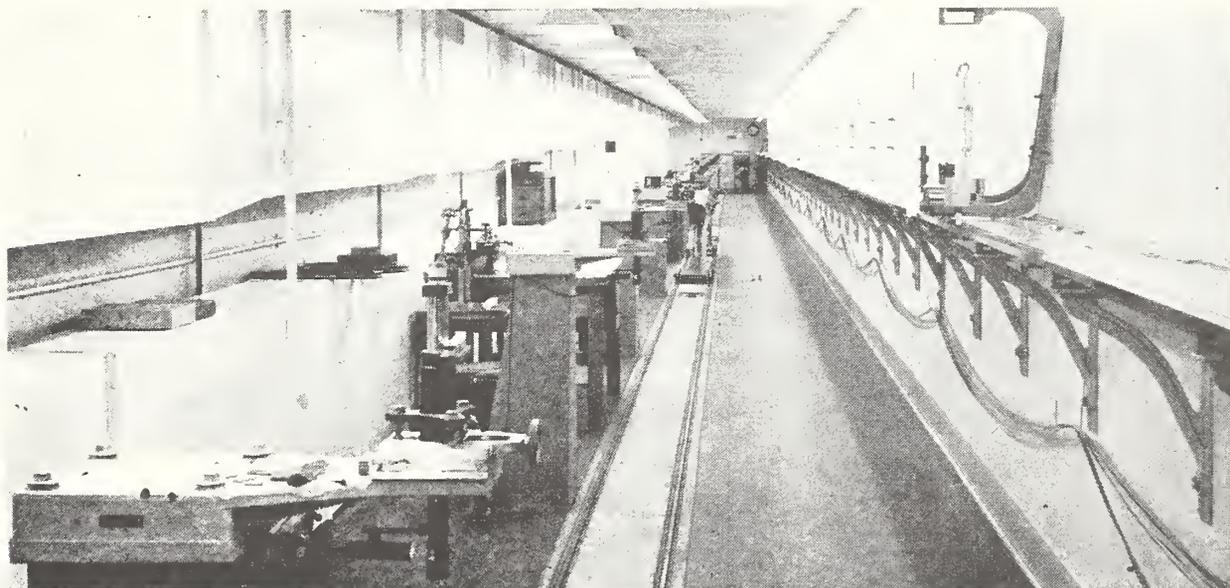


FIGURE 4. NBS Tape Calibration Laboratory.

In this laboratory the Bureau calibrates steel tapes used by surveyors and engineers and the more precise base-line tapes used by the U. S. Coast and Geodetic Survey.

ing Tapes at the National Bureau of Standards, by Lewis V. Judson (in press) contains additional information on this subject.

3.2. Standards of Mass

The primary standard of mass for this country is United States Prototype Kilogram 20, which is a platinum-iridium standard kept at the National Bureau of Standards. The value of this mass standard is known in terms of the International Prototype Kilogram, a platinum-iridium standard which is kept at the International Bureau of Weights and Measures.

For many years the British standards were considered to be the primary standards of the United States. Later, for over 50 years, the U. S. avoirdupois pound was defined in terms of the Troy Pound of the Mint, which is a brass standard kept at the United States Mint in Philadelphia. In 1911 the Troy Pound of the Mint was superseded, for coinage purposes, by the Troy Pound of the National Bureau of Standards. Since 1893 the avoirdupois pound has been defined in terms of the United States Prototype Kilogram 20 by the relation:

$$1 \text{ avoirdupois pound} = 0.453\,592\,427\,7 \text{ kilogram.}$$

Insofar as can be determined, these changes in definition have not made any change in the actual value of the pound.

The grain is $1/7\,000$ of the avoirdupois pound and is identical in the avoirdupois, troy, and apothecaries systems. The troy ounce and the apothecaries ounce differ from the avoirdupois ounce but are equal to each other, and equal to 480 grains. The avoirdupois ounce is equal to $437\frac{1}{2}$ grains.

In Great Britain the Imperial Pound, an avoirdupois pound, is represented by a physical standard made in 1845. According to the most recent published determination

$$1 \text{ British Imperial Pound} = 0.453\,592\,34 \text{ kilogram.}$$

There is substantial evidence that the British standard has diminished by about 1 part in 5 million since 1883 in relation to the kilogram, and is therefore smaller than the U. S. pound by that amount.

a. Distinction Between Mass and Weight

The *mass* of a body is a measure of the quantity of material in the body. The *weight* of a body is defined as the force with which that body is attracted toward the earth. Confusion sometimes arises from the practice of referring to standards of mass as "weights" and from the fact that such standards are compared by "weighing" one against another by means of a balance. Standard "weights" are, in reality, standards of mass.

Another practice which tends to confusion is that of using the terms kilogram, gram, pound, etc., in two distinct senses; first, to designate units of mass, and second, to designate units of weight or force. For example, a body having a mass of one kilogram is called a kilogram (mass) and the force with which such a body is attracted toward the earth is also called a kilogram (force).

The International Kilogram and the U. S. Prototype Kilogram are specifically defined by the International Conference on Weights and Measures as standards of mass. The U. S. pound, which is derived from the International Kilogram, is, therefore, a standard of mass.

So long as no material is added to or taken from a body its mass remains constant. Its weight, however, varies with the acceleration of gravity, g . For example, a body would be found to weigh more at the poles of the earth than at the equator, and less at high elevations than at sea level. (Standard acceleration of gravity, adopted by the International Committee on Weights and Measures in 1901 is 980.665 cm/sec^2 . This value corresponds nearly to the value at latitude 45° and sea level.)

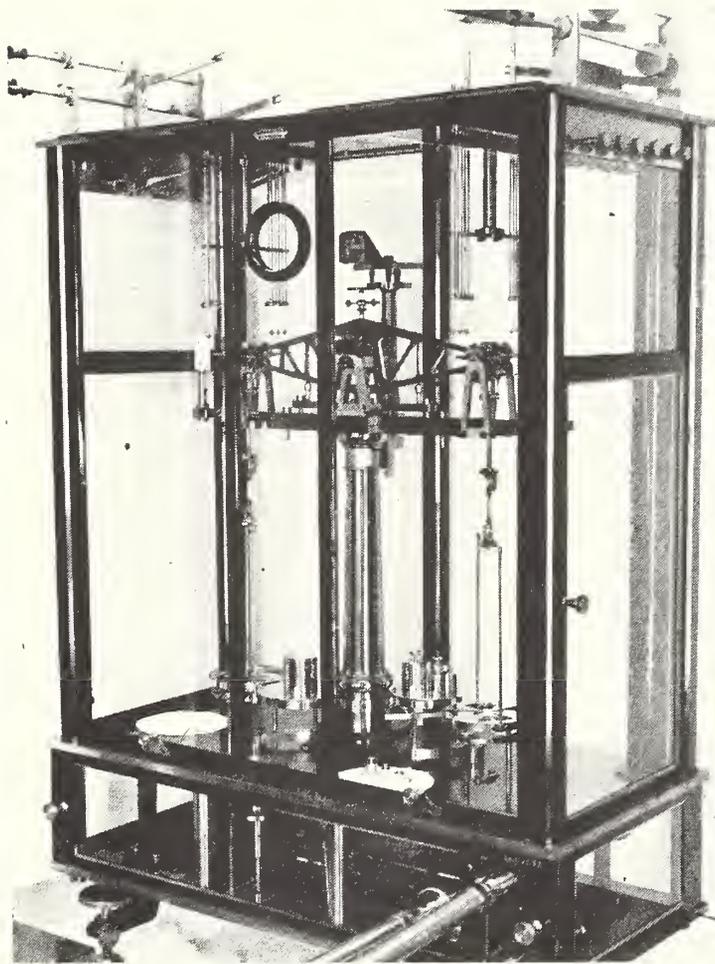


FIGURE 5. *Precision balance.*

The most accurate of the precision balances at NBS is used to compare secondary and laboratory standards of mass with the national prototype kilogram. This balance is operated by remote control in order to avoid errors caused by the heat given off by the body of the observer. By means of rods (above and lower right) the balance pan is arrested and released, the loads on the pans are interchanged, and sensitivity weights are added. A precision better than 1 part in 100 000 000 is obtained.

Because standards of mass (or "weights") are ordinarily calibrated and used on equal-arm balances (fig. 5), the effects of variations in the acceleration of gravity are self-eliminating and need not be taken into account. Two objects of equal mass will be affected in the same manner and by the same amount by any change in the value of the acceleration of gravity, and thus if they have the same weight, i. e., if they balance each other on an equal-arm balance, under one value of g , they will also balance each other under any other value of g .

On a spring balance, however, the weight of the body is not balanced against the weight of another body, but against the resistance of a spring. Therefore, using a very sensitive spring balance, the weight of a body would be found to change if the spring balance and the body were moved from one locality to another locality with a different acceleration of gravity. But a spring balance is usually used in one locality and is adjusted to indicate mass at that locality.

b. Effect of Air Buoyancy

Another point that must be taken into account in the calibration and use of standards of mass is the buoyancy or lifting effect of the air. A body immersed in any fluid is buoyed up by a force equal to the weight of the displaced fluid. Two bodies of equal mass, if placed one on each pan of an equal-arm balance, will balance each other in a vacuum. A comparison in a vacuum against a known mass standard gives "true mass." If compared in air, however, they will not balance each other unless they are of equal volume. If of unequal volume, the

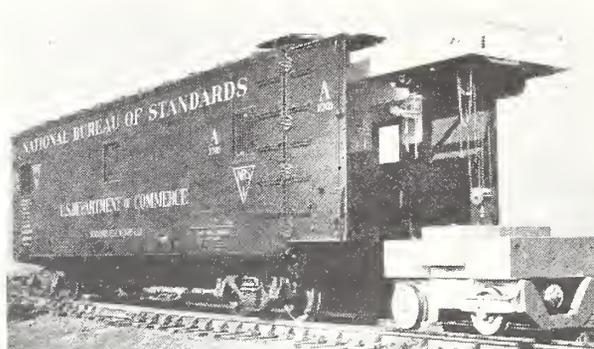
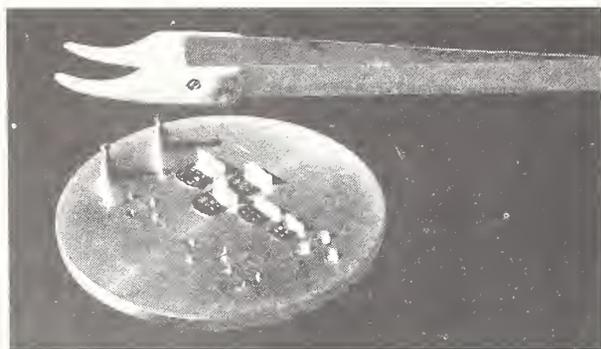


FIGURE 6. *Calibration of weights by NBS.*

These two illustrations indicate extremes of weights routinely calibrated by NBS. The one on left shows the small weights (down to 0.05 mg) for use with microbalances. The illustration on right shows 1 of 2 standard test weight cars, owned and operated by NBS for calibrating and adjusting the master railway track scales of the Nation's railroads. The largest individual weight of these cars is 10 000 pounds. A total test load of 80 000 pounds is carried by each car.

larger body will displace the greater volume of air and will be buoyed up by a greater force than will the smaller body, and the larger body will appear to be lighter in weight than the smaller body. The greater the difference in volume, and the greater the density of the air in which the comparison weighing is made, the greater will be the apparent difference in weight. For that reason, in assigning a precise numerical value of apparent mass to a standard, it is necessary to base this value on definite values for the air density and the density of the mass standard of reference.

The corrections furnished by the National Bureau of Standards for the more precise mass standards are given both (a) on the basis of comparison in vacuum, and (b) on the basis of comparison against normal brass standards in air under standard conditions, with no correction applied for the buoyant effect of the air. Normal brass standards are defined as having a density of 8.4 grams per cubic centimeter at 0° C and a coefficient of cubical thermal expansion of 0.000 054 per deg C. Standard conditions are defined as air of 1.2 milligrams per cubic centimeter and temperature of 20° C. The corrections to be used with precise analytical weights are ordinarily given only in terms of apparent mass against normal brass standards.

c. Tests of Standards of Mass

Weights regularly used in ordinary trade and industry should be tested by State or local weights and measures officials. The National Bureau of Standards calibrates and certifies the values of weights submitted but it does not manufacture or sell weights. Information regarding the various classes of weights, the requirements for each class, the weight-calibration service of the Bureau and the regulations governing the submission of weights to NBS for test are contained in NBS Circular 547, section 1, Precision Laboratory Standards of Mass and Laboratory Weights, by T. W. Lashof and L. B. Macurdy (for sale by the Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C., at 25 cents a copy).

3.3. Standards of Time

There is no physical standard of time corresponding to the standards of length and mass. Time is measured in terms of the motion of the earth; (a) on its axis, and (b) around the sun. The time it takes the earth to make a complete rotation on its axis is called a day, and the time it takes it to make a complete journey around the sun, as indicated by its position with reference to the stars, is called a year. The earth makes about $365\frac{1}{4}$ rotations on its axis ($365.242\ 2$, more exactly) while making a complete journey around the sun. In other words, there are almost exactly $365\frac{1}{4}$ solar days in a tropical or solar year. As it would be inconvenient and confusing to have the year, as used in everyday life, contain a fractional part of a day, fractional days are avoided by making the calendar year contain 365 days in ordinary years

and 366 days in leap years. The frequency of occurrence of leap years is such as to keep the average length of the calendar year as nearly as practicable equal to that of the tropical year, in order that calendar dates may not drift through the various seasons of the tropical year.

The earth, in its motion around the sun, does not move at a uniform speed, and the sun in its apparent motion does not move along the equator but along the ecliptic. Therefore the apparent solar days are not of exactly equal length. To overcome this difficulty time is measured in terms of the motion of a fictitious or "mean" sun, the position of which, at all times, is the same as would be the apparent position of the real sun if the earth moved on its axis and in its journey around the sun at a uniform rate. Ordinary clocks and watches are designed and regulated to indicate time in terms of the apparent motion of this fictitious or "mean sun." It is "mean noon" when this "mean sun" crosses the meridian, and the time between two successive crossings is a "mean solar day." The length of the mean solar day is equal to the average length of the apparent solar day.

In observing on the stars, the time generally used by astronomers is sidereal time. This is defined by the rotation of the earth with respect to the stars. A sidereal day is the interval between two successive passages of a star across a meridian. The sidereal day is subdivided into hours, minutes, and seconds, the hours being numbered from 1 to 24. The sidereal year is 365.256 36 solar days.

The mean solar day is divided into 24 hours, each hour into 60 minutes, and each minute into 60 seconds. Thus the mean solar second is $1/864\ 00$ of a mean solar day, and this mean solar second is the unit in which short time intervals are measured and expressed.

The time at which the "mean sun" crosses the meridian at any point on the earth is known as "local mean noon." As it would be impracticable to use local mean time at each locality, the surface of the earth, by international agreement, has been divided into 24 standard time zones, each zone having a width of approximately 15 degrees of longitude. In each zone the time used is that corresponding to the meridian passing approximately through its center, and adjacent zones have a time difference of 1 hour.

The meridian passing through Greenwich, England, is taken as the standard, or prime meridian, and time throughout the world is reckoned with reference to the time at Greenwich. Each 15 degrees east or west from Greenwich corresponds to a time difference of 1 hour. There are a few exceptions to the above rule. East of Greenwich the time is faster, and west of Greenwich it is slower than at Greenwich.

The United States is divided into four time zones in which time is designated as Eastern, Central, Mountain, and Pacific. The time in these zones is slower than Greenwich time by 5,

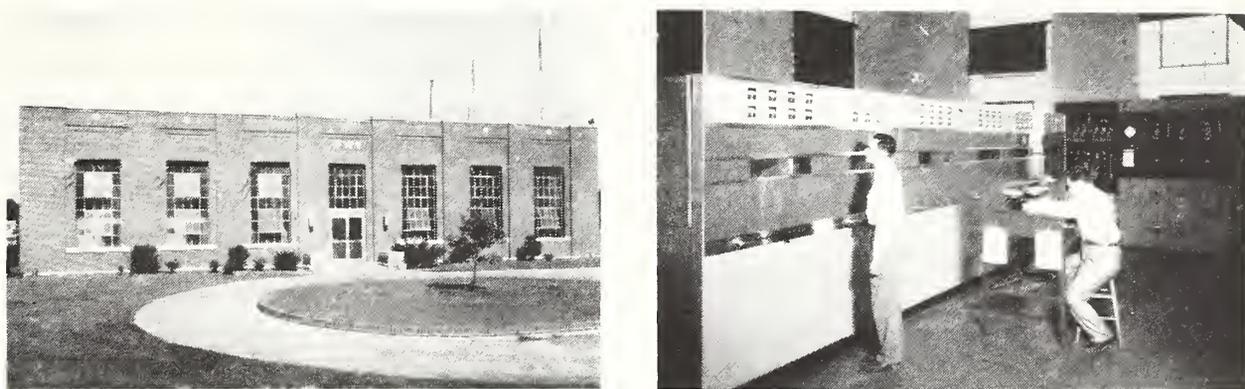


FIGURE 7. *Radio station WWV in Beltsville, Md.*

NBS broadcasts various standard radiofrequency and time signals, day and night, from station WWV. These signals—accurate to 1 part in 50 000 000—are used by utilities to control 60-cycle generators, by manufacturers of electronic equipment to calibrate oscillators, by radio stations to keep their signals within assigned channels, by aircraft and ships, and by scientists and engineers in experimental work. WWV services—including the standard musical pitch (440 cycles per second), precise time intervals (seconds), and time announcements in voice—can be picked up by shortwave receiver at 5, 10, 15, 20, and 25 megacycles per second.

6, 7, and 8 hours, respectively. For further information see NBS Circular 496, *Standard Time Throughout the World*, by R. E. Gould (for sale by the Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C., at 15 cents a copy).

The U. S. Naval Observatory has elaborate transit equipment with which it measures the time of rotation of the earth with respect to various heavenly bodies. The averaged time of rotation over a period of several days is used as a standard interval to which is compared the interval indicated by extremely accurate crystal clocks owned by the National Bureau of Standards. The time difference between these two intervals is published as a correction to the crystal clock time signals transmitted continuously by the National Bureau of Standards broadcasting station WWV (fig. 7) on 2.5, 5, 10, 15, 20, and 25 megacycles per second. The National Bureau of Standards time signal is accurate to one-millionth of a second over a 1-second interval, and accurate to 1 part in 50 million for intervals of 1 minute or longer. This signal provides an indispensable standard time interval for purposes of physical measurements, for quick and accurate calibration of timing devices, and for adjustment of very low frequency oscillators.

3.4. Standards of Capacity

Units of capacity, being derived units, in this country defined in terms of linear units, are not represented by fundamental standards. Laboratory standards have been constructed and are maintained at the National Bureau of Standards. These have validity only by calibration with reference either directly or indirectly with the linear standards. Similarly, standards of capacity have been made and distributed to the several States. Other standards of capacity have been verified by calibration for a wide variety of uses in science, technology, and commerce.



FIGURE 8. *Calibration of a capacity standard at NBS.*

A portable cubic foot standard of volume (left) for field use in the fuel gas industry is being calibrated under laboratory conditions at NBS by comparison with a laboratory standard immersion-type cubic-foot bottle (right center).

a. Tests of Standards of Capacity

Calibrations are made by the Bureau on capacity standards that are in the customary units of trade, that is the gallon, its multiples, and submultiples, or in metric units. Furthermore the Bureau calibrates precision grade volumetric glassware which is normally in metric units. Tests are made in accordance with test-fee schedules, copies of which may be obtained by application to the Bureau.

3.5. Maintenance and Preservation of Fundamental Standards of Length and Mass

There is considerable interest in the maintenance and preservation of the national standards of length and mass at the National Bureau of Standards. In 1955, a special glass door, fully protected by an alarm system, was installed so that during the regular working hours of the Bureau the vault can be viewed by those interested. At other times the steel outer doors are locked. All measurements made with these standards are conducted in special air-conditioned laboratories to which the standards are taken a sufficiently long time before the observations to ensure that the standards will be in a state of equilibrium under standard conditions when the measurements or comparisons are made. Hence it is not necessary to maintain the vault at standard conditions, but care is taken to prevent large changes of temperature. More important is the care to prevent any damage to the standards because of careless handling.

4. Weights and Measures in Everyday Life

As weighing and measuring are important factors in our everyday lives, it is quite natural that questions arise about the use of various units and terms and about the magnitude of quantities involved. Only two items will be considered here, first the weight of coal, and second the definitions and usages of the terms "ton" and "tonnage."

4.1. Weight of Coal

Questions are frequently asked about the weight per unit volume of coal. As there are large variations in the weight per cubic foot of coal, the reader is cautioned that the figures presented herein are only approximate values, and that in the case of any particular delivery of coal, the actual number of cubic feet per ton (of 2 000 pounds), may differ materially from the values given here.

The following values may, however, be satisfactory for use in calculating the approximate size of bin required to contain a given number of tons of coal, the approximate number of tons of coal that a given bin will contain, and the approximate weight of a measured amount of coal. Relatively large shortages can be detected by computing the weights of deliveries, and computed weights may properly be used by a purchaser as a basis of complaint to the weights and measures official; such evidence alone, however, probably would not be accepted by a court, and satisfactory evidence can be procured only by actually weighing the coal comprising a delivery.

The weight per cubic foot of anthracite (hard coal) varies with the size into which the coal is broken, and with the kind of coal or the vein from which the coal comes. According to information published by the Anthracite Institute, "frequently used average weight and volume figures for all sizes of anthracite are 37 cubic feet per ton and 54 lbs per cubic feet." This corresponds to 67 pounds per stricken bushel (2 150.42 cubic inches). Variations from these averages as high as 10 percent may be expected.

The weight of bituminous (common soft) coal also varies according to the locality from which the coal comes. Such weights range from 47 to 55 pounds per cubic foot. These values correspond to 42.6 to 36.4 cubic feet per 2 000-pound ton; and to 58.5 to 68.4 pounds per stricken bushel.

4.2. Definitions and Usages of the Terms "Ton" and "Tonnage"

Because the words "ton" and "tonnage" are used in widely different senses, a great deal of confusion has arisen regarding the application of these terms.

a. Definitions and Uses of "Ton"

The ton is used as a unit of measure in two distinct senses: (1) as a unit of weight, and (2) as a unit of capacity or volume.

In the first sense the term has the following meanings:

- (a). The *short*, or *net* ton of 2 000 pounds.
- (b). The *long*, *gross*, or *shipper's* ton of 2 240 pounds.
- (c). The *metric* ton of 1 000 kilograms, or 2 204.6 pounds.

In the second sense (capacity) it is usually restricted to uses relating to ships and has the following meaning:

- (a). The *register* ton of 100 cubic feet.
- (b). The *measurement* ton of 40 cubic feet.
- (c). The *English water* ton of 224 British Imperial gallons.

In the United States and Canada the ton (weight) most commonly used is the *short* ton, in Great Britain it is the *long* ton, and in countries using the metric system it is the *metric* ton. The *register* ton and the *measurement* ton are capacity units used in expressing the tonnage of ships. The *English water* ton is used, chiefly in Great Britain, in statistics dealing with petroleum products.

There have been many other uses of the term ton such as the timber ton of 40 cubic feet and the wheat ton of 20 bushels, but their use has been local and the meanings have not been consistent from one place to another.

b. Definitions and Uses of "Tonnage"

Properly, the word "tonnage" is used as a noun only in respect to the capacity and dimensions of ships, and to the amount of the ship's cargo. There are two distinct kinds of tonnage, namely, *vessel tonnage* and *cargo tonnage* and each of these is used in various meanings.

The several kinds of *vessel tonnage* are as follows:

Gross tonnage, or *gross register tonnage*, is the total cubical capacity of a ship expressed in *register* tons of 100 cubic feet, or 2.83 cubic meters, less such space as hatchways, bakeries, galleys, etc., as are exempted from measurement by different governments. There is some lack of uniformity in the gross tonnages as given by different nations on account of lack of agreement on the spaces that are to be exempted.

Official merchant marine statistics of most countries are published in terms of the *gross register tonnage*. Press references to *ship tonnage* are usually to the *gross tonnage*.

The *net tonnage*, or *net register tonnage*, is the *gross tonnage* less the different spaces specified by maritime nations in their measurement rules and laws. The spaces that are deducted are those totally unavailable for carrying cargo, such as the engine room, coal bunkers, crews quarters, chart and instrument room, etc.

The *net tonnage* is used in computing the amount of cargo that can be loaded on a ship. It is used as the basis for wharfage and other similar charges.

The *register under-deck tonnage* is the cubical capacity of a ship under her tonnage deck expressed in register tons. In a vessel having more than one deck the tonnage deck is the second from the keel.

There are several variations of *displacement tonnage*.

The *dead weight tonnage* is the difference between the "loaded" and "light" *displacement tonnages* of a vessel. It is expressed in terms of the long ton of 2 240 pounds, or the metric ton of 2 204.6 pounds, and is the weight of fuel, passengers, and cargo that a vessel can carry when loaded to her maximum draft.

The second variety of tonnage, *cargo tonnage*, refers to the weight of the particular items making up the cargo. In overseas traffic it is usually expressed in long tons of 2 240 pounds or metric tons of 2 204.6 pounds. The short ton is only occasionally used. The *cargo tonnage* is therefore very distinct from *vessel tonnage*.

5. General Tables of Weights and Measures

These tables have been prepared for the benefit of those requiring tables of weights and measures for occasional ready reference. In section 5.4 the tables are carried out to a large number of decimal places and exact values are indicated by boldface type. In most of the other tables only a limited number of decimal places are given, thus making the tables better adapted to the average user. More extensive tables will be found in Miscellaneous Publication M214 of the National Bureau of Standards, Units of Weight and Measure—Definitions and Tables of Equivalents (sold by the Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C., at 40 cents a copy).

5.1. Tables of United States Customary Weights and Measures

LINEAR MEASURE

12 inches (in.)	= 1 foot (ft).
3 feet	= 1 yard (yd).
5½ yards	= 1 rod (rd), pole, or perch = 16½ feet.
40 rods	= 1 furlong (fur.) = 220 yards = 660 feet.
8 furlongs	= 1 statute mile (mi) = 1 760 yards = 5 280 feet.

6 076.103 33 . . . feet (1 852 meters) = 1 international nautical mile. This value was adopted effective July 1, 1954, for use in the United States. The value formerly used in the United States was 6 080.20 feet = 1 nautical (geographical or sea) mile.

AREA MEASURE ^a

144 square inches (sq in.)	= 1 square foot (sq ft).
9 square feet	= 1 square yard (sq yd) = 1 296 square inches.
30¼ square yards	= 1 square rod (sq rd) = 272¼ square feet.
160 square rods	= 1 acre = 4 840 square yards = 43 560 square feet.
640 acres	= 1 square mile (sq mi).
1 mile square	= 1 section of land.
6 miles square	= 1 township = 36 sections = 36 square miles.

CUBIC MEASURE ^a

1 728 cubic inches (cu in.)	= 1 cubic foot (cu ft).
27 cubic feet	= 1 cubic yard (cu yd).

GUNTER'S OR SURVEYORS CHAIN MEASURE

7.92 inches (in.)	= 1 link (li).
100 links	= 1 chain (ch) = 4 rods = 66 feet.
80 chains	= 1 statute mile (mi) = 320 rods = 5 280 feet.

LIQUID MEASURE ^b

4 gills (gi)	= 1 pint (pt) = 28.875 cubic inches.
2 pints	= 1 quart (qt) = 57.75 cubic inches.
4 quarts	= 1 gallon (gal) = 231 cubic inches = 8 pints = 32 gills.

^a Squares and cubes of units are sometimes abbreviated by using "superior" figures. For example, ft² means square foot, and ft³ means cubic foot.

^b When necessary to distinguish the *liquid* pint or quart from the *dry* pint or quart, the word "liquid" or the abbreviation "liq" should be used in combination with the name or abbreviation of the *liquid* unit.

APOTHECARIES FLUID MEASURE

60 minims (min or ℥)	= 1 fluid dram (fl dr or ℥ $\bar{5}$) = 0.225 6 cubic in.
8 fluid drams	= 1 fluid ounce (fl oz or ℥ $\bar{5}$) = 1.804 7 cubic inches.
16 fluid ounces	= 1 pint (pt or O) = 28.875 cubic inches = 128 fluid drams.
2 pints	= 1 quart (qt) = 57.75 cubic inches = 32 fluid ounces = 256 fluid drams.
4 quarts	= 1 gallon (gal) = 231 cubic inches = 128 fluid ounces = 1 024 fluid drams.

DRY MEASURE °

2 pints (pt)	= 1 quart (qt) = 67.200 6 cubic inches.
8 quarts	= 1 peck (pk) = 537.605 cubic inches = 16 pints.
4 pecks	= 1 bushel (bu) = 2 150.42 cubic inches = 32 quarts.

AVOIRDUPOIS WEIGHT ^d

[The “grain” is the same in avoirdupois, troy, and apothecaries weight.]

27 11/32 grains	= 1 dram (dr).
16 grams	= 1 ounce (oz) = 437½ grains.
16 ounces	= 1 pound (lb) = 256 drams = 7 000 grains.
100 pounds	= 1 hundredweight (cwt). ^e
20 hundredweights	= 1 ton = 2 000 pounds. ^e

In “gross” or “long” measure, the following values are recognized:

112 pounds	= 1 gross or long hundredweight. ^e
20 gross or long hundredweights	= 1 gross or long ton = 2 240 pounds. ^e

TROY WEIGHT

[The “grain” is the same in avoirdupois, troy, and apothecaries weight.]

24 grains	= 1 pennyweight (dwt).
20 pennyweights	= 1 ounce troy (oz t) = 480 grains.
12 ounces troy	= 1 pound troy (lb t) = 240 pennyweights = 5 760 grains.

APOTHECARIES WEIGHT

[The “grain” is the same in avoirdupois, troy, and apothecaries weight.]

20 grains	= 1 scruple (s ap or ℥ \bar{D}).
3 scruples	= 1 dram apothecaries (dr ap or ℥ $\bar{5}$) = 60 grains.
8 drams apothecaries	= 1 ounce apothecaries (oz ap or ℥ $\bar{3}$) = 24 scruples = 480 grains.
12 ounces apothecaries	= 1 pound apothecaries (lb ap or lb) = 96 drams apothecaries = 288 scruples = 5 760 grains.

5.2. Notes on British Weights and Measures Tables

In Great Britain, the yard, the avoirdupois pound, the troy pound, and the apothecaries pound, are, for all commercial purposes, identical with the units of the same names used in the United States. The tables of British linear measure, troy weight, and apothecaries weight are the same as the corresponding United States tables, except for the British spelling “drachm”

^c When necessary to distinguish the *dry* pint or quart from the *liquid* pint or quart, the word “dry” should be used in combination with the name or abbreviation of the dry unit.

^d When necessary to distinguish the *avoirdupois* dram from the *apothecaries* dram, or to distinguish the *avoirdupois* dram or ounce from the *fluid* dram or ounce, or to distinguish the *avoirdupois* ounce or pound from the *troy* or *apothecaries* ounce or pound, the word “avoirdupois” or the abbreviation “avdp” should be used in combination with the name or abbreviation of the *avoirdupois* unit.

^e When the terms “hundredweight” and “ton” are used unmodified, they are commonly understood to mean the 100-pound hundredweight and the 2 000-pound ton, respectively; these units may be designated “net” or “short” when necessary to distinguish them from the corresponding units in *gross* or *long* measure.

in the table of apothecaries weight. The table of British avoirdupois weight is the same as the United States table up to 1 pound; above that point the table reads:

14 pounds	= 1 stone.
2 stones	= 1 quarter = 28 pounds.
4 quarters	= 1 hundredweight = 112 pounds.
20 hundredweight	= 1 ton = 2 240 pounds.

The present British gallon and bushel, known as the "Imperial gallon" and "Imperial bushel" are, respectively, about 20 percent and 3 percent larger than the United States gallon and bushel. The Imperial gallon is defined as the volume of 10 avoirdupois pounds of water under specified conditions, and the Imperial bushel is defined as 8 Imperial gallons. Also, the subdivision of the Imperial gallon as presented in the table of British apothecaries fluid measure differs in two important respects from the corresponding United States subdivision, in that the Imperial gallon is divided into 160 fluid ounces (whereas the United States gallon is divided into 128 fluid ounces), and a "fluid scruple" is included. The full table of British measures of capacity (which are used alike for liquid and for dry commodities) is as follows:

4 gills	= 1 pint.
2 pints	= 1 quart.
4 quarts	= 1 gallon.
2 gallons	= 1 peck.
8 gallons [4 pecks]	= 1 bushel.
8 bushels	= 1 quarter.

The full table of British apothecaries measure is as follows:

20 minims	= 1 fluid scruple.
3 fluid scruples	= 1 fluid drachm = 60 minims.
8 fluid drachms	= 1 fluid ounce.
20 fluid ounces	= 1 pint.
8 pints	= 1 gallon = 160 fluid ounces.

5.3. Tables of Metric Weights and Measures

In the metric system of weights and measures, designations of multiples and subdivisions of any unit may be arrived at by combining with the name of the unit the prefixes *deka*, *hecto*, and *kilo*, meaning, respectively, 10, 100, and 1 000, and *deci*, *centi*, and *milli*, meaning, respectively, one-tenth, one-hundredth, and one-thousandth. In some of the following metric tables, some such multiples and subdivisions have not been included for the reason that these have little, if any, currency in actual usage.

In certain cases, particularly in scientific usage, it becomes convenient to provide for multiples larger than 1 000 and for subdivisions smaller than one-thousandth. Accordingly, the following prefixes have been introduced and these are now generally recognized:

myria, meaning 10 000,
mega, meaning 1 000 000,
micro, meaning one-millionth.

A special case is found in the term "micron" (abbreviated as μ , the Greek letter mu), a coined word meaning one-millionth of a meter (equivalent to one-thousandth of a millimeter); a millimicron (abbreviated as $m\mu$) is one-thousandth of a micron (equivalent to one-millionth of a millimeter), and a micromicron (abbreviated as $\mu\mu$) is one-millionth of a micron (equivalent to one-thousandth of a millimicron or to 0.000 000 001 millimeter.)

LINEAR MEASURE

10 millimeters (mm)	=1 centimeter (cm).
10 centimeters	=1 decimeter (dm)=100 millimeters.
10 decimeters	=1 meter (m)=1 000 millimeters.
10 meters	=1 dekameter (dkm).
10 dekameters	=1 hectometer (hm)=100 meters.
10 hectometers	=1 kilometer (km)=1 000 meters.

AREA MEASURE

100 square millimeters (mm ²)	=1 square centimeter (cm ²).
10 000 square centimeters	=1 square meter (m ²)=1 000 000 square millimeters.
100 square meters	=1 are (a).
100 ares	=1 hectare (ha)=10 000 square meters.
100 hectares	=1 square kilometer (km ²)=1 000 000 square meters.

VOLUME MEASURE

10 milliliters (ml)	=1 centiliter (cl).
10 centiliters	=1 deciliter (dl)=100 milliliters.
10 deciliters	=1 liter ^f =1 000 milliliters.
10 liters	=1 dekaliter (dkl).
10 dekaliters	=1 hectoliter (hl)=100 liters.
10 hectoliters	=1 kiloliter (kl)=1 000 liters.

CUBIC MEASURE

1 000 cubic millimeters (mm ³)	=1 cubic centimeter (cm ³).
1 000 cubic centimeters	=1 cubic decimeter (dm ³)=1 000 000 cubic millimeters.
1 000 cubic decimeters	=1 cubic meter (m ³)=1 stere=1 000 000 cubic centimeters= 1 000 000 000 cubic millimeters.

WEIGHT

10 milligrams (mg)	=1 centigram (cg).
10 centigrams	=1 decigram (dg)=100 milligrams.
10 decigrams	=1 gram (g)=1 000 milligrams.
10 grams	=1 dekagram (dkg).
10 dekagrams	=1 hectogram (hg)=100 grams.
10 hectograms	=1 kilogram (kg)=1 000 grams.
1 000 kilograms	=1 metric ton (t).

^f The liter is defined as the volume occupied, under standard conditions, by a quantity of pure water having a mass of 1 kilogram. This volume is very nearly equal to 1 000 cubic centimeters or 1 cubic decimeter; the actual metric equivalent is, 1 liter=1 000.028 cubic centimeters. (The change in this equivalent from the previously published value of 1 000.027 is based on a recomputation of earlier data, carried out at the International Bureau of Weights and Measures.) Thus the milliliter and the liter are larger than the cubic centimeter and the cubic decimeter, respectively, by 28 parts in 1 000 000; except for determinations of high precision, this difference is so small as to be of no consequence.

5.4. TABLES OF INTERRELATION

UNITS OF

Units	Inches	Links	Feet	Yards	Rods
1 inch =	1	0.126 263	0.083 333 3	0.027 777 8	0.005 050 51
1 link =	7.92	1	0.66	0.22	0.04
1 foot =	12	1.515 152	1	0.333 333	0.060 606 1
1 yard =	36	4.545 45	3	1	0.181 818
1 rod =	198	25	16.5	5.5	1
1 chain =	792	100	66	22	4
1 mile =	63 360	8000	5280	1760	320
1 centimeter =	0.3937	0.049 709 60	0.032 808 33	0.010 936 111	0.001 988 384
1 meter =	39.37	4.970 960	3.280 833	1.093 611 1	0.198 838 4

UNITS OF

Units	Square inches	Square links	Square feet	Square yards	Square rods	Square chains
1 square inch =	1	0.015 942 3	0.006 944 44	0.000 771 605	0.000 025 507 6	0.000 001 594 23
1 square link =	62.7264	1	0.4356	0.0484	0.0016	0.0001
1 square foot =	144	2.295 684	1	0.111 111 1	0.003 673 09	0.000 229 568
1 square yard =	1296	20.6612	9	1	0.033 057 85	0.002 066 12
1 square rod =	39 204	625	272.25	30.25	1	0.0625
1 square chain =	627 264	10 000	4356	484	16	1
1 acre =	6 272 640	100 000	43 560	4840	160	10
1 square mile =	4 014 489 600	64 000 000	27 878 400	3 097 600	102 400	6400
1 square centimeter =	0.154 999 69	0.002 471 04	0.001 076 387	0.000 119 598 5	0.000 003 953 67	0.000 000 247 104
1 square meter =	1549.9969	24.7104	10.763 87	1.195 985	0.039 536 7	0.002 471 04
1 hectare =	15 499 969	247 104	107 638.7	11 959.85	395.367	24.7104

UNITS OF

Units	Cubic inches	Cubic feet	Cubic yards
1 cubic inch =	1	0.000 578 704	0.000 021 433 47
1 cubic foot =	1728	1	0.037 037 0
1 cubic yard =	46 656	27	1
1 cubic centimeter =	0.061 023 38	0.000 035 314 45	0.000 001 307 94
1 cubic decimeter =	61.023 38	0.035 314 45	0.001 307 943
1 cubic meter =	61 023.38	35.314 45	1.307 942 8

UNITS OF CAPACITY

Units	Minims	Fluid drams	Fluid ounces	Gills	Liquid pints
1 minim =	1	0.016 666 7	0.002 083 33	0.000 520 833	0.000 130 208
1 fluid dram =	60	1	0.125	0.031 25	0.007 812 5
1 fluid ounce =	480	8	1	0.25	0.0625
1 gill =	1920	32	4	1	0.25
1 liquid pint =	7680	128	16	4	1
1 liquid quart =	15 360	256	32	8	2
1 gallon =	61 440	1024	128	32	8
1 milliliter =	16.2311	0.270 518	0.033 814 8	0.008 453 69	0.002 113 42
1 liter =	16 231.1	270.518	33.8148	8.453 69	2.113 42
1 cubic inch =	265.974	4.432 90	0.554 113	0.138 528	0.034 632 0
1 cubic foot =	459 603.1	7660.052	957.506 5	239.376 6	59.844 16

OF UNITS OF MEASUREMENT

LENGTH

Chains	Milles	Centimeters	Meters	Units
0.001 262 63	0.000 015 782 8	2.540 005	0.025 400 05	= 1 inch
0.01	0.000 125	20.116 84	0.201 168 4	= 1 link
0.015 151 5	0.000 189 393 9	30.480 06	0.304 800 6	= 1 foot
0.045 454 5	0.000 568 182	91.440 18	0.914 401 8	= 1 yard
0.25	0.003 125	502.9210	5.029 210	= 1 rod
1	0.0125	2011.684	20.116 84	= 1 chain
80	1	160 934.72	1609.3472	= 1 mile
0.000 497 096 0	0.000 006 213 699	1	0.01	= 1 centimeter
0.049 709 60	0.000 621 369 9	100	1	= 1 meter

AREA

Acres	Square miles	Square centimeters	Square meters	Hectares	Units
0.000 000 159 423	0.000 000 000 249 1	6.451 626	0.000 645 162 6	0.000 000 064 516	= 1 square inch
0.000 01	0.000 000 015 625	404.6873	0.040 468 73	0.000 004 046 87	= 1 square link
0.000 022 956 8	0.000 000 035 870 1	929.0341	0.092 903 41	0.000 009 290 34	= 1 square foot
0.000 206 612	0.000 000 322 831	8361.307	0.836 130 7	0.000 083 613 1	= 1 square yard
0.006 25	0.000 009 765 625	252 929.5	25.292 95	0.002 529 295	= 1 square rod
0.1	0.000 156 25	4 046 873	404.6873	0.040 468 7	= 1 square chain
1	0.001 562 5	40 468 726	4046.873	0.404 687	= 1 acre
640	1	25 899 984 703	2 589 998	258.9998	= 1 square mile
0.000 000 024 710 4	0.000 000 000 038 610 06	1	0.0001	0.000 000 01	= 1 square centimeter
0.000 247 104	0.000 000 386 100 6	10 000	1	0.0001	= 1 square meter
2.471 04	0.003 861 006	100 000 000	10 000	1	= 1 hectare

VOLUME

Cubic centimeters	Cubic decimeters	Cubic meters	Units
16.387 162	0.016 387 16	0.000 016 387 16	= 1 cubic inch
28 317.016	28.317 016	0.028 317 016	= 1 cubic foot
764 559.4	764.5594	0.764 559 4	= 1 cubic yard
1	0.001	0.000 001	= 1 cubic centimeter
1 000	1	0.001	= 1 cubic decimeter
1 000 000	1000	1	= 1 cubic meter

LIQUID MEASURE

Liquid quarts	Gallons	Milliliters	Liters	Cubic inches	Cubic feet	Units
0.000 065 104	0.000 016 276	0.061 610 2	0.000 061 610 2	0.003 759 77	0.000 002 175 79	= 1 minim
0.003 906 25	0.000 976 562	3.696 61	0.003 696 61	0.225 586	0.000 130 547	= 1 fluid dram
0.031 25	0.007 812 5	29.5729	0.029 572 9	1.804 69	0.001 044 38	= 1 fluid ounce
0.125	0.031 25	118.292	0.118 292	7.218 75	0.004 177 52	= 1 gill
0.5	0.125	473.166	0.473 166	28.875	0.016 710 1	= 1 liquid pint
1	0.25	946.332	0.946 332	57.75	0.033 420 1	= 1 liquid quart
4	1	3785.329	3.785 329	231	0.133 680 6	= 1 gallon
0.001 056 71	0.000 264 178	1	0.001	0.061 025 1	0.000 035 315 4	= 1 milliliter
1.056 71	0.264 178	1000	1	61.025 1	0.035 315 4	= 1 liter
0.017 316 0	0.004 329 00	16.3867	0.016 386 7	1	0.000 578 703 7	= 1 cubic inch
29.922 08	7.480 519 5	28 316.22	28.316 22	1728	1	= 1 cubic foot.

UNITS OF CAPACITY

Units	Dry pints	Dry quarts	Pecks	Bushels
1 dry pint =	1	0.5	0.0625	0.015 625
1 dry quart =	2	1	0.125	0.031 25
1 peck =	16	8	1	0.25
1 bushel =	64	32	4	1
1 liter =	1.816 21	0.908 103	0.113 513	0.028 378
1 dekaliter =	18.1621	9.081 03	1.135 13	0.283 78
1 cubic inch =	0.029 761 6	0.014 880 8	0.001 860 10	0.000 465 015
1 cubic foot =	51.428 093	25.714 047	3.214 255 8	0.803 563 95

UNITS OF MASS NOT GREATER

Units	Grains	Apothecaries scruples	Pennyweights	Avoirdupois drams	Apothecaries drams	Avoirdupois ounces
1 grain =	1	0.05	0.041 666 67	0.035 571 43	0.016 666 7	0.002 285 71
1 apoth. scruple =	20	1	0.833 333 3	0.731 428 6	0.333 333	0.045 714 3
1 pennyweight =	24	1.2	1	0.877 714 3	0.4	0.054 857 1
1 avdp. dram =	27.343 75	1.367 187 5	1.139 323	1	0.455 729 2	0.0625
1 apoth. dram =	60	3	2.5	2.194 286	1	0.137 142 9
1 avdp. oz. =	437.5	21.875	18.229 17	16	7.291 67	1
1 apoth. or troy oz. =	480	24	20	17.554 28	8	1.097 142 9
1 apoth. or troy lb. =	5760	288	240	210.6514	96	13.165 714
1 avdp. lb. =	7000	350	291.6667	256	116.6667	16
1 milligram =	0.015 432 356	0.000 771 618	0.000 643 014 8	0.000 564 383 3	0.000 257 205 9	0.000 035 273 96
1 gram =	15.432 356	0.771 618	0.643 014 85	0.564 383 3	0.257 205 9	0.035 273 96
1 kilogram =	15 432.356	771.6178	643.014 85	564.383 32	257.205 94	35.273 96

UNITS OF MASS NOT LESS

Units	Avoirdupois ounces	Avoirdupois pounds	Short hundred-weights	Short tons
1 avoirdupois ounce =	1	0.0625	0.000 625	0.000 031 25
1 avoirdupois pound =	16	1	0.01	0.0005
1 short hundredweight =	1600	100	1	0.05
1 short ton =	32 000	2000	20	1
1 long ton =	35 840	2240	22.4	1.12
1 kilogram =	35.273 957	2.204 622 34	0.022 046 223	0.001 102 311 2
1 metric ton =	35 273.957	2204.622 34	22.046 223	1.102 311 2

DRY MEASURE

Liters	Dekaliters	Cubic inches	Cubic feet	Units
0.550 598	0.055 059 8	33.600 312 5	0.019 444 63	=1 dry pint
1.101 197	0.110 119 7	67.200 625	0.038 889 25	=1 dry quart
8.809 57	0.880 957	537.605	0.311 114	=1 peck
35.2383	3.523 83	2150.42	1.244 456	=1 bushel
1	0.1	61.0251	0.035 315 4	=1 liter
10	1	610.251	0.353 154	=1 dekaliter
0.016 386 7	0.001 638 67	1	0.000 578 703 7	=1 cubic inch
28.316 22	2.831 622	1728	1	=1 cubic foot

THAN POUNDS AND KILOGRAMS

Apothecaries or troy ounces	Apothecaries or troy pounds	Avoirdupois pounds	Milligrams	Grams	Kilograms	Units
0.002 083 33	0.000 173 611 1	0.000 142 857 1	64.798 918	0.064 798 918	0.000 064 798 9	=1 grain
0.041 666 7	0.003 472 222	0.002 857 143	1295.9784	1.295 978 4	0.001 295 978	=1 apoth. scruple
0.05	0.004 166 667	0.003 428 571	1555.1740	1.555 174 0	0.001 555 174	=1 pennyweight
0.056 966 146	0.004 747 178 8	0.003 906 25	1771.8454	1.771 845 4	0.001 771 845	=1 avdp. dram
0.125	0.010 416 667	0.008 571 429	3887.9351	3.887 935 1	0.003 887 935	=1 apoth. dram
0.911 458 3	0.075 954 861	0.0625	28 349.527	28.349 527	0.028 349 53	=1 avdp. ounce
1	0.083 333 33	0.068 571 43	31 103.481	31.103 481	0.031 103 48	=1 apoth. or troy ounce
12	1	0.822 857 1	373 241.77	373.241 77	0.373 241 77	=1 apoth. or troy pound
14.583 333	1.215 277 8	1	453 592.4277	453.592 4277	0.453 592 427 7	=1 avdp. pound
0.000 032 150 74	0.000 002 679 23	0.000 002 204 62	1	0.001	0.000 001	=1 milligram
0.032 150 74	0.002 679 23	0.002 204 62	1000	1	0.001	=1 gram
32.150 742	2.679 228 5	2.204 622 341	1 000 000	1000	1	=1 kilogram

THAN AVOIRDUPOIS OUNCES

Long tons	Kilograms	Metric tons	Units
0.000 027 901 79	0.028 349 53	0.000 028 349 53	=1 avoirdupois ounce
0.000 446 428 6	0.453 592 427 7	0.000 453 592 43	=1 avoirdupois pound
0.044 642 86	45.359 243	0.045 359 243	=1 short hundredweight
0.892 857 1	907.184 86	0.907 184 86	=1 short ton
1	1016.047 04	1.016 047 04	=1 long ton
0.000 984 206 4	1	0.001	=1 kilogram
0.984 206 40	1000	1	=1 metric ton

5.5. Tables of Equivalents

When the name of a unit is enclosed in brackets (thus, [1 hand] ----), this indicates (1) that the unit is not in general current use in the United States, or (2) that the unit is believed to be based on "custom and usage" rather than on formal authoritative definition.

Equivalents involving decimals are, in most instances, rounded off to the third decimal place except where they are exact, in which cases these exact equivalents are so designated.

LENGTHS

1 angstrom (A)*-----	$\left\{ \begin{array}{l} 0.1 \text{ millimicron.} \\ 0.000 \text{ 1 micron.} \\ 0.000 \text{ 000 1 millimeter.} \\ 0.000 \text{ 000 004 inch.} \end{array} \right.$
1 cable's length-----	$\left\{ \begin{array}{l} 120 \text{ fathoms.} \\ 720 \text{ feet.} \\ 219.456 \text{ meters.} \end{array} \right.$
1 centimeter (cm)-----	0.393 7 inch (exactly).
1 chain (ch) (Gunter's or surveyors)-----	$\left\{ \begin{array}{l} 66 \text{ feet.} \\ 20.117 \text{ meters.} \end{array} \right.$
[1 chain] (engineers)-----	$\left\{ \begin{array}{l} 100 \text{ feet.} \\ 30.480 \text{ meters.} \end{array} \right.$
1 decimeter (dm)-----	3.937 inches (exactly).
1 dekameter (dkm)-----	32.808 feet.
1 fathom-----	$\left\{ \begin{array}{l} 6 \text{ feet.} \\ 1.829 \text{ meters.} \end{array} \right.$
1 foot (ft)-----	0.305 meter.
1 furlong (fur.)-----	$\left\{ \begin{array}{l} 10 \text{ chains (surveyors).} \\ 660 \text{ feet.} \\ 220 \text{ yards.} \\ \frac{1}{8} \text{ statute mile.} \\ 201.168 \text{ meters.} \end{array} \right.$
[1 hand]-----	4 inches.
1 inch (in.)-----	2.540 centimeters.
1 kilometer (km)-----	0.621 mile.
1 league (land)-----	$\left\{ \begin{array}{l} 3 \text{ statute miles.} \\ 4 \text{ 828 kilometers.} \end{array} \right.$
1 link (li) (Gunter's or surveyors)-----	$\left\{ \begin{array}{l} 7.92 \text{ inches (exactly).} \\ 0.201 \text{ meter.} \end{array} \right.$
[1 link (li) (engineers)]-----	$\left\{ \begin{array}{l} 1 \text{ foot.} \\ 0.305 \text{ meter.} \end{array} \right.$
1 meter (m)-----	$\left\{ \begin{array}{l} 39.37 \text{ inches (exactly).} \\ 1.094 \text{ yards.} \end{array} \right.$
1 micron (μ [the Greek letter mu])-----	$\left\{ \begin{array}{l} 0.001 \text{ millimeter (exactly).} \\ 0.000 \text{ 039 37 inch (exactly).} \end{array} \right.$
1 mil-----	$\left\{ \begin{array}{l} 0.001 \text{ inch (exactly).} \\ 0.025 \text{ 4 millimeter.} \end{array} \right.$
1 mile (mi) (statute or land)-----	$\left\{ \begin{array}{l} 5 \text{ 280 feet.} \\ 1.609 \text{ kilometers.} \end{array} \right.$
[1 mile (mi) (nautical, geographical, or sea, U. S.)] ^b -----	$\left\{ \begin{array}{l} 1.152 \text{ statute miles.} \\ 6 \text{ 080.20 feet.} \\ 1.853 \text{ kilometers.} \\ 1.001 \text{ international nautical mile.} \end{array} \right.$
1 mile (mi) (nautical, international) ^b -----	$\left\{ \begin{array}{l} 1.852 \text{ kilometers (exactly).} \\ 1.151 \text{ statute miles.} \\ 0.999 \text{ U. S. nautical miles.} \end{array} \right.$
1 millimeter (mm)-----	0.039 37 inch (exactly).

* The angstrom is basically defined in terms of the wavelength of the red radiation of cadmium under specified conditions by the relation $1 \text{ wavelength} = 6 \text{ 438.469 6 angstroms}$.

^b The international nautical mile of 1 852 meters (6 076.103 33 . . . feet) was adopted effective July 1, 1954 for use in the United States. The value formerly used in the United States was 6 080.20 feet = 1 nautical (geographical or sea) mile

1 millimicron ($m\mu$ [the English letter m in combination with the Greek letter mu])	{ 0.001 micron (exactly). 0.000 000 039 37 inch (exactly).
1 point (typography)	{ 0.013 837 inch (exactly). $\frac{1}{72}$ inch (approximately). 0.351 millimeter.
1 rod (rd), pole, or perch	{ 16 $\frac{1}{2}$ feet. 5 $\frac{1}{2}$ yards. 5.029 meters.
1 yard (yd)	{ 0.914 meter.

AREAS OR SURFACES

1 acre ⁱ	{ 43 560 square feet. 4 840 square yards. 0.405 hectare.
1 are	{ 119.596 square yards. 0.025 acre.
1 hectare	{ 2.471 acres.
[1 square (building)]	{ 100 square feet.
1 square centimeter (cm^2)	{ 0.155 square inch.
1 square decimeter (dm^2)	{ 15.500 square inches.
1 square foot (sq ft)	{ 929.034 square centimeters.
1 square inch (sq in.)	{ 6.452 square centimeters
1 square kilometer (km^2)	{ 247.104 acres. 0.386 square mile.
1 square meter (m^2)	{ 1.196 square yards. 10.764 square feet.
1 square mile (sq mi)	{ 259.000 hectares.
1 square millimeter (mm^2)	{ 0.002 square inch.
1 square rod (sq rd), sq pole, or sq perch	{ 25.293 square meters.
1 square yard (sq yd)	{ 0.836 square meter.

CAPACITIES OR VOLUMES

1 barrel (bbl), liquid	{ 31 to 42 gallons. [†]
1 barrel (bbl), standard for fruits, vegetables, and other dry commodities except cranberries	{ 7 056 cubic inches. 105 dry quarts. 3.281 bushels, struck measure.
1 barrel (bbl), standard, cranberry	{ 5 826 cubic inches. 86 $\frac{3}{4}$ dry quarts. 2.709 bushels, struck measure.
1 bushel (bu) (U. S.) struck measure	{ 2 150.42 cubic inches (exactly). 35.238 liters.
[1 bushel, heaped (U. S.)]	{ 2 747.715 cubic inches. 1.278 bushels, struck measure. ^k
[1 bushel (bu) (British Imperial) (struck measure)]	{ 1.032 U. S. bushels, struck measure. 2 219.36 cubic inches.
1 cord (cd) (firewood)	{ 128 cubic feet.
1 cubic centimeter (cm^3)	{ 0.061 cubic inch.
1 cubic decimeter (dm^3)	{ 61.023 cubic inches.
1 cubic foot (cu ft)	{ 7.481 gallons. 28.317 cubic decimeters.
1 cu inch (cu in.)	{ 0.554 fluid ounce. 4.433 fluid drams. 16.387 cubic centimeters.
1 cubic meter (m^3)	{ 1.308 cubic yards.
1 cubic yard (cu yd)	{ 0.765 cubic meter.

[†] The question is often asked as to the length of a side of an acre of ground. An acre is a unit of area containing 43 560 square feet. It is not necessarily square, or even rectangular. But, if it is square then the length of a side is equal to $\sqrt{43\ 560}=208.710+$ feet.

^j There are a variety of "barrels" established by law or usage. For example, Federal taxes on fermented liquors are based on a barrel of 31 gallons; many State laws fix the "barrel for liquids" as 31 $\frac{1}{2}$ gallons; one State fixes a 36-gallon barrel for cistern measurement; Federal law recognizes a 40-gallon barrel for "proof spirits"; by custom, 42 gallons comprise a barrel of crude oil or petroleum products for statistical purposes, and this equivalent is recognized "for liquids" by four States.

^k Frequently recognized as 1 $\frac{3}{4}$ bushels, struck measure.

1 cup, measuring-----	{ 8 fluid ounces. ½ liquid pint.
1 dram, fluid (or liquid) (fl dr or <i>f</i> ̄) (U. S.)-----	{ ⅛ fluid ounce. 0.226 cubic inch. 3.697 milliliters. 1.041 British fluid drachms.
[1 drachm, fluid (fl dr) (British)]-----	{ 0.961 U. S. fluid dram. 0.217 cubic inch. 3.552 milliliters.
1 dekaliter (dkl)-----	{ 2.642 gallons. 1.135 pecks.
1 gallon (gal) (U. S.)-----	{ 231 cubic inches. 3.785 liters. 0.833 British gallon. 128 U. S. fluid ounces.
[1 gallon (gal) (British Imperial)]-----	{ 277.42 cubic inches. 1.201 U. S. gallons. 4.546 liters.
1 gill (gi)-----	{ 160 British fluid ounces. 7.219 cubic inches. 4 fluid ounces. 0.118 liter.
1 hectoliter (hl)-----	{ 26.418 gallons. 2.838 bushels.
1 liter-----	{ 1.057 liquid quarts. 0.908 dry quart. 61.025 cubic inches.
1 milliliter (ml)-----	{ 0.271 fluid dram. 16.231 minims. 0.061 cubic inch.
1 ounce, fluid (or liquid) (fl oz or <i>f</i> ̄) (U. S.)-----	{ 1.805 cubic inches. 29.573 milliliters.
[1 ounce, fluid (fl oz) (British)]-----	{ 1.041 British fluid ounces. 0.961 U. S. fluid ounce. 1.734 cubic inches. 28.412 milliliters.
1 peck (pk)-----	{ 8.810 liters.
1 pint (pt), dry-----	{ 33.600 cubic inches. 0.551 liter.
1 pint (pt), liquid-----	{ 28.875 cubic inches (exactly). 0.473 liter.
1 quart (qt), dry (U. S.)-----	{ 67.201 cubic inches. 1.101 liters.
1 quart (qt), liquid (U. S.)-----	{ 0.969 British quart. 57.75 cubic inches (exactly). 0.946 liter.
[1 quart (qt) (British)]-----	{ 0.833 British quart. 69.354 cubic inches. 1.032 U. S. dry quarts.
1 tablespoon-----	{ 1.201 U. S. liquid quarts. 3 teaspoons. ¹ 4 fluid drams. ½ fluid ounce.
1 teaspoon-----	{ ⅓ tablespoon. ¹ 1⅓ fluid drams. ¹
1 water ton (English)-----	{ 270.91 U. S. gallons. 224 British Imperial gallons (exactly).

¹ The equivalent "1 teaspoon=1½ fluid drams" has been found by the Bureau to correspond more closely with the actual capacities of "measuring" and silver teaspoons than the equivalent "1 teaspoon=1 fluid dram," which is given by a number of dictionaries.

WEIGHTS OR MASSES

1 assay ton ^m (AT)-----	29.167 grams.
1 carat (c)-----	{ 200 milligrams. 3.086 grains.
1 dram, apothecaries (dr ap or <i>f</i> ̄)-----	{ 60 grains. 3.888 grams.
1 dram, avoirdupois (dr avdp)-----	{ 27 ¹ / ₂ (= 27.344) grains. 1.772 grams.
gamma, <i>see</i> microgram.	
1 grain-----	64.799 milligrams.
1 gram (g)-----	{ 15.432 grains. 0.035 ounce, avoirdupois.
1 hundredweight, gross or long ⁿ (gross cwt)-----	{ 112 pounds. 50.802 kilograms.
1 hundredweight, net or short (cwt or net cwt)-----	{ 100 pounds. 45.359 kilograms.
1 kilogram (kg)-----	2.205 pounds.
1 microgram (μ g [the Greek letter mu in combination with the letter g]) ^o -----	0.000 001 gram (exactly).
1 milligram (mg)-----	0.015 grain.
1 ounce, avoirdupois (oz avdp)-----	{ 437.5 grains (exactly). 0.911 troy or apothecaries ounce. 28.350 grams.
1 ounce, troy or apothecaries (oz t or oz ap or <i>f</i> ̄)-----	{ 480 grains. 1.097 avoirdupois ounces. 31.103 grams.
1 pennyweight (dwt)-----	1.555 grams.
1 point-----	{ 0.01 carat. 2 milligrams.
1 pound, avoirdupois (lb avdp)-----	{ 7 000 grains. 1.215 troy or apothecaries pounds. 453.592 grams.
1 pound, troy or apothecaries (lb t or lb ap)-----	{ 5 760 grains. 0.823 avoirdupois pound. 373.242 grams.
1 scruple (s ap or \mathfrak{D})-----	{ 20 grains. 1.296 grams.
1 ton, gross or long ^p -----	{ 2 240 pounds. 1.12 net tons (exactly). 1.016 metric tons.
1 ton, metric (t)-----	{ 2 204.622 pounds. 0.984 gross ton. 1.102 net tons.
1 ton, net or short-----	{ 2 000 pounds. 0.893 gross ton. 0.907 metric ton.

^m Used in assaying. The assay ton bears the same relation to the milligram that a ton of 2 000 pounds avoirdupois bears to the ounce troy; hence the weight in milligrams of precious metal obtained from one assay ton of ore gives directly the number of troy ounces to the net ton.

ⁿ The gross or long ton and hundredweight are used commercially in the United States to only a very limited extent, usually in restricted industrial fields. These units are the same as the British "ton" and "hundredweight."

^o The symbol γ [the Greek letter gamma] is also used.

^p The gross or long ton and hundredweight are used commercially in the United States to a limited extent only, usually in restricted industrial fields. These units are the same as the British "ton" and "hundredweight."

Calibration of Meter Line Standards of Length at the National Bureau of Standards

Benjamin L. Page

The results of intercomparisons of the total lengths of several meter bars and of calibrations of the subintervals of some of these bars for the past twenty years are reported. Information both on the degree of stability of these standards and on the precision with which such measurements can be repeated was obtained. The results are critically analyzed and comments made as to the precautions necessary in the precision comparisons of line standards of length.

1. Introduction

It is known that the length of a linear standard, at any specified temperature, cannot be assumed to remain the same over a period of years. The changes in length may be attributed to secular changes, although these changes are not necessarily uniform throughout the length of any linear standard. It is essential, therefore, that linear standards be recalibrated at sufficiently frequent intervals so that these calibrations may be relied upon to the requisite accuracy in their use.

The standard length in the United States is Prototype Meter 27, kept at the National Bureau of Standards. In addition to this meter bar, the National Bureau of Standards has other line standards of length that are used as working standards, and nine of these were included in the calibrations described in this paper. To calibrate these standards, intercomparisons by pairs are made, using different combinations in order to determine the most probable corrections to their total lengths. As these intercomparisons require measurements of the highest accuracy, only one pair of bars can be compared on any one day. A complete intercomparison, therefore, requires an observing period of months, and these complete intercomparisons are made at intervals of several years. A previous paper¹ describes the determination of the total lengths of some of the present NBS standards and also the results of that and earlier intercomparisons.

Inasmuch as a complete intercomparison of 10 meter bars is a time-consuming labor, the question arises as to whether or not statistical methods would diminish this labor without significant loss of accuracy. This paper discusses this question and gives the analysis. It also reports the intercomparisons that have been made since the report in 1934 and shows the trends in the length changes of these bars. Redeterminations of the calibration corrections of some of the subintervals of the subdivided linear standards have also been made.

2. Description of meter bars

Nine meter bars were intercompared in 1952 and 1953, and the results of these comparisons are dis-

¹ Lewis V. Judson and Benjamin L. Page, *J. Research NBS* **13**, 757 (1934) RP743.

cussed. Four of these bars are made of platinum-iridium, one of 42 percent nickel-steel, three of invar, and one of stainless steel (Fe-Cr).

The four platinum-iridium bars have a modified X cross section generally referred to as the Tresca section; the others have the more familiar H cross section, and all intervals are defined by lines ruled on the plane of the neutral axis. When in use, each bar is supported at the Airy points, which are located 28.55 cm in each direction from the center of the bar.

Prototype Meter 27 is the primary standard of length of the United States, and meter 21 is a second bar of the same series. The other two platinum-iridium meters, 4 and 12, are of an earlier melt known as Alloy of 1874. The four nickel-steel bars are: Meter 39, made of invar, was obtained in 1903; meter 153R, made of 42 percent nickel-steel, with an inlaid platinum strip on which the graduations are ruled, was obtained in 1911; meter 814B, made of invar, was obtained in 1931; and meter 752, made of a type of invar termed "Fix Invar", was obtained in 1938. The other bar of this group, meter 50, is made of stainless steel and was graduated by C. G. Peters, formerly of the Bureau, by use of wavelengths of light.

The four nickel-steel bars were obtained from the Société Gènevoise d'Instruments de Physique of Geneva, Switzerland, and are graduated at intervals of 1 mm throughout their lengths. Meter 4 is similarly graduated. Meters 27 and 21 have only the 1-m interval, which is defined as the interval from a central line of a group of three at one end to the central line of a similar group at the other end. Meter 12 was recently regraduated by C. G. Peters, while a member of the Bureau. Originally, this bar had rather inferior graduations at the terminal ends. This bar is considered as two linear standards for the purpose of these calibrations, because two 1-m intervals were graduated on it with a common zero; one interval is nominally correct at 0° C and the other at 20° C.

On all the bars subdivided to millimeters there is a 1-mm interval subdivided to tenths ruled outside of the terminal graduations. These may be conveniently used for the determination of the screw values of the micrometer microscopes.

Meter 50 has only a zero line and a 1-m line.

On each bar two parallel lines approximately 0.2 mm apart are ruled at right angles to the graduations. Only that portion of a graduation lying between the two parallel lines is considered when measurements are made. These longitudinal lines are also very convenient when alining the bars in the comparator.

Table 1 gives coefficients of the linear thermal expansion of the meter bars, previously determined at the Bureau International des Poids et Mesures (BIPM) and the National Bureau of Standards (NBS). This table gives the values of a and b of the general equation

$$L_t = L_0(1 + at + bt^2), \quad (1)$$

where L_t is the length of the bar at any temperature near 20° C, and L_0 is the length at 0° C.

TABLE 1. Coefficients of expansion of the meter bars

Meter bars ¹	Alloy	Coefficients of expansion	
		a	b
27	Platinum-iridium	8.6210×10^{-6}	$+0.00180 \times 10^{-6}$
4 (1874)	do	8.6014	+ .00180
12 (1874)	do	8.6014	+ .00180
21	do	8.6210	+ .00180
39	Invar	1.54	
153R	42% nickel-steel	7.009	+ .00083
752	Fix Invar	0.997	+ .00402
814B	Invar	1.13	
50	Stainless steel (Fe-Cr)	10.25	

¹ Meters 27 and 21 are of the principal national series of prototypes; meters 12 and 4 of the alloy of 1874; meters 39, 153R, 752, and 814B have serial numbers of the Société d'Instruments de Physique; and meter 50 was graduated at the National Bureau of Standards. Meter 153R has an inlaid platinum strip on which the graduations are ruled.

3. Description of Longitudinal Comparator

The comparator (fig. 1) used in these calibrations is located in a constant-temperature room in the basement of the NBS South Building. It is mounted on a massive concrete pier whose upper surface is approximately 20 cm below the level of the floor of the room and entirely independent of it. This comparator is a longitudinal comparator; the relative motion of the microscope beam and of the bars being calibrated is in the direction longitudinal to the axis of the bars. In the NBS comparator the bars are stationary with their axes colinear, and a carriage

with a beam bearing two microscopes is movable in a longitudinal direction. The microscopes may be clamped to the beam so that a simultaneous comparison of two bars may be made. Subdivisions of a bar up to 2 m in length (approximately 80 in.) may also be calibrated. The comparator has an over-all length of 4½ m.

The longitudinal movement of the carriage is effected by two electric motors geared to provide linear speeds of the carriage from 0.1 to 10 mm/sec. Automatic braking is provided so that the carriage can be stopped within a few microns of any desired position. An automatic cutoff is also provided for stopping the motion of the carriage to prevent its running off the ways of the bed. The total travel of the carriage is 130 cm.

The beam on which the microscopes are mounted is attached to the movable carriage through a system of self-aligning ball bearings and a link, so that the beam will have a minimum strain resulting from any distortions of the carriage caused by longitudinal displacement of any magnitude up to 130 cm, regardless of the distance between microscopes.

The two microscopes have tubes approximately 45 mm in diameter and 450 mm in length and have provision for adjustment. The total magnification as used in these calibrations is approximately 160 diam. The smallest division of the micrometer drums is equivalent to 0.50 μ , measured at the focal plane of the microscope. By use of a vernier-scale adjacent to the micrometer drum, it is possible to estimate to 0.005 μ .

The graduations on the standards are illuminated by means of the vertical illuminators in the microscopes, using low-voltage lamps. Heat-absorbing glass installed between the lamps and microscopes greatly reduces the heat radiation from the lamps and therefore the temperature gradients within the bars being calibrated. Each microscope is mounted in a holder that can be moved along the ways of the beam, and may be securely clamped at any position. Also provided is a slow-motion mechanism for the longitudinal movement of the microscopes. The range in separation of the microscopes is from 15 to 210 cm.

Two main supports for the bars are provided, and each is capable of complete adjustment for focusing and alining the bars, so that the parallel longi-

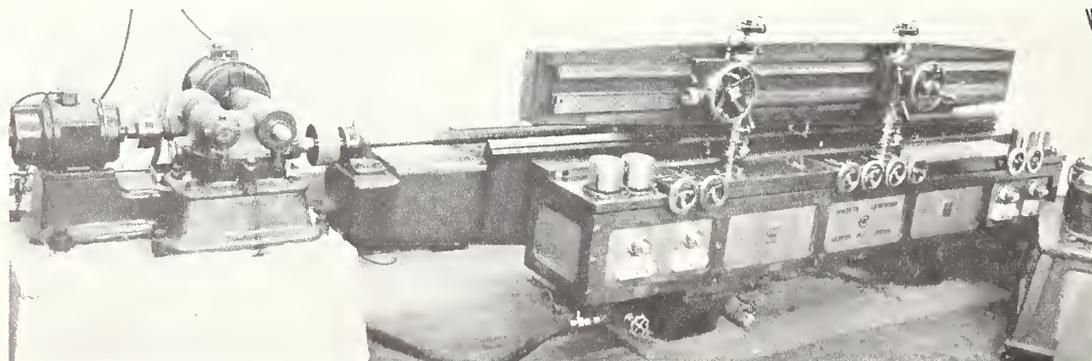


FIGURE 1. Longitudinal comparator of the National Bureau of Standards.

tudinal lines on the bars are parallel to the motion of the microscopes and are centrally located in the fields of the microscopes. Two adjustable roller supports, about 1 cm in diameter, are clamped to each of the main supports, with the proper separation, for supporting the bars. These supports are mounted in the inner of two tanks, which may serve as a double bath, the outer one being suitably insulated. Water may be used in either or both tanks and circulated by means of motor-driven propellers. Apertures, about 25 mm in diameter, are provided in the cover directly above the bar graduations that are under observation.

During the measurements the entire instrument is protected against external sources of heat by a thick quilted insulating cover, so that only the micrometer boxes are exposed.

4. Calibration of the Meter Bars

The latest fundamental measurements on this group of nine meter bars (considered as 10 for the purpose of these calibrations) were begun in 1952 and completed in 1953. The results of previous fundamental and auxiliary measurements of the total lengths of these bars, together with some repetitions of present measurements of some of the subintervals to show the stability of various standards over a period of years and the repeatability of present comparisons, are included in this paper. Another important part of the work was the calibration of certain of the subintervals of the bars subdivided to millimeters to provide accurate secondary standards for the calibration of line standards that are shorter than 1 m.

4.1. Determination of Total Length

All measurements on the bars were made on the longitudinal comparator, with the temperature of the room thermostatically controlled at 20° C. The bars were allowed to remain in the comparator for 24 hr, so that they would be in thermal equilibrium at a temperature very near 20° C before the measurements were begun. All measured differences in the lengths between any two bars were reduced to 20° C, by using the values for their respective coefficients of linear thermal expansion shown in table 1, before computing their respective corrections. For convenience, the corrections to the standards are given instead of the actual lengths. The actual length is equal to the nominal length plus the correction; hence, a plus correction indicates that a standard is longer than the nominal length.

Prior to the comparison in 1952 and 1953, the thermal conditions of the bars in the comparator were investigated. The temperature differences between bars, as indicated either by thermocouples or by mercury thermometers, were in agreement to approximately 0.005 deg C.

In order to determine the effect that water in the outer tank would have on the temperature of the bars in the inner tank, the outer tank was filled with

water at nearly the same temperature as the bars. After the water had been circulated manually, there was no perceptible reduction in the temperature gradients within each bar nor was the difference in temperature less between the bars. The motors were then used to circulate the water for 2 min. Over the period of an hour after the motors had been turned off the temperature of the air rose 0.03 deg C, and the temperature of the bars remained constant. The conclusion is that the thermal conditions in this comparator are considerably less stable when the circulating bath is used, because of the heat generated by the motors.

The 10 bars selected for standardization were placed in the comparator, two at a time and in a random manner with respect to their relative positions, either to the right or to the left. To determine the magnitude of any possible bias because of the location of a standard in either the left or right end of the comparator, several auxiliary intercomparisons of groups of 5 and 6 of the bars were made. A special observing schedule suggested by the Statistical Engineering Section of the National Bureau of Standards, whereby the bars were mounted in a symmetrical manner, showed that there was possibly a small bias, which was considerably smaller than the reproducibility claimed for the measurements. All measurements were made with the bars in air at a controlled temperature of approximately 20° C, and no water was circulated in the outer tank.

To obtain the difference in length of two bars, two microscopes separated by a distance of 1 m were focused on the 0- and 1-m graduations of one of the bars. Micrometer observations were then made on this bar, the procedure being to record 2 observations on the left microscope, 4 on the right, and then 2 on the left. The carriage was then moved so that the microscopes were over the 0- and 1-m graduations of the other bar, and these graduations were brought into focus. Observations were then made on this bar. For convenience in computing, the bar at the right was always designated as *A* and the other as *B*. The comparisons between each pair of bars, *A* and *B*, were repeated 10 times, and the temperature of each bar was recorded. The procedure was to observe first on *B*, move the carriage to the right, observe on *A*, repeat the observations on *A*, return the carriage to the left, and observe on *B*. This cycle was repeated 5 times, and thus a total of 10 differences was obtained. The mean of these 10 differences was reduced to 20° C, and the difference in the length of the bars, $L=A-B$, thus determined. This method was continued until each bar had been compared with all others, and a total of $[N(N-1)]/2$, or 45 differences, had been determined. The results obtained are shown in table 2.

The reduction of these observed differences by the method of least squares was carried out by the use of the form² shown in table 3.

The differences, *v*, between the observed and calculated differences are shown in table 3. These

² A. Pérard and C. Volet, Les mètres prototypes du Bureau International, Travaux et Mémoires du Bureau International des Poids et Mesures 21, (1945).

TABLE 2. Differences in length of meter bars at 20° C

(1953 series of comparisons)

Observations made at temperature, *t*, and reduced to 20° C, using the values of the coefficients of linear thermal expansion given in table 1.

Meter bars compared		Observed difference, <i>L</i>	Observed temperature, <i>t</i>	
			A	B
<i>A</i>	<i>B</i>	<i>μ</i>	°C	°C
4	minus 27	-4.33	19.87	19.78
12 ^a	minus 27	+0.87	20.13	20.03
12 ^b	minus 27	-171.93	20.17	20.05
21	minus 27	+5.11	19.96	19.88
39	minus 27	-177.13	19.89	19.81
153R	minus 27	-23.70	20.01	19.90
752	minus 27	-179.80	20.14	20.04
814B	minus 27	-170.34	19.93	19.89
50	minus 27	-171.40	20.07	20.01
12 ^a	minus 4	+5.36	19.86	19.84
12 ^b	minus 4	-167.68	19.88	19.90
21	minus 4	+9.36	19.89	19.89
39	minus 4	-172.88	20.06	20.04
153R	minus 4	-19.50	20.10	20.00
752	minus 4	-175.53	19.97	19.93
814B	minus 4	-165.99	20.13	20.05
50	minus 4	-167.19	19.87	19.86
12 ^b	minus 12 ^a	-173.01	19.81	19.81
21	minus 12 ^a	+4.33	20.10	20.10
39	minus 12 ^a	-177.99	19.72	19.74
153R	minus 12 ^a	-24.68	20.14	20.13
752	minus 12 ^a	-180.65	19.83	19.84
814B	minus 12 ^a	-171.40	19.89	19.90
50	minus 12 ^a	-172.56	20.09	20.10
21	minus 12 ^b	+177.36	20.12	20.13
39	minus 12 ^b	-5.01	19.74	19.75
153R	minus 12 ^b	+148.40	20.18	20.18
752	minus 12 ^b	-7.62	19.81	19.79
814B	minus 12 ^b	+1.96	19.90	19.91
50	minus 12 ^b	+0.50	20.05	20.05
39	minus 21	-182.36	20.13	20.13
153R	minus 21	-28.80	19.90	19.83
752	minus 21	-184.94	19.86	19.84
814B	minus 21	-175.46	19.96	19.95
50	minus 21	-176.45	19.95	19.95
153R	minus 39	+153.20	19.80	19.85
752	minus 39	-2.66	19.70	19.66
814B	minus 39	+6.96	20.06	19.93
50	minus 39	+5.66	20.04	20.05
752	minus 153R	-155.86	20.00	19.92
814B	minus 153R	-146.68	19.86	19.88
50	minus 153R	-147.62	19.99	19.96
814B	minus 752	+9.35	19.95	19.97
50	minus 752	+8.46	19.82	19.78
50	minus 814B	-1.03	19.98	19.95
Mean of all temperatures.....			19.95	

^a Interval nominally correct at 0° C. ^b Interval nominally correct at 20° C.

45 residuals range from 0.02 to 0.20 μ and the average, neglecting sign, is 0.08 μ . The probable error of the calculated value of the differences in the corrections between any two bars, *r*, is given by the formula³

$$r = 0.6745 \sqrt{\frac{\sum v^2}{N_i(N - N_i)}}, \quad (2)$$

where $\sum v^2$ is the sum of the squares of the residuals; *N*, the number of observational equations; and *N_i*, the number of unknowns. In the example considered, *N_i* is equal to 9 and *N* equal to 45. For the ease given in table 3, *r* is calculated to be ± 0.03 .

³ T. W. Wright and J. F. Hayford, The adjustment of observations, p. 133, 137, 143, (1906).

During the period from 1933 to 1953, inclusive, seven series of determinations of the corrections to different groups of the 10-m bars under consideration were made. These determinations were made with groups of 7, 6, 7, 8, 9, 6, and 10 bars. All observations were made at approximately 20° C, and the results reduced to 20° C. The reductions of the observations were carried out by the method of least squares in all cases. The corrections to the total lengths of the bars, together with the new corrections for the prototype meter bars determined at the BIPM and authorized in 1933 by the Eighth General (International) Conference on Weights and Measures, are given in table 4.

The results of any auxiliary comparisons, made primarily because they served as checks in the interim between fundamental determinations, are not included in the table.

Several points should be noted in considering the data in table 4. The seven values determined for the corrections to meter 4 are consistently smaller than the value reported by BIPM, whereas 6 of the 7 values determined for meter 21 and 4 of the 5 values determined for meter 12, before regraduation, are larger than the values reported by BIPM. The differences between the corrections obtained by NBS and those reported by BIPM for prototype meters 4, 12, and 21 are -0.22, +0.22, and +0.15 μ , respectively. If we disregard the 1936 and 1938 values for meters 12 and 21, respectively, the maximum range in the NBS corrections is 0.25 μ . The maximum range in the corrections for meter 4 is 0.22 μ .

Figure 2 shows the change in the determined correction to the total length of each of the two prototype meter bars, 4 and 21, from 1933 to 1953, inclusive, and of meter 12 from 1933 to 1947, inclusive, and from 1949 to 1953, inclusive.

From the above data it appears that there is considerable evidence that these three bars may have changed in length, two lengthening and one shortening, by about 0.2 μ since first received. It is rather difficult to state to what extent the differences between the BIPM and NBS values represent changes in the actual length of the bars. As the measurements at the NBS were made under most favorable conditions and with extreme care, it is believed that the NBS is justified in using the latest determinations instead of those obtained several decades ago, as representing the true value for the corrections to be applied to the respective meter bars when calibrating line standards of length submitted to the Bureau.

Referring again to table 4, it will be noted that the five laboratory standards of length, meters 39, 153R, 752, 814B, and 50, have changed in length during the 20-year period by a much greater amount than have the platinum-iridium meters. The three nickel-steel meters, 39, 153R, and 814B, and stainless steel meter 50 have continually elongated during this period, but Fix Invar meter 752 has shortened. The magnitudes of the changes in length between successive determinations, however, have generally

TABLE 3. Calculations of corrections to meter bars at 20° C

(1953 series of comparisons, using all 45 values for observed differences)

Directly observed differences of A-B are taken from table 2. Calculated ("most probable") differences for A-B, given in parentheses, are derived by taking the differences of $\Sigma/10$; e. g., meter 4-meter 27 = +84.97 - (+89.26) = -4.29 μ , meter 12a-meter 4 = +90.22 - (+84.97) = +5.25 μ , and meter 12b-meter 12a = -82.82 - (+90.22) = -173.04 μ . Correction to meter 27 = +171.66 μ at 20° C (BIPM certificate). Correction to bar = correction to meter 27 + (bar-27). Length of bar = 1 m + correction. All values expressed in microns.

B \ A	A										Residuals, v
	27	4	12a ^a	12b ^b	21	39	153R	752	814B	50	
27	0	-4.33	+0.87	-171.93	+5.11	-177.13	-23.70	-179.80	-170.34	-171.40	-0.04
		(-4.29)	(+0.96)	(-172.08)	(+5.16)	(-177.11)	(-23.75)	(-179.75)	(-170.32)	(-171.42)	-0.09
4	+4.33	0	+5.36	-167.68	+9.36	-172.88	-19.50	-175.53	-165.99	-167.19	+0.15
			(+5.25)	(-167.79)	(+9.45)	(-172.82)	(-19.46)	(-175.46)	(-166.03)	(-167.13)	-0.05
12a	-0.87	-5.36	0	-173.01	+4.33	-177.99	-24.68	-180.65	-171.40	-172.56	-0.02
				(-173.04)	(+4.20)	(-178.07)	(-24.71)	(-180.71)	(-171.28)	(-172.38)	+0.05
12b	+171.93	+167.68	-173.01	0	+177.36	-5.01	+148.40	-7.62	+1.96	+0.50	-0.05
					(+177.24)	(- 5.03)	(+148.33)	(-7.67)	(+1.76)	(+0.66)	+0.02
21	-5.11	-9.36	-4.33	-177.36	0	-182.36	-28.80	-184.94	-175.46	-176.45	+0.11
						(-182.27)	(-28.91)	(-184.91)	(-175.48)	(-176.58)	+0.11
39	+177.13	+172.88	+177.99	+5.01	+182.36	0	+153.20	-2.66	+6.96	+5.66	-0.09
							(+153.36)	(-2.64)	(+6.79)	(+5.69)	-0.06
153R	+23.70	+19.50	+24.68	-148.40	+28.80	-153.20	0	-155.86	-146.68	-147.62	+0.03
								(-156.00)	(-146.57)	(-147.67)	+0.13
752	+179.80	+175.53	+180.65	+7.62	+184.94	+2.66	+155.86	0	+9.55	+8.46	-0.02
									(+9.43)	(+8.33)	+0.13
814B	+170.34	+165.99	+171.40	-1.96	+175.46	-6.96	+146.68	-9.35	0	-1.03	-0.03
										(-1.10)	+0.14
50	+171.40	+167.19	+172.56	-0.50	+176.45	-5.66	+147.62	-8.46	+1.03	0	-0.11
											+0.05
Σ	+892.65	+849.72	+902.19	-828.21	+944.17	-878.53	+655.08	-904.87	-810.57	-821.63	+0.08
$\Sigma/10$	+89.26	+84.97	+90.22	-82.82	+94.42	-87.85	+65.51	-90.49	-81.06	-82.16	+0.13
Bar-27	0	-4.29	+0.96	-172.08	+5.16	-177.11	-23.75	-179.75	-170.32	-171.42	+0.07
Correction to bar	+171.66	+167.37	+172.62	-0.42	+178.82	-5.45	+147.91	-8.09	+1.34	+0.24	+0.07
											$\Sigma v^2 = 0.4084$
											c.P.E. = ± 0.03

^a Interval nominally correct at 0° C. ^b Interval nominally correct at 20° C. ^c Probable error of the calculated value of the differences in the corrections between any two bars.

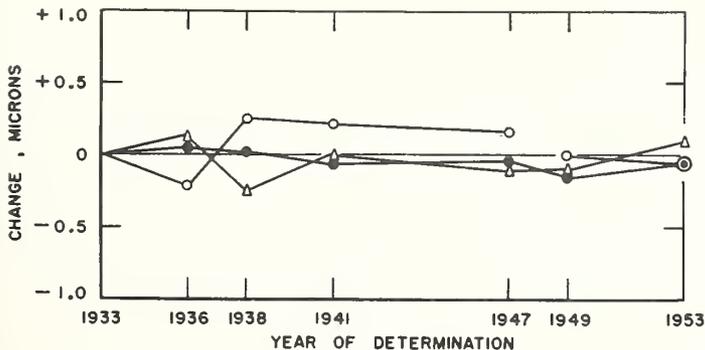


FIGURE 2. Apparent change in lengths of bars. All values from fundamental determinations. ●, Meter 4; ○, meter 12; △, meter 21.

TABLE 4. BIPM and NBS determinations of corrections to meter bars

(1933 to 1953 series of comparisons)

Certified values reported by BIPM at 0° C and reduced to 20° C, using the respective values for their coefficients of linear thermal expansion shown in table 1. Correction to bar=correction to meter 27+(bar-27). All values expressed in microns.

Meter bars	Correction at 20° C									Differences					
	BIPM	NBS								Mean NBS- BIPM	NBS 1953- 1933	NBS 1953- 1938	NBS 1953- 1947	NBS 1953- 1949	NBS 1947- 1933
	1933	1933	1936	1938	1941	1947	1949	1953	Mean						
27	+171.66	+171.66			+171.66	+171.66	+171.66	+171.66	+171.66	+171.66	0.00	0.00			
4 (1874)	+167.62	+167.44	+167.50	+167.44	+167.36	+167.39	+167.28	+167.37	+167.40		-.22	-.07			
12 ^a (1874)	+176.09	+176.23	+176.02	+176.48	+176.45	+176.39				(b)	+176.31				+0.16
12a ^c (1874)										+172.59	+172.60				+0.03
12b ^d (1874)										-0.29	-0.42				-.13
21	+176.56	+176.74	+176.88	+176.48	+176.72	+176.64				+176.67	+176.82	+176.71	+0.15	+0.08	
39		-10.13	-9.17	-8.42	-8.00	-6.66									
153R		+146.32	+146.77	+147.28	+147.38	+147.74	+147.82	+147.91						+4.68	
752		-0.95	-2.73	-3.23	-4.97	-7.05								+1.59	
814B				-0.56	-0.04	+1.10								-7.14	
50						-0.30									+1.90
Probable error ^f		±0.05	±0.11	±0.04	±0.02	±0.02	±0.03	±0.03							±0.54

^a Original graduations. ^b Regraduated. ^c Interval nominally correct at 0° C. ^d Interval nominally correct at 20° C. ^e The mean correction for the 1-m interval on meter 12a, graduated to be nominally correct at 0° C, is +172.60 μ, and the total expansion for a 1-m interval on this bar is 172.75 μ for a change in temperature of 20° C; hence, the correction to the interval is -0.15 μ at 0° C. ^f Probable error of the calculated value of the differences in the corrections between any two bars.

tended to decrease during the period from 1933 to 1953, as shown in figure 3.

How long a period might be required for these standards to become sufficiently stable to eliminate frequent standardizations is difficult to estimate. These laboratory standards are frequently used, between their restandardization, for measurements of moderate precision, where the changes in length over a comparatively short period of time are not significant. When they are used in measurements of the highest precision, they are compared with at least one platinum-iridium standard as a check on their assigned corrections.

The changes in the corrections during the periods between 1933 and 1938, 1938 and 1947, and 1947 and 1953, are shown in table 5.

Meter 39 is one of the first laboratory standards obtained by NBS, and it is of interest to note the average yearly change in length over a period of nearly a half century. The changes in length for this bar during the period from 1907 to 1953 are shown in figure 4. The average changes in length per year, for the first 4, the following 10, and the last 32 years, are +1.2, +0.6, and +0.2 μ, respectively. This shows that the magnitude of the changes in length is gradually becoming smaller.

TABLE 5. Length changes of meter bars

Meter bars	Average changes per year		
	1933 to 1938	1938 to 1947	1947 to 1953
39	+0.3	+0.2	+0.2
153R	+ .2	+ .1	<+ .1
752	-.5	-.4	-.2
814B		+ .2	<+ .1
50			+ .1

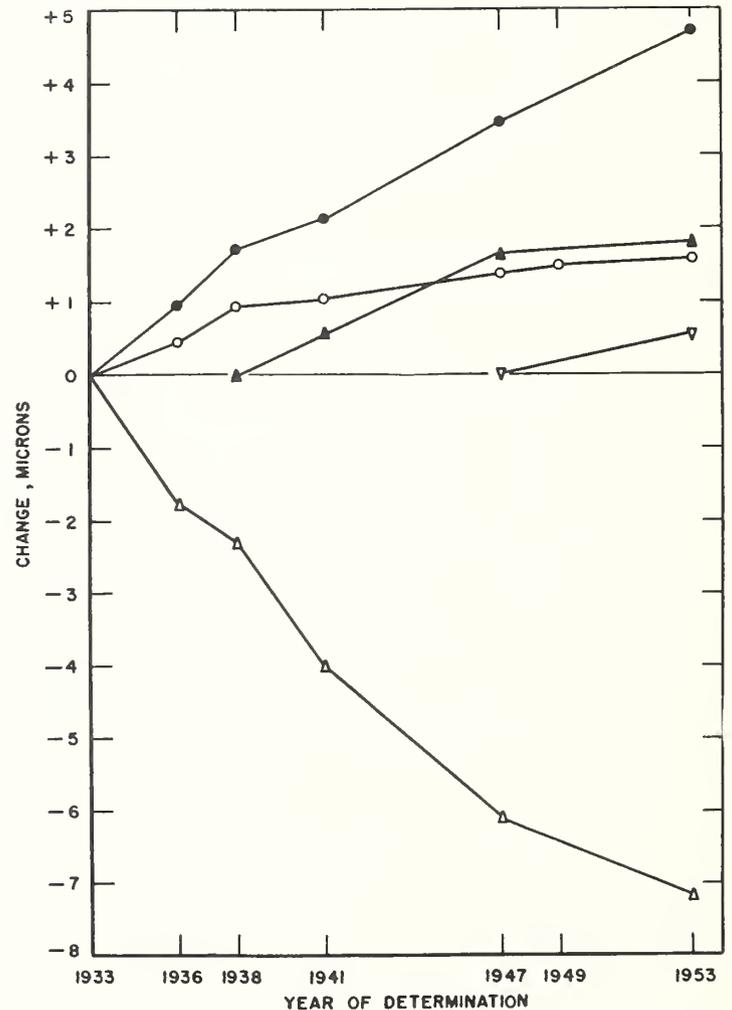


FIGURE 3. Change in lengths of bars.

All values from fundamental determinations. ●, Meter 39; ○, meter 153R; △, meter 752; ▲, meter 814B; ▽, meter 50.

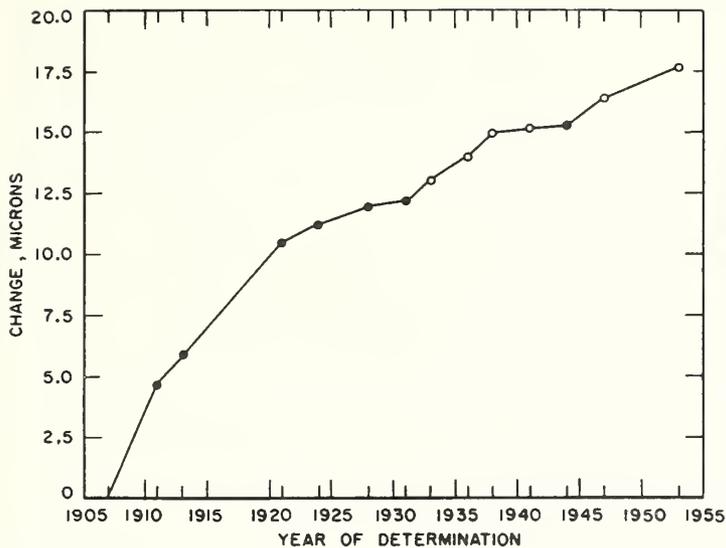


FIGURE 4.—Change in length of meter 39.

○, From fundamental determinations; ●, from check determinations.

As the work required to observe and determine 45 values for the differences between the lengths of a group of 10 standards is rather laborious and consumes considerable time, it was decided to use the information obtained in the 1952 and 1953 series of comparisons for a study of the possibility of using fewer measurements for future intercomparisons of the same number of standards.

According to a scheme,⁴ calculations were made by using 30, 25, and 15 directly observed differences with the remaining differences computed from the observed values. The reduction of these differences, observed and computed, by the method of least squares was carried out by the use of forms (see footnote 2) shown in tables 6, 7, and 8.

The scheme for using 30 directly observed differences and selecting the remaining 15 to be computed is as follows:

Arrange the numbers of the 10-m bars as shown below.

*	27	4	12a	12b
27	*	21	39	153R
4	21	*	752	814B
12a	39	752	*	50
12b	153R	814B	50	*

Then cancel out the rows and columns that contain the number 27; this leaves the numbers 752, 814B, and 50 as the serial numbers of the bars whose differences from meter 27 are to be computed. Restore the rows and columns and next cancel out the rows and columns that contain number 4; this then leaves the numbers 39, 153R, and 50 as the numbers of the bars whose differences from meter 4 are to be computed. This procedure is continued until each number has been cancelled out of its

⁴W. J. Youden and W. S. Connor, New experimental designs for paired observations, *J. Research NBS* **53**, 191 (1954) RP2532.

respective rows and columns and until all the differences between the 15 pairs of bars, whose differences are to be computed from directly observed differences, are determined. To compute these differences, the following method, illustrated by 752-27, was used. First, the mean of the differences between 752 and each of the other bars with which it was directly compared and for which there is a corresponding directly observed comparison of that other bar with 27 was found. Next, the mean of the differences between 27 and each of the others with which it was compared, and for which there is a corresponding directly observed comparison of that other bar with 752, was found. Finally, the mean values were added algebraically. This is the computed value for 752-27, or -179.82μ , as is shown in brackets in table 6. The other bracketed figures shown in table 6 were computed in a similar manner. All further computations to determine the corrections to the bars in this case are the same as in table 3.

The scheme for selecting and using 25 directly observed differences and for leaving the remaining 20 differences to be computed is as follows:

Arrange the numbers of the 10 bars as shown below.

Group 1	---- 27	4	12a	12b	21
Group 2	---- 39	153R	752	814B	50

The 25 directly observed differences to be used are those which are indicated by subtracting each of the numbers in group 1 from each of the numbers in group 2, i. e., $39-27$, $153R-27$, $752-27$, $814B-27$, $50-27$, . . . , $50-21$. The remaining 20 differences are computed in the same manner that was used when 30 directly observed differences were used, and are the bracketed figures shown in table 7. All further computations to determine the corrections to the bars in this case are the same as in table 3.

The scheme for using 15 directly observed differences and computing the remaining 30 differences is the opposite of the scheme for which the 30 directly observed differences were used and the remaining 15 computed. The 30 computed differences were obtained in the same manner that was used where 15 and 20 differences were determined, and are the bracketed figures shown in table 8. All further computations to determine the corrections to the bars in this case are the same as in table 3.

In computing the probable errors in tables 6, 7, and 8, the same formula (see footnote 3) was used as in table 3. This was done for simplicity, although the probable errors thus obtained may be somewhat too small. Alternative methods for computing the differences and probable error are given by Youden and Connor (see footnote 4).

The corrections to the bars obtained in the calibration in which all differences were directly observed, and those obtained when only 30, 25, and 15 were directly observed, are tabulated in table 9. The corrections to the various bars are in good agreement, even though a lesser number of direct differences were observed. This will be true only when all the

observed differences in length between bars are accurately determined.

The values in columns 6 to 8 of table 9 were obtained by taking differences between the corresponding values in columns 3 and 2, 4 and 2, and 5 and 2, respectively. The average difference in the corrections (columns 6 to 8) increases with decrease in the

number (30, 25, and 15) of directly observed differences. The conclusion is that in the comparison of basic standards, a full calibration, where all the differences between bars are directly observed, may be justified. However, for secondary standards or others submitted for calibration, a lesser number of direct comparisons should be sufficient.

TABLE 6. Calculations of corrections to meter bars at 20° C

(1953 series of comparisons, using 30 values for observed differences)

Directly observed differences of A-B are taken from table 2 and are the values not in brackets or parentheses. Values in brackets have been computed from the directly observed differences. All further computations are the same as in table 3. All values expressed in microns.

A \ B	27	4	12a ^a	12b ^b	21	39	153R	752	814B	50	Residuals, <i>v</i>
27	0	-4.33	+0.87	-171.93	+5.11	-177.13	-23.70	[-179.82]	[-170.26]	[-171.48]	0.00 -.09 +.13 -.02 -.04 +.03
		(-4.33)	(+0.96)	(-172.06)	(+5.13)	(-177.09)	(-23.73)	(-179.79)	(-170.34)	(-171.44)	-.03 +.08 -.04 +.07 +.05 -.10
4	+4.33	0	+5.36	-167.68	+9.36	[-172.82]	(-19.35)	-175.53	-165.99	[-167.12]	-.06 +.05 -.07 +.02 -.01 +.01
			(+5.29)	(-167.73)	(+9.46)	(-172.76)	(-19.40)	(-175.46)	(-166.01)	(-167.11)	+.05 -.10 +.06 +.06 -.04 +.10 -.01 -.16
12a ^a	-0.87	-5.36	0	-173.01	[+4.22]	-177.99	[-24.73]	-180.65	[-171.31]	-172.56	-.01 +.07 +.02 -.01 +.01 +.05 +.06 -.04 +.10 -.01 -.16
		+167.68	+173.01	(-173.02)	(+4.17)	(-178.05)	(-24.69)	(-180.75)	(-171.30)	(-172.40)	+.07 +.02 -.01 +.01 +.05 +.06 -.04 +.10 -.01 -.16
12b ^b	+171.93			0	[+177.18]	[-5.04]	+148.40	[-7.71]	+1.96	+0.50	-.01 +.07 +.02 -.01 +.01 +.05 +.06 -.04 +.10 -.01 -.16
					(+177.19)	(-5.03)	(+148.33)	(-7.73)	(+1.72)	(+0.62)	+.07 +.02 -.01 +.01 +.05 +.06 -.04 +.10 -.01 -.16
21	-5.11	-9.36	[-4.22]	[-177.18]	0	-182.36	-28.80	-184.94	-175.46	[-176.52]	-.01 +.07 +.02 -.01 +.01 +.05 +.06 -.04 +.10 -.01 -.16
						(-182.22)	(-28.86)	(-184.92)	(-175.47)	(-176.57)	+.07 +.02 -.01 +.01 +.05 +.06 -.04 +.10 -.01 -.16
39	+177.13	[+172.82]	+177.99	[+5.04]	+182.36	0	+153.20	-2.66	[+6.70]	+5.66	-.12 -.14 +.06 -.02 +.01 +.05 -.16 +.04 -.05 +.01 +.03 -.07 +.09 -.10 +.11 +.07
							(+153.36)	(-2.70)	(+6.75)	(+5.65)	+.06 -.02 +.01 +.05 -.16 +.04 -.05 +.01 +.03 -.07 +.09 -.10 +.11 +.07
153R	+23.70	[+19.35]	[+24.73]	-148.40	+28.80	-153.20	0	[-156.03]	-146.68	-147.62	+.04 -.05 +.01 +.03 -.07 +.09 -.10 +.11 +.07
								(-156.06)	(-146.61)	(-147.71)	+.01 +.03 -.07 +.09 -.10 +.11 +.07
752	[+179.82]	+175.53	+180.65	[+7.71]	+184.94	+2.66	[+156.03]	0	+9.35	+8.46	+.09 -.10 +.11 +.07
									(+9.45)	(+8.35)	+.09 -.10 +.11 +.07
814B	[+170.26]	+165.99	[+171.31]	-1.96	+175.46	[-6.70]	+146.68	-9.35	0	-1.03	Σ <i>v</i> ² =0.2836 c P.E.=±0.03
										(-1.10)	Σ <i>v</i> ² =0.2836 c P.E.=±0.03
50	[+171.48]	[+167.12]	+172.56	-0.50	[+176.52]	-5.66	+147.62	-8.46	+1.03	0	Σ <i>v</i> ² =0.2836 c P.E.=±0.03
											Σ <i>v</i> ² =0.2836 c P.E.=±0.03
Σ	+892.67	+849.44	+902.26	-827.91	+943.95	-878.24	+655.35	-905.15	-810.66	-821.71	
Σ/10	+89.27	+84.94	+90.23	-82.79	+94.40	-87.82	+65.54	-90.52	-81.07	-82.17	
Bar-27	0	-4.33	+0.96	-172.06	+5.13	-177.09	-23.73	-179.79	-170.34	-171.44	
Correction to bar	+171.66	+167.33	+172.62	-0.40	+176.79	-5.43	+147.93	-8.13	+1.32	+0.22	

^a Interval nominally correct at 0° C. ^b Interval nominally correct at 20° C. ^c Probable error of the calculated value of the differences in the corrections between any two bars.

TABLE 8. Calculations of corrections to meter bars at 20° C.

(1953 series of comparisons, using 15 values for observed differences)

Directly observed differences of A-B are taken from table 2 and are the values not in brackets or parentheses. Values in brackets have been computed from the directly observed differences. All further computations are the same as in table 3. All values expressed in microns.

A \ B	27	4	12a ^a	12b ^b	21	39	153R	752	814B	50	Residuals, v
27-----	0	[-4.21] (-4.30)	[+1.06] (+0.89)	[-172.18] (-172.20)	[+5.05] (+5.14)	[-177.30] (-177.24)	[-23.94] (-23.83)	-179.80 (-179.77)	-170.34 (-170.38)	-171.40 (-171.40)	+0.09 +.17 +.02 -.09 -.06 -.11 -.03 +.04 +.00 +.01 +.03 -.18 +.06 +.03 +.11 +.16 -.09 +.06 +.00 -.23 +.04 +.12 -.13 +.17 +.02 +.03 -.13 -.05 +.13 +.11 +.01 -.04 -.07 -.21 +.09 -.03 -.08 +.10 -.15 +.08 -.17 -.12 +.07 +.03 -.04
4-----	[+4.21]	0	[+5.18] (+5.19)	[-167.87] (-167.90)	[+9.26] (+9.44)	-172.88 (-172.94)	-19.50 (-19.53)	[-175.36] (-175.47)	[-165.92] (-166.08)	-167.19 (-167.10)	-.00 -.01 +.03 -.18 +.06 +.03 +.11 +.16 -.09 +.06 +.00 -.23 +.04 +.12 -.13 +.17 +.02 +.03 -.13 -.05 +.13 +.11 +.01 -.04 -.07 -.21 +.09 -.03 -.08 +.10 -.15 +.08 -.17 -.12 +.07 +.03 -.04
12a ^a -----	[-1.06]	[-5.18]	0	[-173.03] (-173.09)	+4.33 (+4.25)	[-178.36] (-178.13)	-24.68 (-24.72)	[-180.54] (-180.66)	-171.40 (-171.27)	[-172.12] (-172.29)	+.03 +.11 +.16 -.09 +.06 +.00 -.23 +.04 +.12 -.13 +.17 +.02 +.03 -.13 -.05 +.13 +.11 +.01 -.04 -.07 -.21 +.09 -.03 -.08 +.10 -.15 +.08 -.17 -.12 +.07 +.03 -.04
12b ^b -----	[+172.18]	[+167.87]	[+173.03]	0	+177.36 (+177.34)	-5.01 (-5.04)	[+148.24] (+148.37)	-7.62 (-7.57)	[+1.95] (+1.82)	[+0.91] (+0.80)	+.03 +.11 +.16 -.09 +.06 +.00 -.23 +.04 +.12 -.13 +.17 +.02 +.03 -.13 -.05 +.13 +.11 +.01 -.04 -.07 -.21 +.09 -.03 -.08 +.10 -.15 +.08 -.17 -.12 +.07 +.03 -.04
21-----	[-5.05]	[-9.26]	-4.33	-177.36	0	[-182.37] (-182.38)	[-29.01] (-28.97)	[-184.98] (-184.91)	[-175.73] (-175.52)	-176.45 (-176.54)	+.03 +.11 +.16 -.09 +.06 +.00 -.23 +.04 +.12 -.13 +.17 +.02 +.03 -.13 -.05 +.13 +.11 +.01 -.04 -.07 -.21 +.09 -.03 -.08 +.10 -.15 +.08 -.17 -.12 +.07 +.03 -.04
39-----	[+177.30]	+172.88	[+178.36]	+5.01	[+182.37]	0	[+153.38] (+153.41)	[-2.61] (-2.53)	+6.96 (+6.86)	[+5.69] (+5.84)	+.03 +.11 +.16 -.09 +.06 +.00 -.23 +.04 +.12 -.13 +.17 +.02 +.03 -.13 -.05 +.13 +.11 +.01 -.04 -.07 -.21 +.09 -.03 -.08 +.10 -.15 +.08 -.17 -.12 +.07 +.03 -.04
153R-----	[+23.94]	+19.50	+24.68	[-148.24]	[+29.01]	[-153.38]	0	-155.86 (-155.94)	[-146.72] (-146.55)	[-147.69] (-147.57)	+.03 +.11 +.16 -.09 +.06 +.00 -.23 +.04 +.12 -.13 +.17 +.02 +.03 -.13 -.05 +.13 +.11 +.01 -.04 -.07 -.21 +.09 -.03 -.08 +.10 -.15 +.08 -.17 -.12 +.07 +.03 -.04
752-----	+179.80	[+175.36]	[+180.54]	+7.62	[+184.98]	[+2.61]	+155.86	0	[+9.46] (+9.39)	[+8.40] (+8.37)	+.03 +.11 +.16 -.09 +.06 +.00 -.23 +.04 +.12 -.13 +.17 +.02 +.03 -.13 -.05 +.13 +.11 +.01 -.04 -.07 -.21 +.09 -.03 -.08 +.10 -.15 +.08 -.17 -.12 +.07 +.03 -.04
814B-----	+170.34	[+165.92]	+171.40	[-1.95]	[+175.73]	-6.96	[+146.72]	[-9.46]	0	[-1.06] (-1.02)	Σp ² =0.4775 c P. E. = ±0.03
50-----	+171.40	+167.19	[+172.12]	[-0.91]	+176.45	[-5.69]	[+147.69]	[-8.40]	[+1.06]	0	
Σ-----	+893.06	+850.07	+902.04	-828.91	+944.54	-879.34	+654.76	-904.63	-810.68	-820.91	
Σ/10-----	+89.31	+85.01	+90.20	-82.89	+94.45	-87.93	+65.48	-90.46	-81.07	-82.09	
Bar-27-----	0	-4.30	+0.89	-172.20	+5.14	-177.24	-23.83	-179.77	-170.38	-171.40	
Correction to bar-----	+171.66	+167.36	+172.55	-0.54	+176.80	-5.58	+147.83	-8.11	+1.28	+0.26	

^a Interval nominally correct at 0° C.

^b Interval nominally correct at 20° C.

^c Probable error of the calculated value of the differences in the corrections between any two bars.

TABLE 9. Summary of corrections to the total length of meter bars, using different numbers of directly observed differences

(1953 series of comparisons)

1	2	3	4	5	6	7	8
Meter bars	Corrections at 20° C for various numbers of direct differences				Differences in corrections		
	45	30	25	15	30-45	25-45	15-45
	μ	μ	μ	μ	μ	μ	μ
27-----	+171.66	+171.66	+171.66	+171.66	0	0	0
4-----	+167.37	+167.33	+167.41	+167.36	-0.04	+0.04	-0.01
12a ^a -----	+172.62	+172.62	+172.65	+172.55	.00	+0.03	-0.07
12b ^b -----	-0.42	-0.40	-0.46	-0.54	+0.02	-0.04	-0.12
21-----	+176.82	+176.79	+176.79	+176.80	-0.03	-0.03	-0.02
39-----	-5.45	-5.43	-5.46	-5.56	+0.02	-0.01	-0.11
153R-----	+147.91	+147.93	+147.95	+147.83	+0.02	+0.04	-0.08
752-----	-8.09	-8.13	-8.10	-8.11	-0.04	-0.01	-0.02
814B-----	+1.34	+1.32	+1.36	+1.28	-0.02	+0.02	-0.06
50-----	+0.24	+0.22	+0.19	+0.26	-0.02	-0.05	+0.02
Average difference-----					±0.02	±0.03	±0.05

^a Interval nominally correct at 0° C.
^b Interval nominally correct at 20° C.

5. Calibration of the Subintervals

Methods for the calibration of the subintervals of a standard of length have been described in Bureau Circular 329, Calibration of a Divided Scale, by Lewis V. Judson. In the recent calibrations of the subdivided meter bars, the methods described in Circular 329 have been used. In certain cases the method⁵ of double or cross calibration has been used.

The calibration of the subintervals of subdivided meter bars 4, 39, 153R, 752, and 814B was carried out in four steps, namely, the decimeter intervals, the centimeter intervals of all of the decimeter intervals, the millimeter intervals of the 1- and 92-cm intervals, and the 1/10-m intervals at each end of the bars.

Because of these calibrations, it is possible to compare lengths from 0.1 mm up to and including 1 m with calibrated intervals on any of these five standards.

The results are expressed as "elements of calibration", that is, the corrections which the subintervals will have at a temperature at which the whole interval has its nominal length. Thus, it is only necessary to distribute proportionately any newly determined total correction to the elements of calibration in order to obtain the new corrections to the subintervals.

5.1. Calibration of the Decimeter Intervals

The computed values for the several elements of calibration of the decimeter intervals of the 5 meter bars are given in table 10. The probable errors of the calculated values for the elements of calibration

⁵ C. E. Guillaume, L'étalonnage des échelles divisées, Travaux et Mémoires du Bureau International des Poids et Mesures 13, 1 (1907).

TABLE 10. Elements of calibration of meter bars to decimeters

(1953 series of calibrations)

Elements of calibration=corrections to intervals, assuming that the meter bars have their nominal lengths. Relative length of interval=nominal length+element of calibration.

Interval	Elements of calibration of meter bars—				
	4 ^a	39	153R	752	*814B
	μ	μ	μ	μ	μ
0 to 1-----	-0.46	-7.36	+1.41	+1.91	-0.76
0 to 2-----	-3.16	-1.39	+3.86	+4.18	-0.54
0 to 3-----	+0.06	+0.98	+3.51	+3.60	-0.74
0 to 4-----	-0.60	+2.26	+4.09	+2.43	-0.88
0 to 5-----	-2.36	-1.61	+5.36	+1.86	-0.48
0 to 6-----	-2.28	+2.12	+2.37	+1.87	-0.94
0 to 7-----	-1.58	-0.06	+2.82	+1.78	-0.14
0 to 8-----	+0.08	-5.87	+4.84	+0.60	-1.56
0 to 9-----	-1.64	+0.94	+2.82	+0.50	-1.25
0 to 10-----	0.00	.00	0.00	.00	0.00
Probable error range	{ ±0.02 to ±0.03	{ ±0.02 to ±0.03	{ ±0.02 to ±0.03	{ ±0.02 to ±0.03	{ ±0.02 to ±0.03

^a Mean of two calibrations.

are also shown. They were computed by using the formula⁶

$$r_x = 0.6745 \sqrt{\frac{\sum v^2}{K}}, \quad (3)$$

where $\sum v^2$ is the sum of the squares of the differences between the observed and computed values for the elements of calibration. As there are $(N-1)(N-2)$ equations of condition, K is $[(N-1)(N-2)]/2$, the value r_x is the probable error of any x , and x_2 is the element of calibration to the first interval, x_3 is the element of calibration to the first two intervals, x_4 is the element of calibration to the first three intervals, etc.

That the differences between the observed and calculated values for the elements of calibration are relatively small is indicated by the small probable errors, and that all of the measurements are apparently of nearly equal accuracy, regardless of the character of the lines on the various bars, is indicated by the probable errors being of the same magnitude.

It is of interest to note how well the values for the elements of calibration of the decimeter intervals determined during the past 25 years agree with each other, and also to note the differences between the mean of these values and those obtained 50 years earlier. For this purpose, meter 39 was selected as an example, and the values determined in 1903 and those determined in 1928, 1931, 1932, 1947, and 1953 are given in table 11.

The maximum deviations from the mean of five determinations from 1928 to 1953, inclusive, range from 0.04 to 0.17 μ , the average being 0.11 μ .

The probable cause of some of the large differences between the mean of the 1928 to 1953 determinations and those determined in 1903 is that the old Saxton

⁶ O. J. Broch, Note sur l'étalonnage des sous-divisions d'une règle, Travaux et Mémoires du Bureau International des Poids et Mesures 5, 1 (1886).

TABLE 11. Elements of calibration of meter 39 to decimeters

(1903 to 1953 series of calibrations)

Elements of calibration=corrections to intervals, assuming that the meter bar has its nominal length.
Relative length of interval=nominal length+element of calibration.

Interval	Elements of calibration in the year							Differences	
	1903	1928	1931	1932	1947	1953	Mean	Maximum deviation from mean	Mean-1903
	μ	μ	μ	μ	μ	μ	μ	μ	μ
0 to 1-----	-7.43	-7.20	-7.38	-7.38	-7.20	-7.36	-7.30	0.10	+0.13
0 to 2-----	-1.45	-1.41	-1.48	-1.52	-1.28	-1.39	-1.42	.14	+ .03
0 to 3-----	+1.10	+1.02	+0.95	+1.06	+0.87	+0.98	+0.98	.11	- .12
0 to 4-----	+2.29	+2.29	+2.25	+2.30	+2.23	+2.26	+2.27	.04	- .02
0 to 5-----	-1.48	-1.59	-1.56	-1.54	-1.48	-1.61	-1.56	.08	- .08
0 to 6-----	+2.42	+2.27	+2.25	+2.25	+2.36	+2.12	+2.25	.13	- .17
0 to 7-----	+0.29	+0.05	+0.02	+0.02	+0.22	-0.06	+0.05	.17	- .24
0 to 8-----	-6.13	-5.75	-5.85	-5.84	-5.62	-5.87	-5.79	.17	+ .34
0 to 9-----	+0.72	+0.97	+0.95	+0.86	+1.11	+0.94	+0.97	.14	+ .25
0 to 10-----	.00	.00	.00	.00	.00	.00	.00	.00	.00
Probable error range-----	± 0.10 to ± 0.11	± 0.03 to ± 0.04	± 0.02 to ± 0.03	± 0.02 to ± 0.03	± 0.03 to ± 0.04	± 0.03 to ± 0.04	-----	----	-----

comparator used in 1903 was not entirely adequate for the work. It is fortunate, however, that the values for the elements of calibration thereby determined are in fairly good agreement with those obtained by the use of more recent and better equipment.

5.2. Calibration of the Centimeter Intervals

In determining the elements of calibration to the centimeter subintervals, the double- or cross-calibration method was used, that is, a comparison of each interval with the others in all possible combinations; namely, the centimeter intervals of the first and sixth decimeters, those of the second and seventh, the third and eighth, the fourth and ninth, and the fifth and tenth.

5.3. Calibration of the Millimeter Intervals

The elements of calibration of the millimeter intervals of the centimeter intervals, 0 to 1 cm and 91 to 92 cm, were obtained by the same method as was used to determine the elements of calibration of the centimeter subintervals.

For the sake of brevity the results of the determinations of the elements of calibration of the subintervals of only meter 4 are reported in this paper. The two NBS calibrations for the decimeter, centimeter, and millimeter subintervals on meter 4, together with those reported in 1907 for the same subintervals by BIPM, are shown in table 12.

The probable errors for the elements of calibration of any centimeter or millimeter subinterval range from ± 0.03 to $\pm 0.07 \mu$, and were computed by the use of the formula ⁷

$$r_x = 0.6745 \frac{\sqrt{2}}{(N-1)\sqrt{N-1}} \sqrt{\sum v^2}, \quad (4)$$

⁷ T. W. Wright and J. F. Hayford, The adjustment of observations, p. 133 and 142 (1906); Dziobek, Ueber die Ermittlung der inneren Theilungsfehler zweier Maassstäbe nach der Methode des Durchschiebens, Wiss. Abhandl. Kaiserlichen Normal-Messungs-Kommission 4, 1 (1903).

TABLE 12. Elements of calibration of meter 4 to millimeters at BIPM and NBS

(NBS 1953 series of calibrations)

Elements of calibration=corrections to intervals, assuming that the meter bar has its nominal length. Relative length of interval=nominal length+element of calibration.

Interval	Elements of calibration			NBS mean ^a	NBS maximum deviation from mean	NBS-BIPM
	BIPM	NBS				
	μ	μ	μ	μ	μ	μ
0 to 1-----	-0.2	+0.01	-0.15	-0.07	0.08	+0.1
0 to 2-----	+ .3	+ .42	+ .34	+ .38	.04	+ .1
0 to 3-----	- .8	- .56	- .67	- .62	.06	+ .2
0 to 4-----	-1.1	- .72	- .78	- .75	.03	+ .4
0 to 5-----	-1.3	-1.06	-1.05	-1.06	.01	+ .2
0 to 6-----	-1.6	-1.31	-1.24	-1.28	.04	+ .3
0 to 7-----	-1.8	-1.43	-1.41	-1.42	.01	+ .4
0 to 8-----	-1.7	-1.45	-1.46	-1.46	.01	+ .2
0 to 9-----	-1.8	-1.50	-1.55	-1.52	.03	+ .3
0 to 10-----	-2.2	-1.93	-2.02	-1.98	.05	+ .2
0 to 20-----	-2.5	-2.20	-2.37	-2.28	.09	+ .2
0 to 30-----	-2.4	-2.30	-2.37	-2.34	.04	+ .1
0 to 40-----	-0.8	-1.03	-1.09	-1.06	.03	- .3
0 to 50-----	- .7	-0.96	-0.90	-0.93	.03	- .2
0 to 60-----	- .9	-1.12	-1.22	-1.17	.05	- .3
0 to 70-----	-1.4	-1.60	-1.53	-1.56	.04	- .2
0 to 80-----	-1.2	-1.52	-1.36	-1.44	.08	- .2
0 to 90-----	-1.1	-1.24	-1.22	-1.23	.01	- .1
0 to 100-----	-0.5	-0.42	-0.49	-0.46	.04	.0
0 to 110-----	- .6	- .42	- .62	- .52	.10	+ .1
0 to 120-----	- .4	- .31	- .53	- .42	.11	.0
0 to 130-----	-1.4	-1.28	-1.48	-1.38	.10	.0
0 to 140-----	-1.0	-0.98	-1.17	-1.08	.10	-0.1
0 to 150-----	-2.0	-2.02	-2.16	-2.09	.07	- .1
0 to 160-----	-3.7	-3.48	-3.66	-3.57	.09	+ .1
0 to 170-----	-2.4	-2.15	-2.36	-2.26	.11	+ .1
0 to 180-----	-1.3	-1.29	-1.39	-1.34	.05	.0
0 to 190-----	-2.3	-2.10	-2.16	-2.13	.03	+ .2
0 to 200-----	-3.6	-3.11	-3.20	-3.16	.05	+ .4
0 to 210-----	-3.0	-2.49	-2.64	-2.56	.08	+ .4
0 to 220-----	-2.1	-1.63	-1.82	-1.72	.10	+ .4
0 to 230-----	-2.0	-1.50	-1.85	-1.68	.18	+ .3
0 to 240-----	-2.6	-1.95	-2.24	-2.10	.15	+ .5
0 to 250-----	-2.2	-1.62	-2.05	-1.84	.22	+ .4
0 to 260-----	-1.9	-1.20	-1.60	-1.40	.20	+ .5
0 to 270-----	-2.0	-1.41	-1.77	-1.59	.18	+ .4
0 to 280-----	-2.6	-2.16	-2.47	-2.32	.16	+ .3
0 to 290-----	-1.8	-1.24	-1.53	-1.38	.15	+ .4
0 to 300-----	0.0	+0.12	+0.01	+0.06	.06	+ .1
0 to 310-----	+ .4	+ .60	+ .58	+ .59	.06	+ .2
0 to 320-----	+ .5	+ .77	+ .67	+ .72	.05	+ .2
0 to 330-----	+1.6	+2.02	+1.86	+1.94	.08	+ .3
0 to 340-----	+0.2	+0.27	+0.17	+0.22	.05	.0
0 to 350-----	-2.9	-2.63	-2.88	-2.76	.13	+ .1
0 to 360-----	-3.2	-2.73	-2.90	-2.82	.09	+ .4

TABLE 12. Elements of calibration of meter 4 to millimeters at BIPM and NBS—Continued

Interval	Elements of calibration			NBS mean ^a	NBS maximum deviation from mean	NBS-BIPM
	BIPM	NBS				
<i>mm</i>	μ	μ	μ	μ	μ	μ
0 to 370	-1.7	-1.20	-1.46	-1.33	.13	+ .4
0 to 380	-0.3	+0.06	+0.07	+0.06	.01	+ .4
0 to 390	-1	+ .17	+ .20	+ .18	.02	+ .3
0 to 400	- .8	- .59	- .61	- .60	.01	+ .2
0 to 410	- .7	- .38	- .53	- .46	.08	+ .2
0 to 420	- .4	- .21	- .21	- .21	.00	+ .2
0 to 430	.0	+ .24	+ .18	+ .21	.03	+ .2
0 to 440	+ .4	+ .60	+ .57	+ .58	.02	+ .2
0 to 450	- .2	- .16	- .10	- .13	.03	+ .1
0 to 460	+ .2	+ .47	+ .24	+ .36	.12	+ .2
0 to 470	- .4	- .26	- .44	- .35	.09	.0
0 to 480	+ .3	+ .48	+ .36	+ .42	.06	+ .1
0 to 490	- .2	- .08	- .20	- .14	.06	.0
0 to 500	- .2	- .28	- .24	- .26	.06	- .1
0 to 510	- .5	- .28	- .38	- .33	.05	+ .2
0 to 520	- .7	- .50	- .57	- .54	.04	+ .2
0 to 530	- .9	- .81	- .80	- .80	.01	+ .1
0 to 540	- .3	- .26	- .27	- .27	.05	+ .3
0 to 550	- .5	- .25	- .21	- .23	.02	+ .3
0 to 560	- .2	- .80	- .76	- .78	.02	+ .3
0 to 570	- .5	- .32	- .22	- .27	.05	+ .2
0 to 580	- .2	- .83	- .80	- .82	.02	+ .3
0 to 590	- .3	- .05	- .09	- .07	.02	+ .2
0 to 600	- .5	- .22	- .35	- .28	.07	+ .2
0 to 610	- .3	- .09	- .05	- .07	.02	+ .1
0 to 620	- .3	- .61	- .67	- .64	.03	+ .3
0 to 630	- .4	- .05	- .22	- .14	.09	+ .3
0 to 640	- .2	- .94	- .16	- .05	.11	+ .2
0 to 650	- .2	- .79	- .98	- .88	.10	+ .3
0 to 660	- .3	- .04	- .11	- .08	.04	+ .2
0 to 670	+ .4	+ .70	+ .56	+ .63	.07	+ .2
0 to 680	- .6	- .21	- .29	- .25	.04	+ .4
0 to 690	- .3	- .83	- .80	- .82	.02	+ .3
0 to 700	- .2	- .55	- .61	- .58	.03	+ .4
0 to 710	- .1	- .63	- .88	- .76	.13	+ .3
0 to 720	+ .7	+ .24	+ .00	+ .12	.12	+ .4
0 to 730	- .3	+ .14	- .04	+ .05	.09	+ .4
0 to 740	- .3	- .14	- .26	- .20	.06	+ .1
0 to 750	- .6	- .21	- .43	- .32	.11	+ .3
0 to 760	- .1	+ .02	- .17	- .08	.10	.0
0 to 770	+ .7	+ .86	+ .70	+ .78	.08	+ .1
0 to 780	+ .6	+ .60	+ .50	+ .55	.05	.0
0 to 790	+ .8	+ .13	+ .93	+ .103	.10	+ .2
0 to 800	.0	+ .01	+ .06	+ .08	.03	+ .1
0 to 810	+ .7	+ .76	+ .75	+ .76	.01	+ .1
0 to 820	- .5	- .14	- .34	- .24	.10	+ .3
0 to 830	- .5	- .35	- .55	- .45	.10	.0
0 to 840	- .5	- .21	- .38	- .30	.09	+ .2
0 to 850	+ .3	+ .53	+ .33	+ .43	.10	+ .1
0 to 860	- .4	- .24	- .38	- .31	.07	+ .1
0 to 870	- .1	- .04	- .18	- .11	.07	- .1
0 to 880	- .8	- .65	- .69	- .67	.02	+ .1
0 to 890	- .4	- .36	- .36	- .36	.00	.0
0 to 900	- .2	- .61	- .66	- .64	.03	- .4
0 to 910	- .3	- .99	- .79	- .89	.10	+ .2
0 to 911	- .3	- .12	- .27	- .24	.18	+ .2
0 to 912	- .3	- .30	- .81	- .296	.15	+ .2
0 to 913	- .3	- .89	- .29	- .284	.05	+ .2
0 to 914	- .3	- .296	- .280	- .288	.08	+ .3
0 to 915	- .3	- .30	- .309	- .310	.01	+ .2
0 to 916	- .2	- .67	- .59	- .63	.04	+ .2
0 to 917	- .3	- .73	- .73	- .73	.00	+ .3
0 to 918	- .3	- .28	- .24	- .276	.02	+ .2
0 to 919	- .2	- .58	- .55	- .56	.02	+ .1
0 to 920	- .2	- .49	- .240	- .244	.05	+ .2
0 to 930	- .2	- .13	- .12	- .12	.01	+ .2
0 to 940	- .2	- .44	- .48	- .46	.02	+ .1
0 to 950	- .3	- .67	- .70	- .68	.02	.0
0 to 960	- .2	- .48	- .47	- .48	.01	+ .1
0 to 970	- .1	- .63	- .56	- .60	.04	+ .1
0 to 980	- .1	- .68	- .65	- .66	.02	+ .1
0 to 990	- .2	- .98	- .203	- .203	.05	+ .3
0 to 1000	.0	.00	.00	.00	.00	.0

^a Mean of two calibrations.

where Σd^2 is the sum of the squares of the differences between the observed and computed values for the elements of calibration, N , the number of subintervals into which any considered interval is divided, and r_x is the probable error of the element of calibration of any subinterval.

The deviations from the mean of two determinations for all of the elements of calibration for meter 4 range from 0.00 to 0.22 μ , the average being 0.06 μ .

The discrepancies between the values originally reported by BIPM for these elements of calibration and the values determined by NBS vary from -0.3 to +0.5 μ . However, over a period of years repeated determinations of the elements of calibration of the decimeter subintervals on meter 4 have shown highly consistent results, and it is indicated, therefore, that the NBS values are very nearly the present true values for all of the elements of calibration for the subintervals. Taking into consideration that the NBS values were determined by using a comparator of more modern design, with more accurate auxiliary equipment and under more favorable conditions than were available in 1907 at BIPM, it is believed that the NBS is justified in adopting these new values as representing more nearly the true values than those originally determined.

5.4. Calibration of the One-tenth Millimeter Intervals

The $\frac{1}{10}$ -mm subintervals at each end of the meter bars were also calibrated by using the same double- or cross-calibration method used in the calibration of the centimeter and millimeter subintervals. The

TABLE 13. Elements of calibration of the one-tenth millimeter intervals of meter 4 (NBS 1953 series of calibrations)

Elements of calibration=corrections to intervals, assuming that the subdivided millimeter intervals have their nominal lengths. Relative length of interval=nominal length+element of calibration.

Interval	Elements of calibration		Mean	Deviation from mean
	First	Second		
<i>mm</i>	μ	μ	μ	μ
-1.0 to				
-0.9	-1.29	-1.34	-1.32	0.03
- .8	-1.04	-1.07	-1.06	.02
- .7	-1.18	-1.26	-1.22	.04
- .6	-0.83	-1.03	-0.93	.10
- .5	- .43	-0.56	- .50	.07
- .4	- .33	- .40	- .36	.04
- .3	- .07	- .14	- .10	.04
- .2	- .07	- .06	- .06	.01
- .1	- .24	- .14	- .19	.05
.0	.00	.00	.00	.00
1000.0 to				
1000.1	-0.40	-0.45	-0.42	0.03
1000.2	- .04	- .04	- .04	.00
1000.3	- .27	- .41	- .34	.07
1000.4	- .71	- .66	- .68	.03
1000.5	- .60	- .58	- .59	.01
1000.6	- .45	- .42	- .44	.02
1000.7	- .37	- .37	- .37	.00
1000.8	- .30	- .37	- .34	.04
1000.9	- .29	- .30	- .30	.01
1001.0	.00	.00	.00	.00
Probable error	± 0.04	± 0.04	-----	-----

values determined for the elements of calibration for meter 4 are shown in table 13.

The probable error for the elements of calibration of any $\frac{1}{10}$ -mm subinterval is $\pm 0.04 \mu$. The deviations from the mean of two sets of determinations range from 0.00 to 0.10μ , the average deviation being 0.03μ .

6. Conclusions

In making the measurements on the meter bars, the results of which are reported in this paper, every reasonable precaution was taken so that the results would reflect the accuracy and precision that is obtainable with the present NBS equipment.

In the comparison of the bars to determine their total corrections and the corrections to the subintervals, the smallness of the residuals indicates that the standards were in good thermal equilibrium while the observations were being made. Although the observations were made when the temperatures of the bars were very close to 20°C , the small residuals also indicate that the values used for their respective coefficients of linear expansion are very nearly the true values. In calibrating these meter bars, the effect of any error of 0.01 deg C in temperature varies from approximately $0.01 \mu/\text{m}$ for the invar bars to $0.10 \mu/\text{m}$ for the stainless steel (*Fe-Cr*) bar.

To a large extent the effect of the imperfections of the lines and surfaces of the bars was compensated for by sufficient repetitions of the observations. It is believed that the final correction to the total length of each of the standards calibrated is not in error by more than 0.2μ , and in most cases is not in error by more than 0.1μ .

There is no evidence that the lengths of the platinum-iridium bars have changed a significant amount

during the past 20 years, whereas there is definite evidence that the other bars have changed. These changes, however, have become smaller during the ensuing years.

Regarding the possibility of making fewer measurements when intercomparing a group of standards, the 1952 and 1953 series of calibrations showed remarkably good agreement with the final corrections to the bars if a lesser number of direct differences had been considered. This could only be the case if the observed differences in length between the standards are all in excellent agreement. In the calibration of basic standards, a full calibration should be made where all differences between lengths of the bars are directly observed. However, where standards of a lower degree of accuracy are calibrated, a lesser number of direct comparisons would suffice.

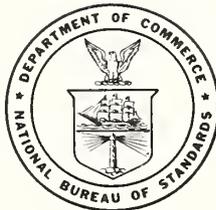
As it is known that bars of many materials are unstable in length, the 1952 and 1953 series of comparisons continues the program of maintaining all line standards of length and of obtaining additional knowledge about them, so as to enable the NBS to meet the needs and demands of the present and future for increased accuracy.

The author expresses his appreciation to Lewis V. Judson, Chief, NBS Length Section, for his untiring assistance and guidance and for the many helpful suggestions through the years during which the data reported in this paper were obtained, and to John S. Beers for his assistance with recent observations and computations.

WASHINGTON, July 30, 1954.

Calibration of Line Standards of Length and Measuring Tapes at The National Bureau of Standards

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Calibration of Line Standards of Length and Measuring Tapes at the National Bureau of Standards

Lewis V. Judson

The methods used at the National Bureau of Standards in calibrating line standards of length and measuring tapes submitted for standardization are outlined. The equipment used is described briefly. There is a discussion of some considerations that should be given as to whether or not a standard should be submitted to the Bureau. Instructions are given for submitting items to the Bureau for calibration. The appendix contains useful information on the use of steel tapes.

1. Introduction

The Bureau receives many requests from scientists, industry, and governmental agencies to calibrate line standards of length and measuring tapes. This Monograph is issued as a guide for anyone contemplating the submission of such items to the Bureau for standardization. It replaces Circular 332, Testing of line standards of length; Circular 328, Testing of measuring tapes at the National Bureau of Standards; and Circular 572, Calibration of line standards of length and measuring tapes at the National Bureau of Standards.

2. Line Standards of Length at NBS

The primary standard of length in the United States is the National Prototype Meter 27 (fig. 1), which is identical in form and material with the International Prototype Meter deposited at the International Bureau of Weights and Measures at Sèvres, near Paris, and also with the other national prototype meters distributed in 1889 in accordance with the treaty known as the Convention of the Meter, dated May 20, 1875. Except for expressing data in feet derived from and published as a result of geodetic surveys within the United States, for which the relation 1 foot=1200/3937 meter continues to be used, the yard in the United States is defined in terms of the meter by the relation

$$1 \text{ yard} = 0.9144 \text{ meter.}$$

At the Bureau there are secondary and laboratory length standards of various materials, sizes, and degrees of precision. Their lengths are known

in terms of the prototype meter. These secondary and laboratory standards are used in the calibration work done by the Bureau.

Proposals have been made to adopt, as the definition of the meter, a specified number of wavelengths of a suitable isotope of an element such as mercury (for example, the 5461-A wavelength of mercury 198). Interferometric calibrations of end standards of length have been made for many years. Such calibrations of line standards of length have been made in Germany, and the method is being followed with considerable interest at the Bureau.

3. Facilities for Calibrating Line Standards of Length

For the calibration of line standards of length the most precise measurements now being made at the Bureau are on its 1-m longitudinal comparator (fig. 2). This has been briefly described by Page.¹ It is especially suited for the calibration of the subintervals of a bar, using any one of the several methods that have been developed. Although this comparator is nominally a 1-m comparator, it is readily adaptable for standardizing 48-in. bars, and can be used for calibrating even longer bars.

For comparing two bars with an accuracy not better than 1 micron (0.001 mm or 0.00004 in.) a simple transverse comparator with no thermal insulation is commonly used. In this instrument the bars are mounted parallel to each other, and the carriage supporting the two bars moves back

¹ B. L. Page, Calibration of meter line standards of length at the National Bureau of Standards, *J. Research NBS* 54, 1 (1955) RP2559.



FIGURE 1. U. S. National Prototype Meter 27.

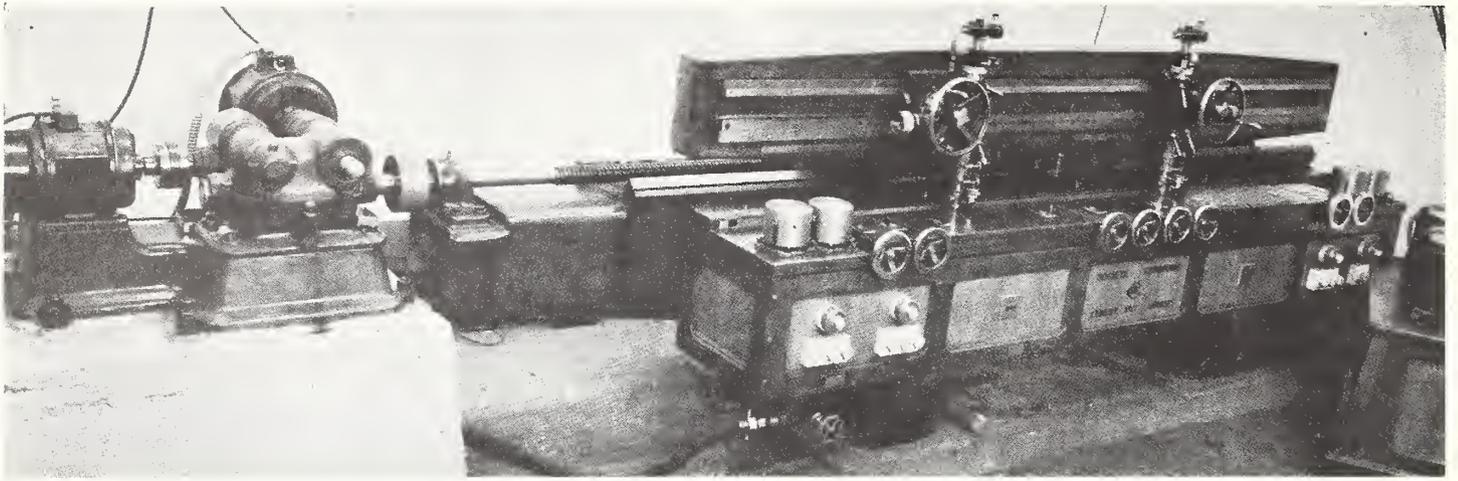


FIGURE 2. *Longitudinal comparator of the National Bureau of Standards.*

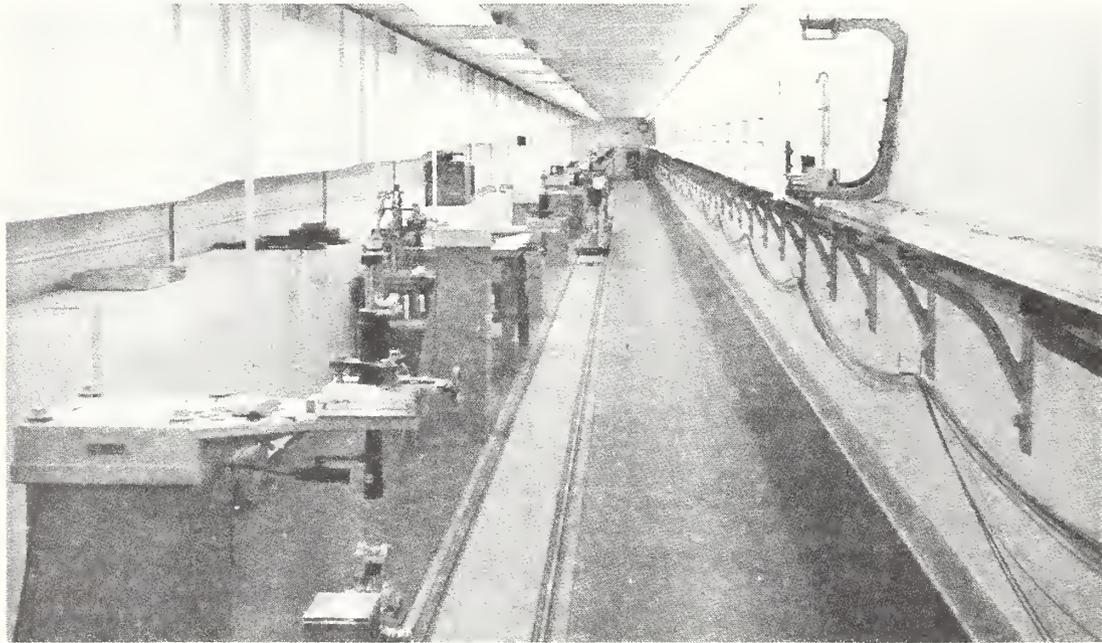


FIGURE 3. *NBS tape calibration laboratory.*

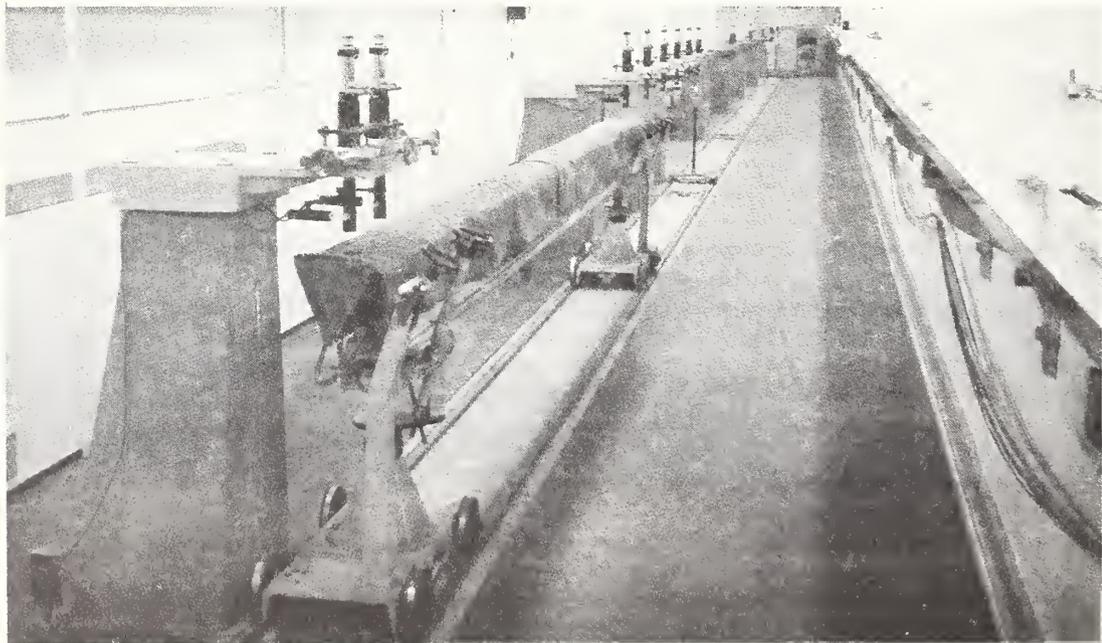


FIGURE 4. *NBS tape calibration laboratory, showing some details of the geodetic comparator.*

and forth in a direction perpendicular to a line parallel to the axis of each of the two bars. The micrometer microscopes are not moved during a comparison with this transverse comparator.

Short bars and small scales are often more conveniently calibrated by means of a linear dividing engine having two microscopes conveniently mounted so that they can be focused on the bars mounted on the longitudinally moving carriage. The instrument is then, in effect, a longitudinal comparator.

Micrometer microscopes having a pair of parallel "cross hairs" moving in the focus of a positive-type ocular are probably the most important parts of any instrument for comparing line standards of length. The movement of the cross wires is measured by a precision screw and a graduated drum. In the comparators in the length section at the Bureau the axes of the microscopes are vertical.

4. Facilities for Calibrating Measuring Tapes

At the Bureau there is a temperature-controlled laboratory about 212 ft long (fig. 3). Two distinct installations have been made in this laboratory. One is a 200-ft steel bench for calibrating steel tapes used in general surveying and engineering practice, as shown at the right in figure 3. The other is a geodetic comparator designed especially for calibrating the 50-m invar base-line tapes, such as are used by the U. S. Coast and Geodetic Survey. Its use is not limited, however, to a 50-m length. It is shown at the left in figure 3.

4.1. Steel-Tape Bench

The steel-tape bench has an over-all length of 200 ft 1 in., a width of 2½ in., and a thickness of ¾ in., and is constructed of stainless-steel bars approximately 12 ft long, with individually lapped and fitted ends. Stainless-steel conical dowel pins having threaded lower ends for locking nuts securely hold adjoining bars in the correct position. The supports for the bench are attached firmly to the wall, but can be adjusted whenever necessary. The bench is graduated at intervals ordinarily required for testing tapes graduated in the metric or in the United States customary system. The equipment is furnished with the necessary supports for the tape when it is to be supported at specified points, with apparatus for applying the tension, thermometers for observing the temperature, and the necessary clamps and other auxiliary equipment.

Comparisons of a tape with the bench standard are ordinarily made with a low-power microscope and a precision steel scale graduated either to 1/100 in. or to 1/2 mm. When unusually high accuracy is required and the character of the graduation lines warrants, comparisons with the bench can be made with a micrometer microscope.

4.2. Geodetic Tape Comparator

A 5-m bar, packed in melting ice when measurements are being taken, is the working standard used as the basis of measurements in the geodetic tape comparator. Piers bearing microscopes are spaced 5 m apart for a total length of 50 m. Auxiliary piers 1 m apart, also bearing microscopes, are placed between the 20-m and the 25-m piers. These provide intervals so that the 5-m bar can be standardized by means of a calibrated 1-m bar. By use of a special bracket at the 15-m pier, a double pier at the 30-m location, and an additional pier near the 45-m pier, it is possible to mount microscopes at the 50-, 100-, and 150-ft points. In figure 4 are shown the 15-m and 50-ft microscopes at the extreme left, then the covered trough and carriages for the 5-m bar, immediately beyond that the six piers with their microscopes spaced 1-m apart, and other piers beyond them.

In addition to the basic equipment of this comparator, there is the necessary auxiliary equipment, such as tape clamps, thermometers, etc.

Most of the work done with this comparator is with the 50-m interval, and the use of this interval will be assumed for the remainder of this section. After the 50-m interval is established by moving the 5-m bar to measure the distance between each of the 10 possible 5-m intervals between microscopes, and the positions of the 0- and 50-m points are transferred from the focuses of the microscopes to centers of two hemispheres in piers at floor level, the 5-m bar is moved outside the 50-m interval. An invar tape to be calibrated is then mounted in the comparator, supported on ball-bearing wheels, and proper tension is applied to the tape by means of a calibrated weight.

The difference between the interval on the tape and the established 50-m interval is obtained by the micrometer microscopes.

5. Calibration of Line Standards of Length

The measurement of line standards of length undertaken by the Bureau is classified under the several headings that follow. A complete calibration of this type of length standard includes the determination of the length at a known temperature and also of the expansivity usually expressed by the average coefficient of linear thermal expansion over a small range of temperature that embraces the temperatures at which the standard is likely to be used. For most work with any standard except one of the highest grade it is sufficient to assume a coefficient of expansion derived from a knowledge of the composition of the materials of which it is made. If the standard is subdivided, a calibration of the subdivisions may also be necessary. The number of intervals that it is advisable to compare will depend on the character of the standard and on the use to which it is to be put. In many cases

it is unnecessary to determine the correction for the subdivisions; a proportionate part of the errors for the total length may be assumed. Where it is necessary to measure a greater or less distance than the full length of the standard it is only necessary, in many cases, to test a few subdivisions; for example, any distance in even feet may be accurately measured with a 100-ft tape if the corrections are known for the entire length of the tape, for each 10-ft subdivision, and for each foot of the first 10 ft.

It should be noted that while platinum-iridium may be the best material for fundamental length standards, other materials are better suited to the needs of manufacture and industry; for example, steel rules and tapes are better for determining the sizes of steel and iron machine parts and structural members, because changes in length with changes in temperature are practically the same in the steel rule or tape as in the steel or iron part being measured.

Metric standards should be graduated to be correct at either 0° or 20° C, the latter temperature being preferred for everyday use because it eliminates to a great extent the question of differences in expansion. In this country, standards in the customary units of yards, feet, and inches are made to be correct at 68° F (20° C).

Requests for calibrations to be made by the Bureau should state the use to which the results are to be applied and also the accuracy desired, in order that the tests may be adequate for the end in view, avoiding unnecessary labor and expense. Where the highest attainable accuracy is needed, it is sometimes advisable to have the standard verified immediately before and again immediately after the important measurements in order to guard against possible changes due to undetected injury or structural alteration.

5.1. Reference Standards

This class includes standards of the highest type suitable for reference standards for makers of precision apparatus and for use in the most exact scientific investigations. One of the best constructions is that conforming to the specifications adopted for the prototype meters by the International Committee on Weights and Measures; but less expensive constructions are sufficient for all but exceptional purposes. The bars should be of the X-shaped or of the H-shaped cross section with all rulings in the plane of the neutral axis. The graduations should be accurately perpendicular to the longitudinal axis of the standard and lines must be fine, with sharp, smooth edges, and ruled on plane surfaces that have been given a faultless mirror or dull polish, depending upon the type of illumination used.

The lines on the prototype meters are about 0.006 mm wide. Since the time of the construction of these meters there has been a very marked

tendency toward narrower lines and the use of microscopes of higher magnification than those used in the latter part of the last century. But, whatever may be the width of the line, it is most important that the line be symmetrical, that is, that the bottom of the groove made by the tracing mechanism be symmetrical with the edges of the groove line. The portion of the graduations to be used should be defined by two parallel longitudinal lines about 0.2 mm apart. A material should be used that does not oxidize or otherwise tarnish on exposure to air or moisture. The graduations must not be covered by varnish or other protective covering.

A very useful alloy is one made of approximately 36 percent of nickel and 64 percent of iron, known as invar. Invar possesses a coefficient of expansion that is almost negligible at ordinary temperatures; in addition, it does not rust or tarnish readily on exposure to the atmosphere. It is important to remember that most materials, alloys in particular, undergo slight changes in the course of time, especially if subjected to considerable changes of temperature or to mechanical disturbances. Hence, whenever the highest accuracy is desired, standards should occasionally be verified. Standards of pure nickel and 42 percent of nickel have been found to be sufficiently stable for use as standards where verifications of the length of such bars can be made at suitable intervals of time.

The owner of a length standard of the reference class is usually justified in having a calibration made by the Bureau.

5.2. Working Standards

This class includes standards suitable for all ordinary precision work and suitable for the needs of college laboratories, manufacturers of the better grades of scientific apparatus, State superintendents of weights and measures, and most scientific work. Either an H-shaped cross section or a rectangular cross section with provision for supports at definite positions should be used. Short bars, such as decimeter bars, may be of rectangular cross section with the graduations on the upper surface and with the bars supported directly on a flat surface. The lines of the graduations should be sharp and less than 0.03 mm wide, and they should be ruled on a plane, well-polished surface that will not tarnish readily on exposure to the atmosphere. No varnish or other protective covering should be used. If the metal of the bar tarnishes readily, the lines should be ruled on plugs or strips of nontarnishing metal. Some such means as a pair of parallel longitudinal lines about 0.2 mm apart should be provided for defining the portion of the graduations to be used, and to facilitate the alinement of the bar. The graduation lines should be accurately perpendicular to the longitudinal axis of the standard. Standards of this class are compared with working standards of the Bureau and are certified to 0.001 mm if their quality justifies it.

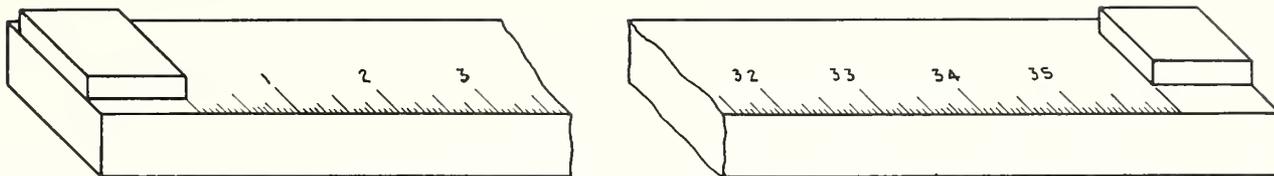


FIGURE 5. Line standard of length with contact pieces.

In the case of new working standards for State superintendents of weights and measures, it is recommended that these be graduated in millimeters for a distance of 1,010 mm or in $\frac{1}{32}$ in. for 37 in. In addition to the graduation, contact stops (fig. 5) are a convenience in some routine work, and a satisfactory arrangement is to have a contact extending across the standard at the zero line with a contact extending not over halfway across at the yard or meter point, this latter contact being so placed as not to interfere with or cover any part of the graduations. The meter standard should be accurate to 0.01 mm or better at a temperature² of 20° C. The yard standard should be accurate to 0.0005 in. at 68° F.

The owner of a working standard is sometimes justified in having a calibration made at the Bureau, but he may find that a calibration made by the maker or by himself is sufficient if a reference standard is available with which to make the comparison.

5.3. Commercial Standards

This class includes flat metal bars graduated along the center of the surface or along one or more edges, with lines less than 0.06 mm (0.0024 in.) wide. They should be of sufficiently good grade to meet the requirements for drafting, machine work, etc., and to meet the needs of local sealers of weights and measures. It is recommended that those for the use of sealers be graduated in millimeters for a distance of 1,010 mm or in $\frac{1}{32}$ in. for 37 in.³ New meter standards should be accurate to 0.1 mm or better at a temperature of 20° C, and new yard standards should be accurate to 0.005 in. at 68° F.

Commercial standards seldom require calibration by the Bureau. Local sealers of weights and measures will usually have their standards calibrated by State officials, and most other users will be justified in relying upon the reputation of the manufacturers, especially if purchase specifications have prescribed the accuracy of the standards.

6. Calibration of Measuring Tapes

Steel and invar tapes used in surveying, engineering, manufacturing, and construction work are calibrated at the Bureau. Tapes intended for geodetic base-line work require a very accurate

² Some bars have been made which were accurate at 0° C, and these will also be considered as meeting the requirements.

³ Contact stops as described for working standards may also be used on commercial standards for use by local sealers of weights and measures.

calibration; steel tapes used for more routine work can be calibrated to sufficient accuracy, using simpler apparatus and procedures.

For the most accurate work, such as primary triangulation or extensive surveys, tapes of the alloy of nickel and steel, known as invar, when properly made, have been found to be the most satisfactory because of their low coefficient of expansion and the slowness with which they tarnish from exposure to the atmosphere. However, they require very careful handling in the field to prevent twists and kinks. Standardization before and after field measurements of first-order bases for triangulation is usually desirable. The width of the graduations should be uniform and not more than 0.04 mm. The graduations should extend to one edge of the tape but not to the other, and all measurements should be made at the graduated edge.

Steel tapes have also been used for precise geodetic work, but require many precautions in their use to avoid errors caused by uncertainties as to the temperature of the tape. The coefficient of thermal expansion of both invar and steel tapes should be determined when they are used for work of the highest precision.

Ordinary 100-ft steel tapes to be used for engineering or construction work where an accuracy not better than 0.001 ft is required may consist of a steel ribbon with the graduations directly on the tape, or the intervals may be indicated by grooves or notches cut in sleeves securely fastened to the tape. Tapes having the intervals marked by rivets are not suitable for accurate work. Tapes having the zero mark at the edge of a ring and those having the terminal mark on a tension handle are not recommended for accurate work.

Tapes are compared with the steel-tape bench at the Bureau when supported throughout their entire length or when supported at specified points. These comparisons are accurate to the nearest 0.001 ft or 0.2 mm. When an accuracy greater than this is necessary, the Bureau is prepared to compare tapes of suitable length on its geodetic-tape comparator.

The standardization of a tape includes the comparison of the total length and of specially designated intervals. For most work it is sufficient to determine the total length only, and unless specifically requested to verify intermediate intervals, the Bureau calibrates the total length only. If, however, it is desired to have a tape for measuring accurately any integral number of feet, it is usually sufficient to have measurements

at every 10-ft point and at each foot of the first or of the last 10 ft.

As a basis for issuing a certificate and for placing its seal on a steel tape the Bureau has adopted the following specification and procedure.

6.1. Specification for Standard Steel Tapes

A steel tape is considered as standard when it has been calibrated by the Bureau and found to conform to the following specification: It shall be made of a single piece of metal ribbon, and none of the graduations shall be on pieces of solder or on sleeves attached to the tape or wire loops, spring balances, tension handles, or other attachments liable to be detached or changed in shape. The error in the total length of the tape, when supported horizontally throughout its length at the standard temperature of 68° F (20° C) and at standard tension, shall not be more than 0.1 in. per 100 ft (2 mm per 25 m). The standard tension is 10 lb (4.5 kg) for tapes 25 to 100 ft or from 10 to 30 m in length and 20 lb (9 kg) for tapes longer than 100 ft or 30 m.

6.2. Certification of Tapes

Tapes conforming to the specification will be certified by the Bureau and a precision seal showing year of standardization will be placed on each tape so certified. For tapes not conforming to the specification, a report will be issued, but the tapes will not be sealed. The Bureau's serial number on a tape simply signifies that it has been tested by the Bureau and either a certificate or a report issued.

Ordinarily the length of a steel tape is certified or reported by the Bureau to the nearest 0.001 ft or 0.0002 m (0.2 mm).

The specification given in section 6.1 is intended to cover steel tapes of highest precision and should not be taken to imply that only tapes conforming to it are suitable for use. For instance, tapes having a terminal graduation at the end of a ring are very desirable for certain purposes, where convenience is of more importance than high accuracy, and the Bureau will make the necessary observations and report on such tapes if satisfactory in other respects.

6.3. Standard Procedures in Steel-Tape Calibrations

Unless otherwise stated, the comparisons of the tape with the bench standard are made at the center of the lines on the edge to which the shortest graduations are ruled. If all the graduations extend entirely across the tape, the ends farthest from the observer when the zero of the tape is at his left hand are used. On tapes that have been cut off at the zero mark, the extreme end of the steel ribbon is taken as the zero point and not the center of any line that may be at that point. On tapes that have the zero point on a loop attached

to the steel ribbon at the end, the zero is taken at the outside of this loop, unless noted to the contrary.

Measurements for the calibration of a tape at the Bureau are made only at points at which the tape is supported.

The standard tension given in the specification is for a tape when supported on a horizontal flat surface. No standard tension has been officially adopted for a tape when supported in any other manner. The Bureau is sometimes requested to calibrate a tape supported throughout and supported at ends only under "standard tension." In such cases the Bureau uses the same tension for the tape supported at the ends as is used as standard tension when supported throughout. Many surveyors and engineers use a tension somewhat greater than this standard tension when they are using a tape supported at the ends only. The correction for sag for a given tension increases directly as the square of the increase of the weight per unit length of the tape. In the case of very heavy tapes the standard tensions of 10 and 20 lb (4.5 and 9 kg) may be found to be inadequate for tapes supported at the ends only. It is suggested that, dependent on the length of the tape and its weight per unit length, tensions in the range of 20 to 40 lb (9 to 18 kg) be used for tapes used in single catenary type of suspension. Tapes weighing 0.018 lb/ft or more may be considered as heavy tapes. Tapes weighing 0.009 to 0.011 lb/ft may be considered as light tapes.

In the standardization of invar tapes the following tensions are used, unless otherwise specified: 20 lb for 50-, 100-, and 150-ft tapes, and 15 kg for 30- and 50-m tapes.

When the lengths of intervals are desired on a tape supported at points that are more than 200 ft or 50 m apart, these lengths must be computed from quantities directly observed, using the mathematical expression for the sag as given in the appendix.

Since the present tape testing laboratory has been in use the observations have usually been made at a temperature very close to 68° F (20° C). Occasionally this may not be possible. The coefficient of expansion of the bench standard has been determined, and the difference between this value and the coefficient of expansion of steel tapes is taken into account whenever necessary. A value for the coefficient of expansion of 0.00000645 per deg F (0.0000116 per deg C) is assumed for steel tapes. This coefficient may be considered as correct for all except the most precise geodetic tapes. Determinations of the coefficients of expansion of both ordinary steel tapes and stainless-steel tapes give the value stated above within the limits of all ordinary measurements ever made with these tapes. The Bureau determines the coefficient of expansion of steel tapes only when sufficient need for such a determination is clearly indicated and when the value of the coefficient usually assumed for steel tapes may lead to difficulty.

Tapes made by the manufacturers that supply the surveying and engineering trade with high-grade steel tapes are almost universally correct for total length at standard tension and temperature within 0.01 ft, and ordinarily within 0.006 ft. Hence, for most work not requiring an accuracy better than 0.01 ft, where a certified tape is not required by law or by the terms of a contract, it is not necessary to submit the tape to the Bureau for a calibration. As high-grade tapes are ordinarily uniformly graduated within a few thousandths of a foot, the calibration of subintervals of a tape is not required except when rather precise measurements are to be made with the tape, that is, measurements to a few thousandths of a foot. The owner of a tape can often check the uniformity of division of the tape by measuring, for example, a 25-ft distance by using the intervals from 0 to 25 ft, 25 to 50 ft, 50 to 75 ft, and 75 to 100 ft on the tape.

6.4. Corrections Applicable to Steel Tapes

When a tape has been calibrated under one set of conditions and it is desired to use it under another set of conditions, it is often possible to calculate with sufficient accuracy the difference in the length of the tape under the two conditions. Information on this subject useful to surveyors has been included in the appendix.

6.5. Base-Line Tapes

When an invar base-line tape is submitted the first time for calibration on the geodetic comparator, the normal procedure is to determine its coefficient of thermal expansion and its weight per unit length before undertaking the standardization of its length. At one time it was thought necessary to check the stability of invar tapes by "whipping" them rhythmically and determining the changes in length caused by each group of whippings. Tapes received in recent years have been found to be sufficiently stable, and the whipping test has been discontinued. For the determination of its coefficient of expansion the electrical-resistance method described by Peter Hidnert and Richard K. Kirby⁴ is used.

Calibrations are usually made on the geodetic comparator under conditions similar to those used in the field. The standard calibrations now made for the U. S. Coast and Geodetic Survey on their base-line tapes are those to determine the length at 25° C of the interval 0 to 50 m with the tape under a horizontal tension of 15 kg when supported at the 0-, 25-, and 50-m points, and then when supported at the 0-, 12.5-, 37.5-, and 50-m points, with the 12.5- and 37.5-m points 6 in. above the place of the 0- and 50-m supports. This second condition of supports is to duplicate conditions used in the field when surveying along railroad

rails. For these field conditions the tapes are supported at the 12.5- and 37.5-m points on rollers, with the tops 6 in. above the rails. The height of 6 in. was chosen because all invar tapes possessed by the U. S. Coast and Geodetic Survey would then be lifted off the rail at the 25-m point. All measurements are made in the laboratory and in the field with thermometers weighing 45 g each attached at points 1 m inside the terminal graduations. For first-order work the values for the lengths of the intervals are certified not to be in error by more than 1 part in 500,000. Although all measurements are made on a new tape with the same care and just as accurately as in subsequent calibrations, the Bureau has made it a practice not to certify any base-line tape to first-order accuracy until further calibrations indicate that it is a stable tape so that first-order accuracy has a meaning. The terms "first-order" and "second-order" are those adopted in geodetic field surveying. Tapes certified to second-order accuracy have a certified accuracy of 1 part in 250,000.

When base-line tapes are submitted to the Bureau rather frequently it is recommended that, in calibrations after the initial, and one or more subsequent standardizations, the work be abbreviated. Measurements sufficient to show that values of the length have not changed more than the limit of error claimed in the previous calibration are made more easily than those required for a new certification. A statement that the previous certificate may continue to be used is issued when the abbreviated test shows that the values in the previous certificates are still valid.

If thermometers having a weight other than 45 g are used with base-line tapes in the field, that fact should be stated when the tapes are submitted for calibration. The thermometers may be submitted so that they can be placed at the proper positions on the tapes during the calibration.

7. Certificates and Reports

If apparatus submitted for calibration fulfills the requirements for certification, a certificate is issued. Values given in certificates or reports are applicable at the time of the calibration; there is no guarantee of the continued applicability of these values.

A National Bureau of Standards serial identification number similar to that shown below

NBS No. 2147

is usually engraved, stamped, or etched on apparatus, tapes, and standards that have been calibrated by the Bureau and for which a certificate or report has been issued. An identification number is not ordinarily placed on any precise standard when there is any danger of possible injury or of minute changes of length being caused, nor in certain other cases where there is already adequate identification.

⁴ P. Hidnert and R. K. Kirby. A new method for determining linear thermal expansion of invar geodetic surveying tapes, *J. Research NBS* 50, 179 (1953) RP2407.

Tapes and other articles for which definite tolerances have been prescribed are marked with a seal by the Bureau if the article is found to conform to the specifications. If a certificate or report is issued for such an article, a serial identification number, referred to above, is also placed on the article in addition to the seal.

8. Cooperation with Officials and Others

The Bureau will be pleased to assist investigators, manufacturers, and others by furnishing any information at its disposal concerning methods of measurement. It is also the desire of the Bureau to cooperate with manufacturers, scientists, weights and measures officials, and others in bringing about more satisfactory conditions relating to standards, physical constants, measuring instruments, and methods involved in length measurements. It is always desirous of receiving samples or descriptions of new apparatus or improvements on any type of apparatus mentioned in this Circular. Persons interested are invited to visit the laboratories of the Bureau, examine the apparatus and methods, and confer with those in charge regarding any problems they may have.

9. General Instructions to Applicants for Calibrations

9.1. Apparatus Accepted for Calibration

The Bureau will accept for calibration only a standard or an apparatus that is well made and not likely to change excessively. An adequate degree of quality is insisted upon as a prerequisite to acceptance for calibration. Line standards of length should be made of suitable material, such as iron alloy, brass, bronze, platinum alloy, or glass, and should be of a construction and workmanship suitable for the intended accuracy. Poorly divided scales on metal, and scales graduated on wood, plastics, or paper will not be accepted. In general, a linear scale of a grade better than the best machinist's scales will be required for a calibration by the Bureau.

9.2. Application for Calibration

All articles submitted for calibration should be accompanied by a written request. This request should enumerate the articles, giving the identification marks of each, and should state explicitly the nature of the calibration desired and an approximate valuation of the articles should be stated for use in insuring the return shipment.

9.3. Nature of Calibration

The application should state clearly the nature of the calibration desired, namely, the points at which measurements are to be made and the temperature, especially if other than standard, or any

other conditions. The most accurate results can be obtained and delays are avoided only when such full information is given. It is also desirable that the Bureau be informed as to the conditions under which the apparatus is used and the character of the work for which it is employed.

9.4. Special Calibrations

The Bureau will gladly cooperate with scientific investigators, manufacturers of apparatus, and others who need higher precision than is provided in the regular schedules by undertaking special calibrations as far as the regular work of the Bureau will permit. Kinds of calibrations not at present provided for may be undertaken if the work is important and the facilities and time are available. Approved calibrations not provided for in the regular schedules are considered special, and a special fee is charged for them. These should be arranged for by correspondence before shipment of the apparatus. The application should state fully the purpose for which the apparatus has been used or is to be used in the future, the need for the calibration, and the precision desired. The special fee charged depends chiefly upon the time consumed and the amount of alteration required in the Bureau's regular calibrating apparatus. It is ordinarily possible to give an estimate of the fee when so requested.

9.5. Condition of Apparatus

Before submitting apparatus for calibration, the applicant should ascertain that it fully satisfies the requirements for the calibration desired. It must be in good working condition. No repair work is done at the Bureau. If repairs are needed, either they should be made by the applicant, or the item should be sent to the maker before it is submitted for calibration. When defects, found after observations have been begun, exclude any apparatus from receiving the usual certificate, a report is rendered giving such information as has been found. In such cases a fee is charged, depending upon the time consumed. All possible care is taken in handling apparatus, but the risk of injury or breakage in shipment or at the Bureau must be borne by the applicant.

9.6. Identification of Apparatus

All packages should be plainly marked with the shipper's name and address and should contain a list of the contents. Each separate piece of apparatus or sample of material should be provided with an identification mark or number. The identification mark should be given in the application.

9.7. Shipping Instructions

Apparatus should be securely packed in cases or packages that will not be broken in transportation and that may be used in returning them to the

owner. The shipment in both directions is at the applicant's risk. Great care should be taken in packing. Clean, dry excelsior is a suitable packing material in most cases. Metal parts should be well protected from corrosion by oil or grease. Each instrument should also be wrapped in strong, waxed paper or other suitable covering to exclude dust and excelsior. The tops of boxes should be put on with screws, as any shock due to nailing and the subsequent opening is likely to cause damage. The tops of the shipping boxes should have the return or forwarding address on the underside. Transportation charges are payable by the party making the request. The charges for shipment to the Bureau must be prepaid, and, unless otherwise arranged, articles are usually returned or forwarded by express "collect." Information regarding the disposition of apparatus after completion of the measurements, including a statement as to the address to which shipment should be made and as to choice of carrier, should be supplied. In case such information is not given, it is understood that the calibrated apparatus is to be returned to the shipper.

9.8. Schedules of Test Fees

Schedules of test fees for the calibration of line standards of length and for steel tapes and invar base-line tapes have been established. Copies of these schedules may be obtained on application.

9.9. Address

Apparatus submitted for calibrations described in this Circular, as well as correspondence relating thereto, should be addressed: National Bureau of Standards, Washington 25, D. C., Attention: Division 2.4.

The author expresses his appreciation to B. L. Page, R. F. Ackermann, and J. S. Beers for their suggestions during the final revision of the manuscript.

10. Appendix—Use of Steel Tapes

In the use of steel tapes for precision work, attention should be paid to the temperature of the tape and to the tension applied to the tape. Corrections are sometimes necessary to take account of variations between the conditions of field measurement from those of the laboratory calibration as shown in the certificate or report. The accuracy of the balance or tension handle employed for applying tension should be checked by comparison with a calibrated balance or by the use of calibrated weights used with a pulley wheel so that the balance is being calibrated in a horizontal position.

Young's Modulus of Elasticity. A value for Young's modulus of elasticity is sometimes required in computing the length of a tape as used in the field under conditions differing from those used in the calibration laboratory.

In a series of measurements made several years ago at the National Bureau of Standards on a group of 45 steel

tapes, the value for Young's modulus of elasticity was determined to be 28.0×10^6 (that is 28,000,000) lb/in².

Variations in the material in the different tapes cause an uncertainty of 0.2 or 0.3×10^6 (200,000 or 300,000) lb/in².

A tape having the customary black finish has a smaller modulus of elasticity than has the original steel ribbon from which it is made, because there is included in the cross-sectional area of the finished tape not only the steel but also the protective coating which has a lower modulus. A value of 28.0×10^6 lb/in². may for all practical purposes be assumed for any steel tape, because the maximum error introduced by this assumption in using a 200-ft tape with a change in tension of 15 lb would be about 0.001 ft.

*Correction for Sag.*⁵ The difference in the length of a tape when supported throughout and when supported at equidistant points at the same height, the tension remaining constant, is equal to

$$\frac{L}{24} \left(\frac{wd}{P} \right)^2,$$

where L is the length of the tape, w is the weight of the tape per unit length, d the distance between points of support, and P the tension on the tape. These must be in consistent units; usually L and d in feet, w in pounds per foot and P in pounds.

In a study of a group of 45 tapes, previously mentioned, it was found that, using tensions between 10 and 25 lb, the difference between the observed value and that obtained from the formula for the correction for sag did not exceed 0.001 ft when the interval between the supports was 25 or 50 ft. Furthermore, when supported at the 0 and 100-ft points at 10 lb tension, the difference between the observed value and the computed value for the correction for sag did not exceed 0.002 ft, provided the correction for sag was not larger than 0.060 ft. When the tension was increased to 15, 20, or 25 lb, under the same conditions of support, the difference between the observed values and the computed value for the correction for sag, with the exception of a few tapes, did not exceed 0.001 ft. Moreover, there may be an agreement between the observed value and the computed value to 0.001 ft for individual tapes of relatively heavy weight when under a tension less than 15 lb. The formula was shown to be applicable to 100-ft tapes of nonuniform density, provided sufficient tension was used, e. g., 15 lb or more.

As an example of the use of the formula expressing the correction for sag one may consider a tape weighing 0.0080 lb per linear foot that has a length of 200.004 ft when supported horizontally throughout at a tension of 20 lb and that is to be used supported at the 0- and 200-ft points at that tension. The length may be readily computed to be

$$200.004 - \frac{200}{24} \left(\frac{0.0080 \times 200}{20} \right)^2 = 200.004 - 0.053 = 199.951 \text{ ft.}$$

It will be noted that nominal values for L and d are used. If the tape is to be used at a tension of 25 lb, then the length of the tape supported throughout at 25 lb should be determined.

Tension of Accuracy. If the length of a tape is known when supported throughout its length at standard tension and standard temperature, this information, together with a knowledge of certain constants concerning the physical properties of the tapes will enable one to determine the tension (tension of accuracy) to apply to the tape when supported at equidistant points at any known temperature to obtain the nominal length. These constants should be accurately determined. The case in which the observed temperature is the same as the standard temperature is given in text books on surveying, and the tension to be used in this case is often termed the normal tension. It is found by equating the correction for tension to the cor-

⁵ Lewis V. Judson, Effect of concentrated loads on the length of measuring tapes, BS Sci. Pap. 21, 390 (1926) S534.

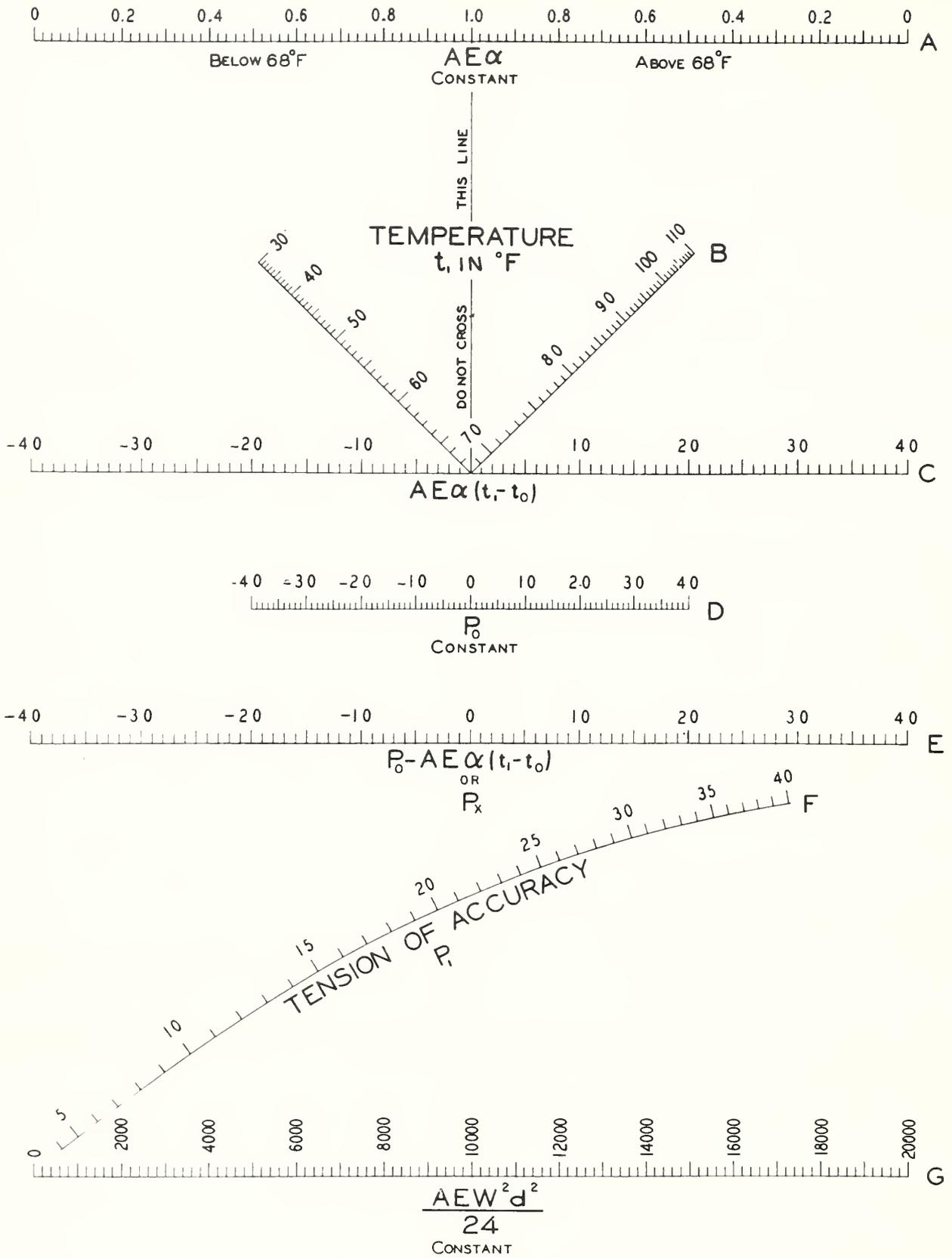


FIGURE 6. Nomogram for steel tapes.

rection for sag. The general case is an extension of this specified case. The equation is

$$P_1^3 - [P_0 - AE\alpha(t_1 - t_0)]P_1^2 - \frac{AEw^2d^2}{24} = 0,$$

where P_1 = Tension to be applied to the tape, when used at the observed temperature, t_1 , and supported at equidistant points at a distance, d , apart, in order that the tape will have its nominal length.

P_0 = Tension at which tape has its nominal length when supported throughout at standard temperature, t_0 .

A = Cross-sectional area of the tape.

E = Young's modulus of elasticity.

α = Coefficient of thermal expansion.

t_1 = Observed temperature.

t_0 = Standard temperature.

w = Weight of the tape per unit length.

d = Distance between the equidistant points of support.

As it is difficult to determine A directly with sufficient accuracy, it is usually advisable to determine the product AE , which is equal to $(P_2 - P_1)L/\Delta L$, where L is the nominal length of the tape supported throughout, and ΔL is the change in length when the tension is changed from P_1 to P_2 .

Nomogram for Tension of Accuracy. The solution of the cubic equation to determine the tension of accuracy of a tape supported in a catenary can be carried out rapidly by graphical means, using a nomographic chart, the equation being in Soreau's⁶ canonical form IV₀.

Figure 6 is such a chart, the value of α being 0.00000645 per deg F, and t_0 is 68° F. To use this chart to find the tension of accuracy, P_1 , for a tape, place a straightedge on the point representing the constant $AE\alpha$, axis A, and on the point representing the observed temperature t_1 , inclined scale B. The intersection of the straightedge with the dummy axis C is noted. The straightedge is then placed on this point and on the point representing the value P_0 , axis D. The point where the straightedge intersects axis E is determined. Place the straightedge

on this point and the point representing the constant $(AEw^2d^2)/24$, axis G. The point where the straightedge intersects curve F determines the tension to apply to the tape under the given method of support and at the observed temperature t_1 .

It should be noted that the constants $AE\alpha$, P_0 , and $(AEw^2d^2)/24$ are constants of the tape, independent of the conditions of its use and can be permanently marked on the chart for each tape, in the office, in preparation for use of the chart in the field.

It should be noted that for short lengths of tapes the value of the constant $K = (AEw^2d^2)/24$ will be small. The point of intersection of the straightedge with axis F will then often be somewhat indefinite on the chart, although the calculated value of the intersection is of course definite. This, however, is not serious as the length of a short interval of a tape changes very little for a 1 or 2 lb change in tension. For example, a 25-ft length of tape weighing 0.010 lb/ft will change in length only approximately 0.002 in. when the tension is changed from 10 to 12 lb when supported at the 0 and 25-ft intervals.

For 100-ft tapes, using the 100-ft interval, the change in length is very apparent for a 0.5-lb change in tension. In this case the chart and formula have been found to be in agreement with the observed values determined by actual test for various tapes tested at the Bureau. Although fewer tests have been made for tapes longer than 100 ft, it is believed that the chart and formula will apply equally as well.

Other nomograms may be drawn whose constants do not come within the scales on the nomogram shown in figure 6, or if conditions are such that a greater accuracy can be used than can be obtained on this chart, a new one can be drawn on suitable paper, with axes E and G further apart.

Attention is also called to the fact that the value P_0 determined for the total length or for one specified interval of a tape does not necessarily apply to all intervals on the tape. This may be due to one or more of the following conditions: A slight nonuniformity of graduation, a lack of homogeneity, or a lack of uniformity in cross section of the tape. For example, for one tape weighing 0.010 lb/ft, the value P_0 was found to vary from 0.5 to 1 lb for the intervals 0 to 25 ft, 0 to 50 ft, 0 to 75 ft, and 0 to 100 ft.

⁶ Soreau, *Nomographie*, pages 172, 174, 184, and 192 (Chiron, Paris, 1921).

WASHINGTON, D.C., February 16, 1960

The Measurement of Thickness

George Keinath

The survey on which this Circular is based was completed in 1955. More recent developments are not included. Since no other comparable compilation has appeared in the meantime, it is considered that the contents will be of value even though incomplete.



National Bureau of Standards Circular 585

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For sale by the Superintendent of Documents, U.S. Government Printing Office, Washington 25, D. C.

Price 50 cents

To Accompany
National Bureau of Standards Circular 585
THE MEASUREMENT OF THICKNESS

NOTE

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ERRATA

Page	Section	
13	1.152	The illustrations of figures 33 and 34 are transposed
17	1.203 (3d par. last line)	should read $d < D/4$
22	1.234	penultimate line should be deleted
41	4.41	transpose last five lines of left-hand column to top of page
43	4.53	caption omitted on last figure: Fig. 105, Circuit of sheet metal thickness comparator
52	5.4	McNicholas and Curtis reference should be numbered (143a)
52	5.4	Ref. (126) does not apply to this section
54	6.3	Ref. (66) does not apply to this section
	Reference	
72	(120)	should read Instruments in Industry, London (now called Automation in Industry)
72	(126)	second citation should read: Engrs. Digest (Amer. Ed.) 6, 156-7, (1945)
73	(161)	title should read: Elektrische Messung mechanischer Groessen
73	(165)	Citation should read: Revue Générale de l'Electricite (Paris), 44, 265-268(1938)

Foreword

Preparation of this monograph on thickness measurement was undertaken to bring together available information on the various methods and problems of measuring thickness that are frequently encountered in scientific and industrial fields. The usefulness of this survey to scientists and engineers is expected to be enhanced by the inclusion of discussion of ranges, accuracy, advantages, and limitations. No attempt has been made to describe methods and instruments in detail. Such detailed information should be sought in the literature references that are appended to the text. A discussion of the bounds that have been set on the scope of the work will be found in the introduction.

Names and descriptions of specific proprietary instruments are included for the convenience of the user, but completeness in this respect is recognized to be impossible. The omission of any method or device does not imply that it is considered unsuitable or unsatisfactory. Conversely, inclusion of descriptive material on any proprietary instrument, product, or process, does not constitute endorsement.

Some of the information included in this report was obtained from the open literature. The assistance obtained from American instrument manufacturers who furnished catalogs and other information descriptive of their products was vitally necessary to the work and is gratefully acknowledged.

The survey of the field of thickness measurement and the preparation of this Circular were carried on as a project of the NBS Office of Basic Instrumentation, which administers a Bureau-wide program of research, development, and dissemination of information relating to measurements and instruments. This program is cooperatively supported in part by the Office of Naval Research, the Office of Scientific Research of the Air Research and Development Command, and the Atomic Energy Commission.

A. V. ASTIN, *Director.*

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inch	10^{-8}	10^{-7}	10^{-6}	10^{-5}	10^{-4}	0.001	0.01	0.1	1	10^0	inch
mil	10^{-5}	10^{-4}	0.001	0.01	0.1	1	10	100	1000	10^4	mil
microinch	0.01	0.1	1	10	100	1000	10^4	10^5	10^6	10^7	microinch
cm	10^{-8}	10^{-7}	10^{-6}	10^{-5}	10^{-4}	0.001	0.01	0.1	1	10	cm
mm	10^{-7}	10^{-6}	10^{-5}	10^{-4}	0.001	0.01	0.1	1	10	100	mm
μ	10^{-4}	0.001	0.01	0.1	1	10	100	1000	10^4	10^5	micron
$m\mu$	0.1	1	10	100	1000	10^4	10^5	10^6	10^7	10^8	millimicron
Å	10	100	1000	10^4	10^5	10^6	10^7	10^8	10^9		Angstrom

coatings, films

sheets

plates

Conversion table.

Conversions among a number of units in which thickness is expressed.

The Measurement of Thickness

George Keinath¹

The numerous methods for the measurement of thickness in laboratory or shop are treated in seven groups according to physical operating principles: Mechanical—weight/dimension relationships, acoustics, vibration, displacement with various conversions; chemical—stripping, spectrochemical analysis; electrical—dielectric breakdown, resistance, electrochemical, capacitance, thermoelectricity; magnetic—attractive force, reluctance, saturation, inductance, eddy currents; optical—microscopy (also electron microscopy), interferometry, diffraction, shadow; X-ray—absorption, diffraction, backscatter, spectrometry; radioactive radiation—absorption, backscatter, tracers. Ranges, accuracies, advantages, and limitations are discussed. A bibliography of references, a limited list of suppliers, and a detailed index of the gages, methods, applications, and trade names covered are appended.

Introduction

The measurement of thickness is the measurement of the distance from a point on one bounding surface of a material body, through the body, to the opposite bounding surface. If the surfaces of a body are parallel, as the two sides of a sheet of metal or the inner and outer surfaces of a pipe, the thickness is obviously the distance between the bounding surfaces measured along a normal. However, if the surfaces are not parallel the direction in which the thickness is to be measured must be defined. Sometimes the measuring instrument itself determines the direction along which the thickness is measured.

Further complications in measuring thickness are introduced by the lack of definition of the bounding surfaces of a body. For example, the thickness of a textile material is rather indefinite. Here, also, the measuring instrument importantly influences the "measured" thickness.

Thickness measurements as above described deal with the measurement of a distance between two points. However, for some purposes the measurement of the average thickness of a body is of more significance.

This survey is intended to assemble for convenient reference as many of the methods of dealing with the problems of thickness measurement as possible. It is not limited to direct measurement of thickness, but gives sufficient background information on the types and principles of operation of the devices to reduce the need to seek the basic information elsewhere. The survey includes information on the measurement of physical parameters involved in the practical measurement of thickness, such as displacement, for example. It also deals with some more general aspects of the measurement, such as dynamic response. The extent to which these are treated is determined in general, by the principles and makeup of existing instruments.

Considering only industrial problems, the maximum thickness is about 10 million times

the minimum. The low limit is 0.01 microinch, as found in very thin coatings on glass. The high limit may be one or more inches, as in rolled or cast metals. This discussion will be primarily concerned with the measurement of the thickness of walls, sheets, and coatings of all kinds. Thickness of wires and threads is also included, although "diameter" is the term more generally used if the sample has a circular or an elliptical cross section. Distance measurement (as in spark gaps) is omitted, though the "feeler gages" for such measurements are called thickness gages.

The methods of thickness measurements may be destructive or nondestructive, contacting or noncontacting, with relation to the sample. It is very difficult to establish clear definitions of these terms for methods of measurement. For instance, the use of a needle to penetrate an insulating coat on a metallic base is a destructive method at the point of measurement. However, the manufacturer of the gage may consider his method nondestructive, because the tiny hole in a paint coating is practically invisible and unimportant in the use of the product. In the simplest problems the object of measurement is a small specimen accessible from both sides. Difficulties arise when the object is accessible from only one side. Such an object might be a moving sheet coming from a steel mill red hot, vibrating violently, covered with water or grease, and made almost invisible by a cloud of steam. In such a case the use of noncontacting thickness meters would be necessary.

Units of thickness: In this compilation, thickness has usually been expressed in "mils," i.e., thousandths of an inch, so that values of thickness will be easy to compare. It should be noted, however, that although this practice of stating thicknesses of flat metal sheets and plates and diameters of wires in mils is increasing, the specification of such sizes by numbers in one of several gage systems is still widely prevalent. In these systems, a given number is used to designate different thicknesses depending on the material under consideration, e.g., steel,

¹ Larchmont, N. Y.

brass, or aluminum. Since 1941 the American Standards Association has issued Standards for Preferred Thicknesses for uncoated thin flat metals under one-half inch, covering in the latest issue the range from 4 to 236 mils in 40 steps by values progressing in accordance with the 40-series of preferred numbers, each step being 3 percent higher than the preceding one. The 1941 issue has only 20 steps, with a difference of 6 percent from step to step.

Conversions among a number of units in which thickness is expressed are presented in the accompanying table on preceding page IV.

Methods of Measurement

1.00. Mechanical Methods

In this section are assembled methods in which the first measurement action is mechanical. The mechanical displacement, which is a function of the thickness, may in turn be measured by optical, pneumatic, electric, or other means, generally with the purpose of increasing the accuracy.

1.01. Weight [147; 190, p. 7].² If a material is homogeneous and of known density, a sample of controlled size can be weighed, and its thickness calculated from its weight. It is possible with sheet materials to cut samples of a specified size at each measurement and thus facilitate the determination of thickness. Wires are measured in standard lengths. Balances that indicate thickness directly are available or can be calibrated according to need.

1.02. Volume [168]. The thickness of non-porous insulation of wires may be determined in the following way: A length of wire of about 8 inches is cut off and put in a narrow cylindrical measuring vessel, filled with alcohol. The rise of the level gives the total volume. After stripping and dissolving the insulation, the measurement is repeated. From the volume difference the average thickness may be calculated. This method has been used successfully for several years by the Physikalisch-Technische Reichsanstalt in Berlin, for wires from 0.75- to 10-mm² section (AWG No. 18 to No. 7) with an accuracy of about 2 percent.

1.03. Oblique Cut.

1.031. Vitreous Enamel [111] (fig. 1). A method has been developed to measure the thickness of acid-resistant enamel coatings on iron plumbing fixtures (fig. 1). From a section of suitable size, along a new-cut edge, the enamel is ground off in a plane at an angle of approximately 5 degrees to the surface, exposing an oblique section of the enamel coating and part of the underlying metal. The lines (aa_3 , a_1d_1 , a_2d_2 , and a_3d_3) shown in figure 1 are marked with a ceramic underglaze pencil. The specimen

References to the literature and to patents are indicated by numbers, in brackets and parentheses, respectively, referring to the bibliographic listings on pages 69 and 75.

In writing this monograph the author has received a variety of assistance from the staff of the Office of Basic Instrumentation. This assistance is gratefully acknowledged. Deserving particular mention are the extensive editorial review and the contributions to the sections on application of interferometry, radiographic, and nuclear radiation methods.

is refired just enough to obtain a fire polish on the oblique section of the enamel. After cooling, the specimen is immersed in 10-percent citric acid and dried. Colored wax pencil is applied to the whole surface, which is then rubbed with a cloth. The colored wax is readily removed from the acid-resistant section, which will have retained its fire polish. This section is now clearly bounded by the line aa_3 , and the colored wax deposit. From measurements made along the parallel lines a_1d_1 of the thickness of the total coating and lengths along the cut, the thickness of the acid-resistant coating is computed. The method is accurate to about 0.5 mil in a total acid-resistant thickness of 2.5 to 8 mils.

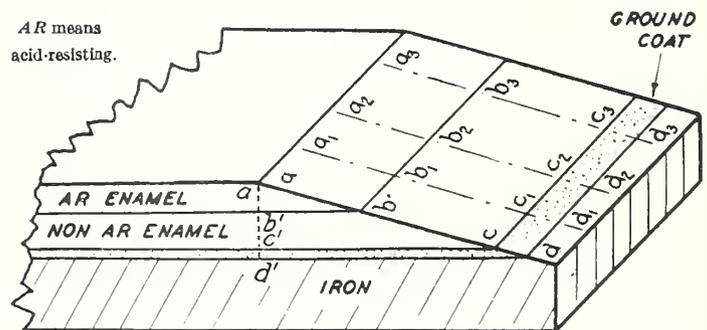


FIGURE 1. Sketch of specimen showing cross section and oblique section of enamel applied in three coats over iron.

1.032. Case Depth (Hardness Traverse) [10, 157, 172, 175]. Effective case depth in steel is defined as a distance measured perpendicularly from the surface of a hardened case to a point of hardness equivalent to Rockwell "C" 50. Total case depth is defined as a distance measured perpendicularly from the surface down to the point where carbon enrichment ceases. The specimen is cut, ground, and polished in either steps, taper, or perpendicular section. The exposed surface is tested for hardness until the effective case depth is reached.

1.04. Chord Cut [35] (fig. 2). Mesle's chord method for measuring the thickness of coatings depends on barely cutting through the coating—on a plane surface, with a grinding wheel of

² Figures in brackets indicate the literature references on page 69.

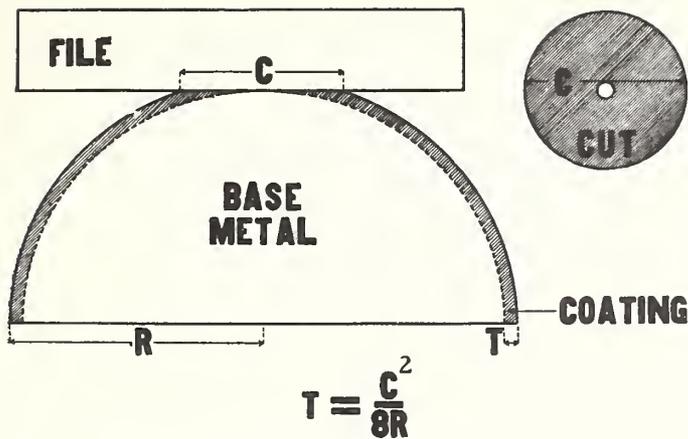


FIGURE 2. Chord method to determine thickness of coating on a spherical surface.

known radius fed perpendicularly; on a curved surface, by a chordal cut. In either case the thickness can be computed from the chord and the radius of the segment removed. The reference cited [35] gives the formula for calculating thickness for a number of different intersecting shapes: Plane surface and grinding wheel; convex spherical surface and flat file; convex spherical surface and grinding wheel; and concave spherical surface and small-radius grinding wheel. The curvature of spherical surfaces is determined with a spherometer. The measurement is difficult when coating and base have about the same color, in which case the cut surface is treated with chemicals that cause a color change.

With a grinding wheel radius of 3 to 4 inches the least thickness measurable is 0.05 mil, the chord length being about 40 mils. The accuracy is about 5 percent for nickel or composite coatings on steel plates, and about 10 percent for nickel coatings on copper or brass plates. For very thin coatings of chromium (about 0.02 mil) used for decorating purposes, a very small grinding wheel is recommended to produce a narrow but well-defined cut which can be measured with a microscope.

1.05. Manual Roller Gages.

1.051. *Euverard Gage for Wet Films* (figs. 3, 4). This gage, developed for the measurement of wet film thicknesses, is essentially a graduated eccentric wheel supported by two concentric wheels (fig. 4), all machined to an accuracy of 0.008 mil. When rolled over a plane surface, there is a clearance ranging from 0 to 4 mils (fig. 3) between the inner eccentric wheel and the surface upon which the outer two concentric wheels rest. When the gage is placed on a wet surface in the position of the greatest clearance, and rolled until the clearance is eliminated, the eccentric wheel will at some intermediate point contact and "pick up" the wet film. The measurement is usually performed in two perpendicular directions and the average of the readings taken as the true wet-film thickness.

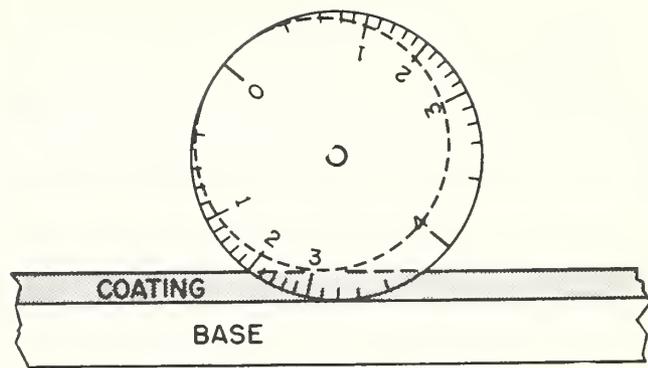


FIGURE 3. Roller thickness gage.

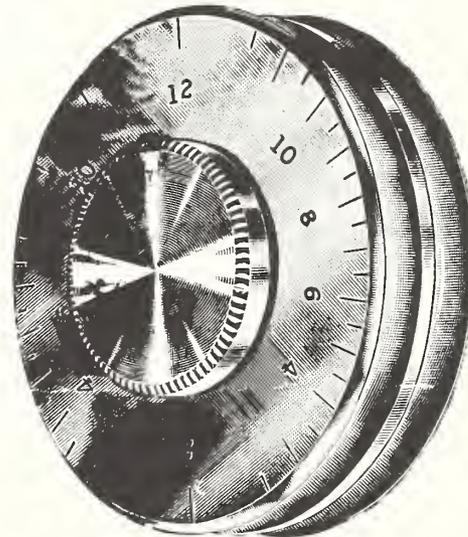


FIGURE 4. Roller thickness gage.

The standard instrument with a wheel 2 in. in diameter has a measuring range of 0 to 4 mils. Each scale division on the eccentric wheel represents 0.2 mil and readings to the nearest 0.1 mil are possible. The following instrument ranges are also available: 0 to 0.4 mil, 0 to 1 mil, 0 to 2 mils, 0 to 12 mils, 10 to 30 mils, and 20 to 60 mils; and the scale graduations, 0.02, 0.05, 0.1, 0.5, 1.0, and 2.0 mils.

Manufacturer: Henry A. Gardner Laboratory, Bethesda, Md.

1.052. *Euverard Gage for Dry Films* (figs. 5, 6). An adaptation of the wet-film gage described above makes it suitable for measuring the thickness of coatings, especially paint coatings, on a nonconducting base like wood or glass. The same wheel is used, but the film is removed from the base along two suitably spaced strips, to permit the outside concentric wheels of the gage to rest on the uncoated base. By the use of a suitably adjusted mirror or lens, the degree of clearance between the measuring surface of the gage and the film coating can be seen with the aid of a diffused-light source directed toward the gage. As long as there is a gap between the measuring surface of the eccentric wheel and the coating, it is possible to observe a brightly illuminated area which has the

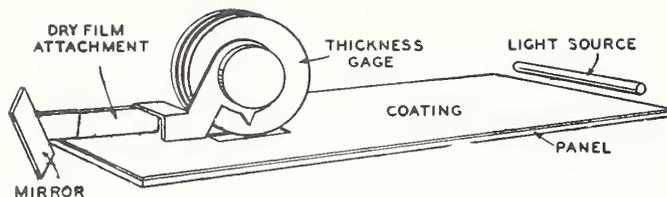


FIGURE 5. Roller gage to measure thickness of dry coatings.

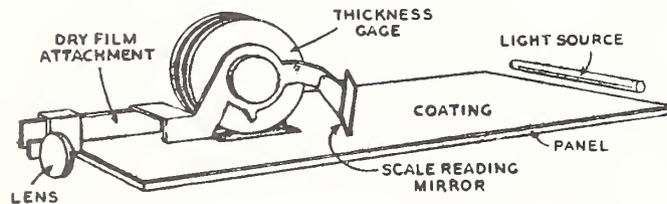


FIGURE 6. Roller gage to measure thickness of dry coatings.

width of the measuring surface and the height of the space between this surface and the coating. The gage is slowly turned until the illuminated area disappears, at which point the reading is taken, in two directions if desired. A magnifying lens is provided for better observation of the luminous area. The scale can be read from the mirror without moving the operator's eye from the viewing position (figs. 5, 6). It is claimed that this method, which applies no pressure to the film, gives the true thickness more accurately than other methods which require decoating.

Manufacturer: Henry A. Gardner Laboratory, Bethesda, Md.

1.06. Sphere Penetration Gage (figs. 7, 8, 9).

The Pfund paint-film thickness gage is used for the determination of the thickness of wet paint or varnish films (fig. 7). A convex lens, L, whose lower surface has a radius of curvature of 250 mm, is mounted in a short tube, T_1 , which slides freely in the outer tube, T_2 . The compression springs, S, keep the convex surface out of contact with the paint film until pressure is applied to the top of T_1 . The instrument is rested on the painted surface and the lens is forced down as far as it will go. Upon releasing the pressure and removing the gage, a circular spot is left on the lens as well as on the painted surface. The diameter of the spot on the lens is measured to 0.1 mm with a steel scale and then converted to thickness by using tables. The accuracy has been found to be within 0.04 mil for films 1.6 to 2.6 mils thick.

Manufacturer: Koehler Instrument Co., Jamaica 33, N. Y.

1.10. Mechanical Gages, Direct Reading [78; 190, p. 6-8]. Direct-reading mechanical gages, examples of which are discussed below, are the most universally used thickness meters, especially for making measurements on small pieces. Their use is prescribed in many of the ASTM specifications and standards. They give the thickness at the spot where the measurement is

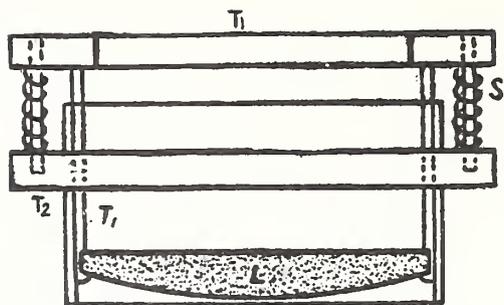


FIGURE 7. Pfund film-thickness gage.



FIGURE 8. Pfund film-thickness gage.

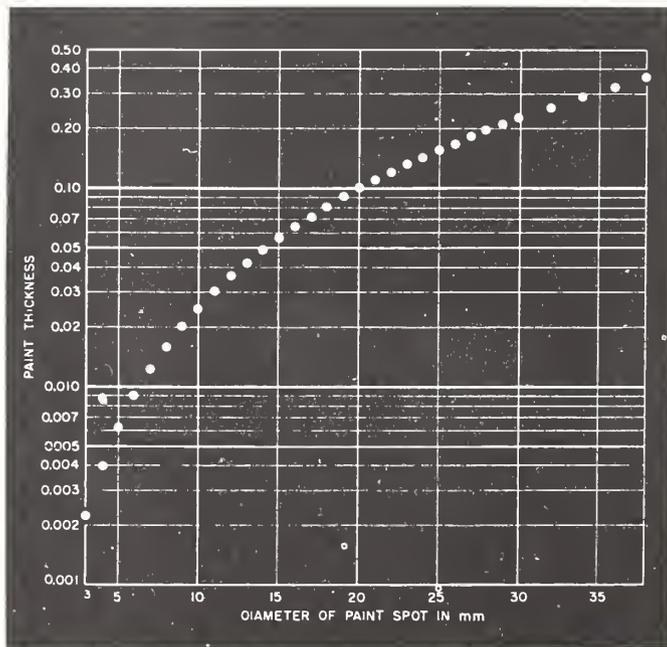


FIGURE 9. Paint thickness as a function of the paint-spot diameters for the Pfund gage.

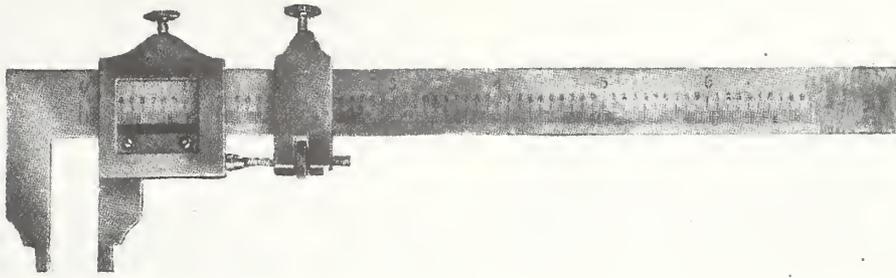


FIGURE 10. Slide caliper with vernier attachment.

made. For large objects one must determine the average thickness by making a number of measurements at a number of evenly distributed spots and calculating the arithmetic mean. The thickness of very fine paper, such as that used for capacitors, is often measured by stacking an assembly of 10 sheets and using a parallel-plate gage. With paper about 0.25 mil thick, using dial gages or screw micrometers, the thickness of one sheet might be measured as 0.34 mil, whereas the average thickness based on 10 stacked sheets might be only 0.24 mil. The difference in values between measurements on a single sheet and measurements on stacked sheets depends on the "packing fraction" (the extent to which the thicker areas of one sheet coincide with the thinner areas of another sheet). The stacked thickness values are preferred as a practical matter because they tend to be more nearly uniform.

1.11. Slide Calipers [27] (fig. 10). Slide calipers are among the simplest devices for thickness measurement. The reading accuracy is about 0.1 mm (4 mils) for direct reading, because the smallest discernible distance is about 0.07 mm for the average human eye [27]. With a vernier attachment the accuracy can be much increased, with 0.018 mm as the possible limit. In view of the thickness of the graduation lines a practically feasible value is 0.050 mm (2 mils). By using a magnifying lens, a vernier with 50 divisions, and a line thickness of not more than 0.07 to 0.1 mm, a least reading of 0.02 mm may be obtained.

1.12. Joint Calipers (fig. 11). There are three types of joint calipers (fig. 11) which are customarily used as comparators to transfer a dimension to a steel scale for measurement:

(a) Firm-joint calipers with legs that hold their position by friction, giving the advantage of very quick adjustment.

(b) Screw-adjusted firm-joint calipers, similar to (a), but with fine adjustment.

(c) Spring calipers, the most widely used type.

1.13. Screw Micrometers [15, 20, 27, 118, 206] (fig. 12).

1.131. Micrometer Caliper [27]. The micrometer is essentially a calibrated screw. By turning the thimble the screw revolves in a

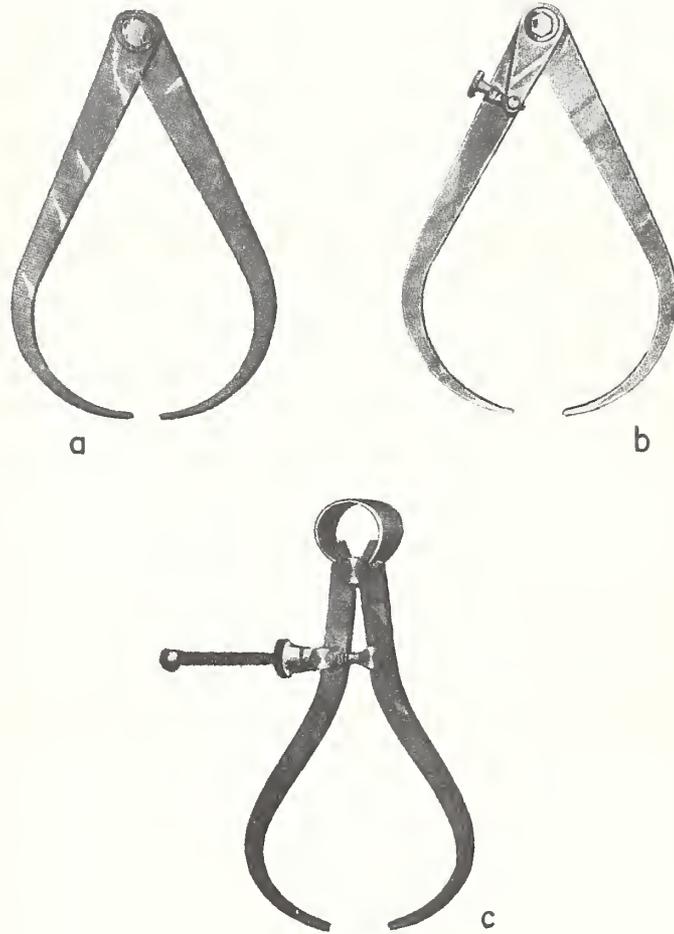


FIGURE 11.
a, Firm-joint caliper; b, firm-joint caliper with screw adjustment;
c, spring caliper.

fixed nut in the frame, and the spindle, which is the unthreaded portion of the screw, moves up or down relative to the anvil. The spindle moves through a definite distance depending on the pitch of the screw. As a rule, there are 40 threads to the inch; hence one turn of the thimble moves the spindle $\frac{1}{40}$ in. = 0.025 in. = 25 mils. The major divisions of 25 mils are marked on the hub and 25 subdivisions are marked on the beveled portion of the thimble attached to the screw.

Other micrometers of greater precision are also made. It should be noted, though, with the more finely divided screw and the more precise

instrument, greater care must be taken in its use. Accuracy of measurement depends largely on the pressure exerted on the work and on the compressibility of the material. The more compressible the material, the greater the error. Many types of screw micrometers use a friction clutch or ratchet for rotating the screw in order to obtain constant and correct pressure. Generally the accuracy obtained is ± 0.1 to ± 0.5 mils.

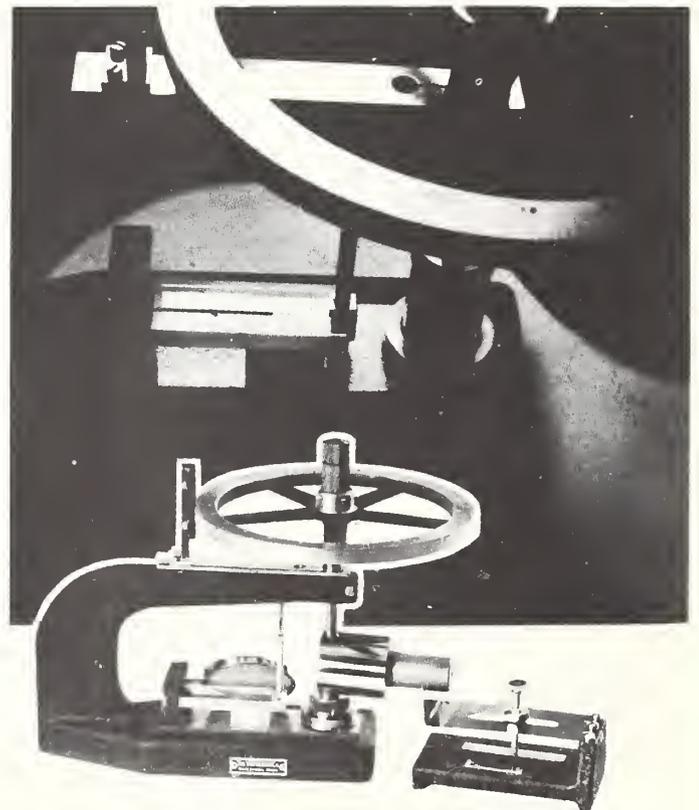
One design of micrometer caliper with a non-rotating sleeve reads by vernier down to 0.025 mil. Optical magnification with a simple 5 or 10 \times hand lens is often used with such instruments. In the most elaborate designs [27] the reading error is reduced to 1 μ (0.04 mil) and even 0.1 μ . The total accuracy of measurement is, of course, less than the reading accuracy.

Procedure and permissible errors of micrometer calipers with and without ratchets are discussed in ASTM standard D374.



FIGURE 12. *Micrometer caliper.*

1.132. *Screw Micrometer With Optical Indication of Pressure* [206] (figs. 13, 14). This micrometer (figs. 13, 14), which utilizes optical pressure indication, has been developed to provide the highest accuracy of screw micrometers [206]. The micrometer screw, machined with great precision, carries a 6-in.-diameter aluminum wheel with 0.1-mil graduations about 100 mils apart, a magnification of 1,000. This permits readings of 0.01 mil. Combined with this mechanical-screw micrometer is an optical indicator of pressure, which enables the operator to apply exactly the same pressure on the object for each measurement. This pressure indicator is a combination of an optical flat, a chromium-plated steel flat, and a red selenium-glass screen, so arranged that the slightest upward pressure on the micrometer spindle causes interference bands to move past a reference line. Each interference band is a measuring unit of 0.01 mil. The bands are spaced about $\frac{1}{8}$ in. apart. Any measuring load from 0 to 2.5 lb can be applied. When in proper adjustment, two or three bands will show, from the left-hand edge of the steel flat up to the cross-reference line, when the spindle is not touching the anvil. Each additional band that is moved up to this reference line corresponds to an increase of measuring load of 6 oz. The load is selected to suit the material under test. The ranges of this light-wave screw micrometer are 0 to 1, 0 to 2, or 0 to 3 in. The threshold sensitivity of measurement



FIGURES 13 and 14. *Pressure indication on light-wave screw micrometer.*

is 0.005 mil, and of load, $\frac{1}{2}$ oz. Readings can be duplicated to 0.01 mil.

A special design has a range of 0 to 3 in. and a number of features that insure still higher accuracy and convenience in use.

Manufacturer: The Van Keuren Co., Watertown 72, Mass.

1.133. Screw Micrometer With Electrical Indication of Contact [15, 118] (figs. 15, 16). Most mechanical gages for thickness measurement impose a load on the object. For compressible materials any load interferes with making precise measurements, unless controlled in some way, such as that described in 1.132. For metallic specimens, another solution of the problem is the use of a low-voltage signal lamp to indicate contact of the metal tip. This is not fully satisfactory, because the current over the contact points may cause pitting.

Carson Electronic Micrometer: The circuit of the Carson Electronic Micrometer [15, 118] is shown in figures 15 and 16. The circuit adopted uses a negligible current to light an indicator lamp by means of a relay at the moment of contact. A displacement of 0.005 mil is sufficient to trigger the relay. Electrical contact is established through the work piece itself, when measuring metallic materials. For nonconductors, electrical contact is made within the gage head when mechanical contact with the work closes the small gap between two elements in the gage head. It was found that the presence of appreciable current at the contact surfaces tended to build up carbonized layers which soon reduced accuracy.

The dial diameter is 3 in., with 250 divisions about 40 mils wide, each representing 0.1 mil, corresponding to a screw pitch of 25 mils. The magnification is about 400. The accuracy of the instrument is given as 0.02 mil. For the measurement of compressible materials the standard anvil pressure (supplied by a spring in the upper anvil) is $\frac{1}{8}$ oz (3.4 g), but with interchangeable springs it can be increased to 2, 4, or 8 oz.

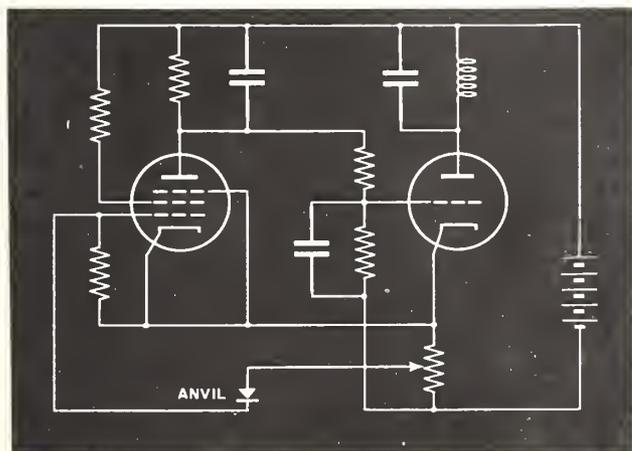


FIGURE 15. Simplified circuit of the Carson gage.

Manufacturer: J. W. Dice Co., Englewood, N. J.

ASTM standard D374 covers the measurement of the thickness of solid electrical insulation with machinist's micrometers and dead-weight dial micrometers; it allows tolerances considerably wider than those obtainable with the Carson micrometers.

Wall Thickness Meter for Tubes: A mechanical micrometer with electrical contact is used to test the wall thickness of hollow propellers and of tubes up to 6 ft long. The gage consists of two long arms mounted on a base. Contact points at the ends of the arms are connected to an electronic control box, giving both optical and acoustical signals when contact is established between the micrometer screw and the object under test. After a preliminary adjustment of the equipment, two readings on the vernier dial of the micrometer screw are taken, one with the tubing under test in place, the

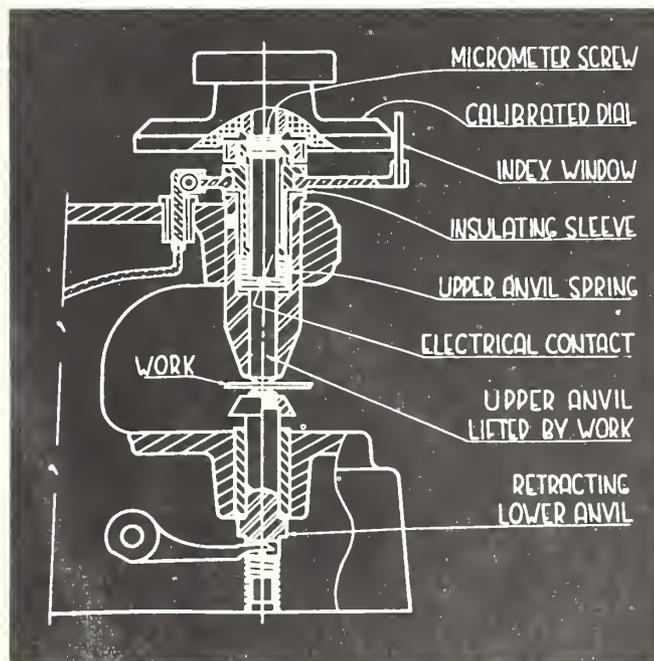
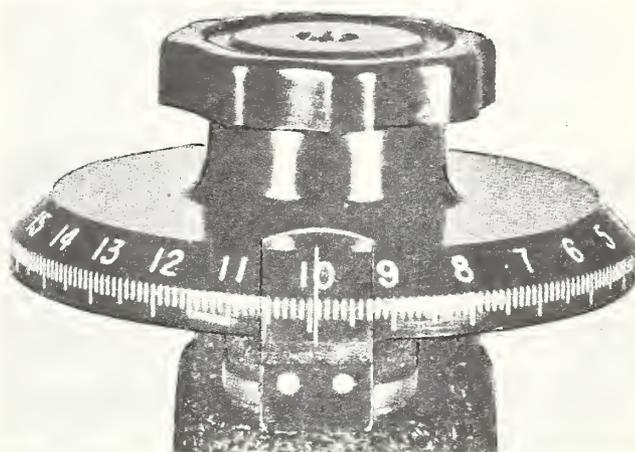


FIGURE 16. Carson gage to measure thickness.

other with it removed and the upper contact point touching the lower contact point. Differences in wall thickness of 1 mil can be determined in this way.

Manufacturer: Fairchild Aircraft Co., Hagerstown, Md.

1.134. *Penetrating Needle (figs. 17, 18)*. A penetrating-needle-type gage is used for the measurement of the coating on either flat or curved metallic surfaces. The body of the gage rests with three points on the surface to be tested. The dial screw, 4, moves the needle, 8,

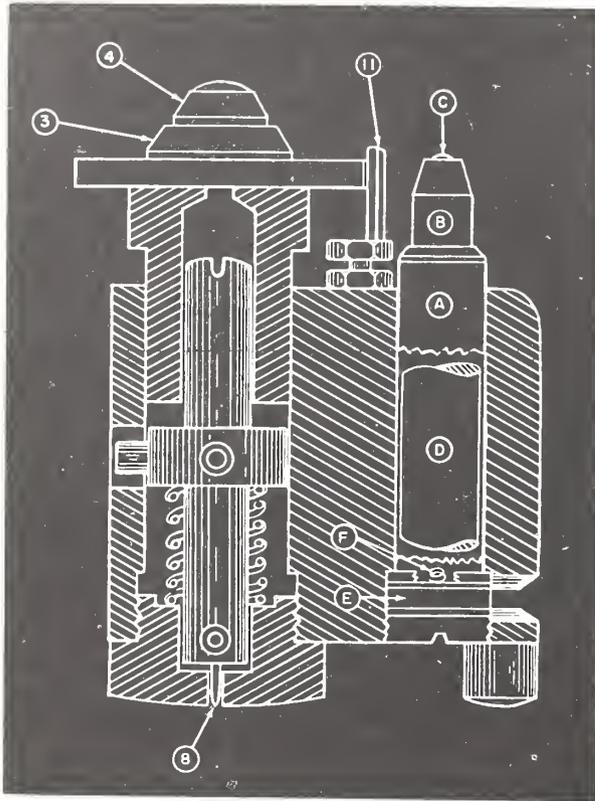


FIGURE 17. Cross section through measuring head of Gardner gage.

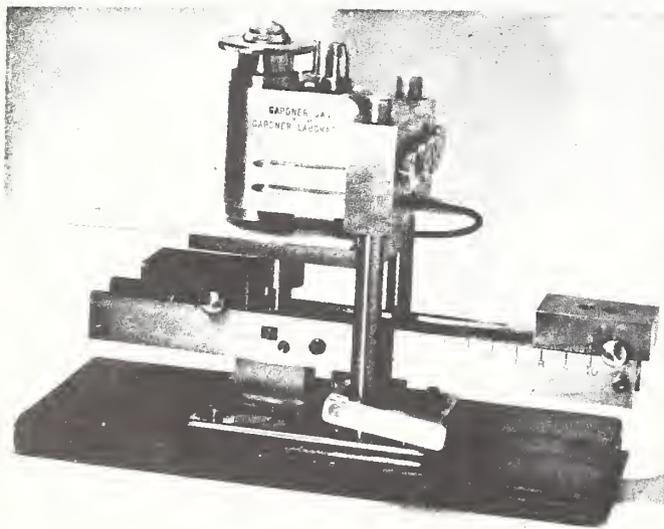


FIGURE 18. Gardner thickness gage with stand.

downward until it has penetrated the film of insulation, and makes contact with the metallic base. This lights signal lamp C. The dial makes one full revolution for 2 mils movement of the needle and the scale can be read with an accuracy of 0.01 mil. The instrument is generally used in a gage stand, which holds the gage vertical. A pressure of 0 to 10 lb is applied on the large foot that carries the needle; this pressure is adjusted according to the hardness of the surface film. For zeroing, the gage is placed on an uncoated plane metal surface.

Manufacturer: Gardner Laboratories, Bethesda, Md.

A gage, operating on the same principle, with an electronic relay circuit to light the lamp, is manufactured under the trade name "Bathytrol."

Manufacturer: Electronic Control Corp., Detroit 7, Mich.

1.14. Dial Micrometers [20].

1.141. *Simple Dial Micrometers (Medium Sensitivity) (figs. 19, 20)*. Even a simple dial micrometer provides much higher magnification than is possible with the standard screw micrometer, thus facilitating the reading of small changes of thickness. The movement of the anvil is magnified by mechanical means, the simplest using the lever principle. Mechanical gears are often used (fig. 19). Very sensitive gages of this type make one full revolution of the pointer for 4 mils displacement of the anvil, with graduations of 0.05 mil. For greater thickness, the pointer makes up to 10 revolutions, which are read on a smaller dial (fig. 20). In this device, the magnification of the anvil movement is about 2,500. Generally the magnification of dial micrometers is 500 to 1,000.

An instrument with a fan-shaped housing and $\pm 45^\circ$ deflection, "Micronar," has a range of

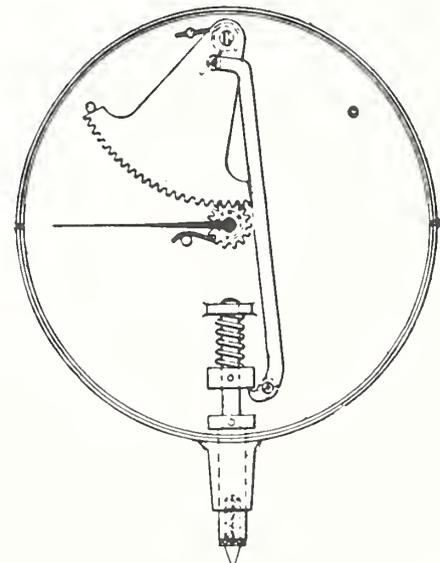


FIGURE 19. Principle of a dial micrometer.

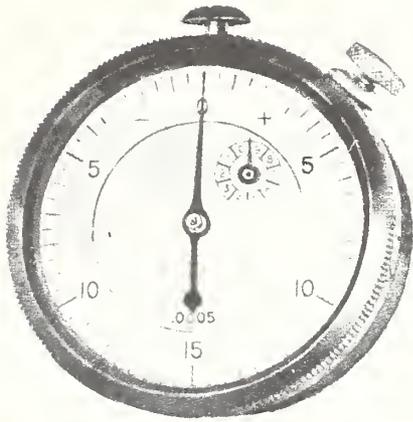


FIGURE 20. Dial micrometer with revolution counter and a range of ± 15 mils in divisions of 0.5 mils.

± 1.8 mils. Other divisions of 0.1 mil are spaced 150 mils apart, whereas divisions near the scale center are subdivided to 0.02 mil. The magnification is 1,500.

Manufacturer: Standard Gage Co., Poughkeepsie, N. Y.

1.142. *Dial Gage Micrometer With Screw Micrometer* (fig. 21). This instrument can be used either by reading the dimension on the barrel or by setting the barrel to the desired value and reading the deviation on the dial. The range of the dial indicator is ± 1 mil, with each division 0.1 mil.

Manufacturer: Federal Products Corp., Providence, R. I.

1.143. *Dial Gage Micrometer With Joint Caliper* (figs. 22, 23). This meter is used to determine the thickness of walls, sheets, and sections of castings and forgings. It is trigger-operated and the dial micrometer has a revolution counter. One mil is represented by 2 in. on the dial, corresponding to a magnification of 2,000. The maximum jaw opening is 4 in.

1.144. *Dead-Weight and Spring-Loaded Gages* [20, 116a] (figs. 24, 25, 26, 27). For exact measurements of objects that are not perfectly flat or that have a fibrous structure, the measured thickness depends very much on the applied pressure. For the measurement of paper a gage foot between 14.3 and 16.5 mm in diameter is used, so that local variations of thickness are eliminated, and the maximum thickness of the sheet is read. For special applications, e.g., to measure the thickness of capacitor paper 0.2 to 0.4 mil thick, mechanical gages with ball and anvil have been used by the Bell Telephone Laboratories. The balls used were 10 or 20 mils in diameter and the load 30 to 200 grams, giving, despite the light load, a relatively high pressure.

For highly compressible materials, special mechanical dead-weight gages have to be used. Examples are knitted, woven, and pile fabrics; blankets, felts, rug underlays; sheet rubber (foam or solid); paper, etc. The following defi-

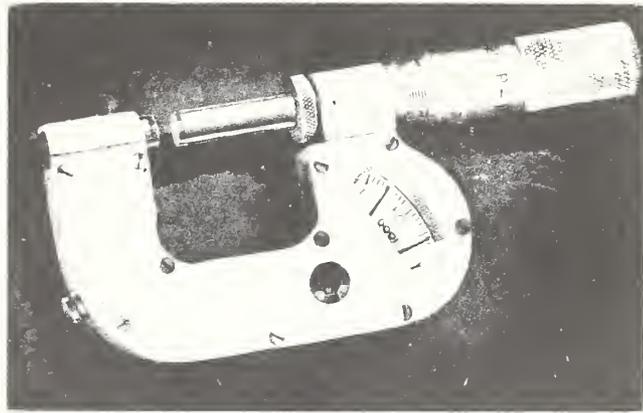


FIGURE 21. Combined screw micrometer and dial indicator.

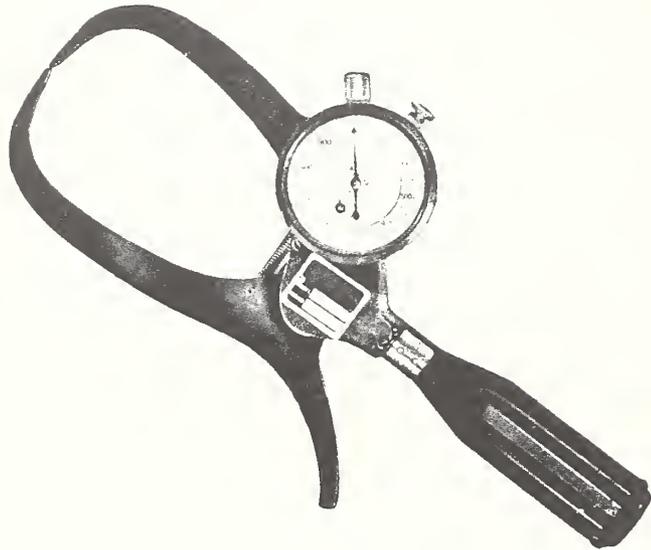


FIGURE 22. Combined joint caliper and dial gage.

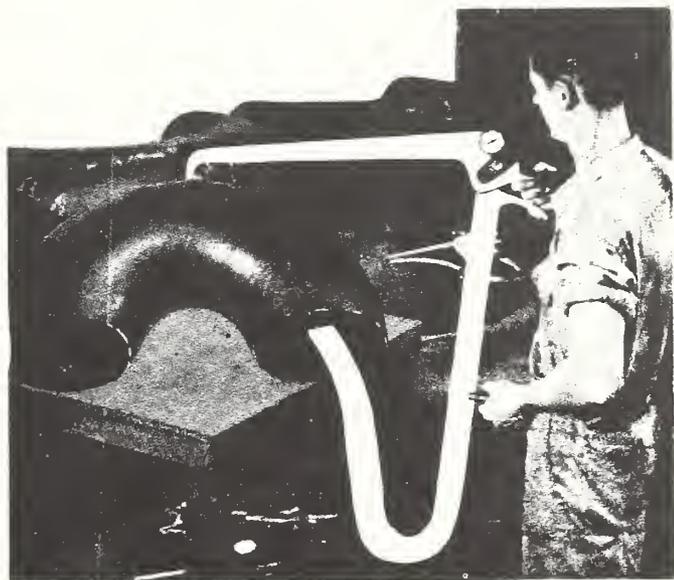


FIGURE 23. Combined joint caliper and dial gage to measure the wall thickness of elbow tubes.

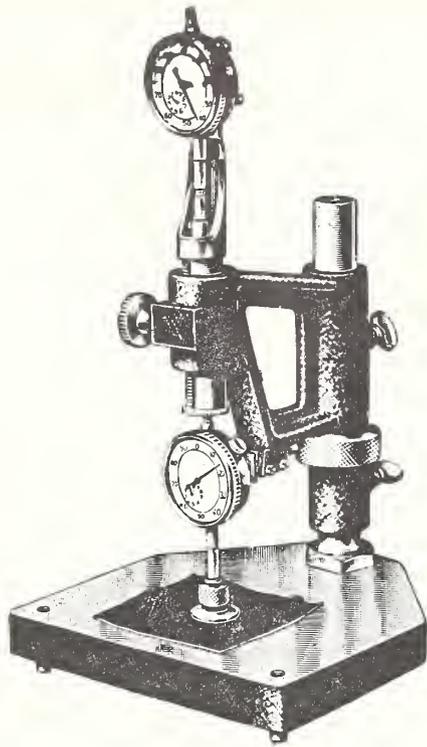


FIGURE 24. *Compressometer, thickness gage for compressible material.*

nitions for the thickness of compressible materials have been given: (1) The thickness of a specimen is the distance between the foot and the anvil when the pressure has reached a specified level. (2) The standard thickness is the thickness when the pressure has been increased to 1 lb/in.², all other conditions being standard.

ASTM Standard D1056 provides that in measuring the thickness of cellular rubber a dead weight of 25 grams on a circular area 1¼ in. in diameter be used.

Typical instruments are following:

(a) "*Compressometer*" [179] (fig. 24). In this instrument the specimen is placed on the anvil and the knob turned until the circular foot is resting on the fabric. The upper dial, by measuring the extension of the calibrated spring, indicates the amount of pressure being exerted on the material under test. The distance between the anvil and the foot, i.e., the thickness, is indicated on the lower dial. The compressibility of a material can also be determined with this instrument by taking a series of readings with alternately increasing and decreasing pressures.

Manufacturer: American Instrument Co., Silver Spring, Md.

(b) *Amthor Dead-Weight Thickness Gage* (fig. 25). The handle on the top is used to lift the weight before the material is put in place. The 6-in. scale of the instrument has 0.1-mil divisions, with a maximum range of 40 mils for one full revolution.

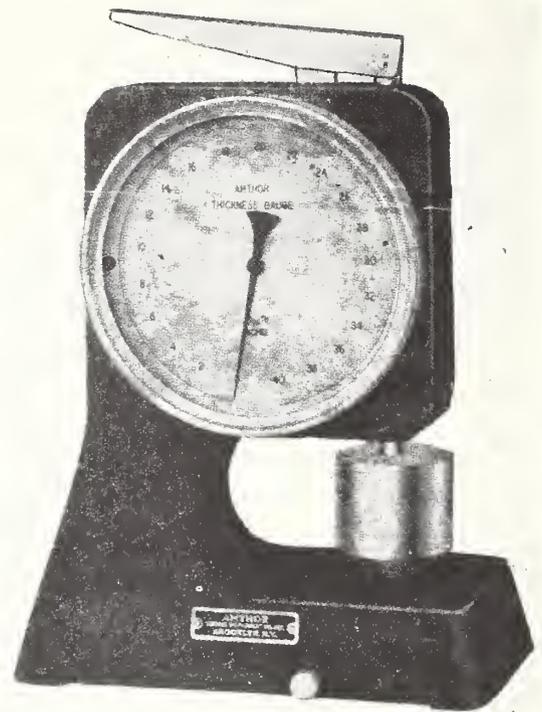


FIGURE 25. *Dead-weight thickness gage.*

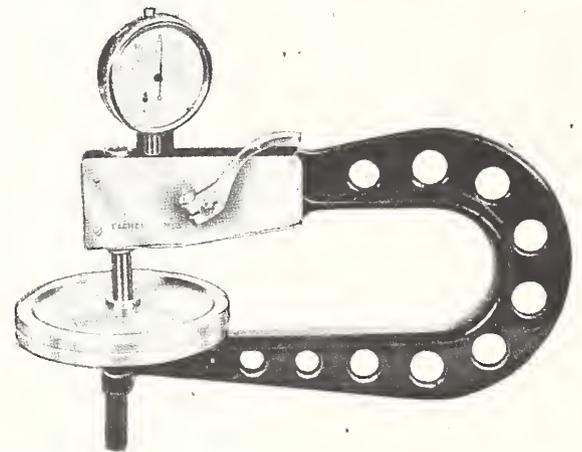


FIGURE 26. "*Pileometer*" to measure thickness of plush.

Manufacturer: Amthor Testing Instrument Co., Brooklyn, N. Y.

(c) "*Pileometer*" (fig. 26). This instrument is used to measure the thickness of plush, carpets, etc. Capacities of the standard types are 500, 750, and 1,000 mils. The disk has a diameter of 4 in. One revolution of the dial gage pointer represents 100 mils, with graduations of 1 mil.

Manufacturer: F. F. Metzger & Son, Philadelphia, Pa.

(d) "*Measure-Matic Gage*" (fig. 27). This gage is used to measure mats and blankets of loosely associated fibrous materials such as jute, wool, or glass fibers. The platen covers an area 1 ft² under a load of approximately 2 g/in.², which requires a total weight of 0.635 lb, with

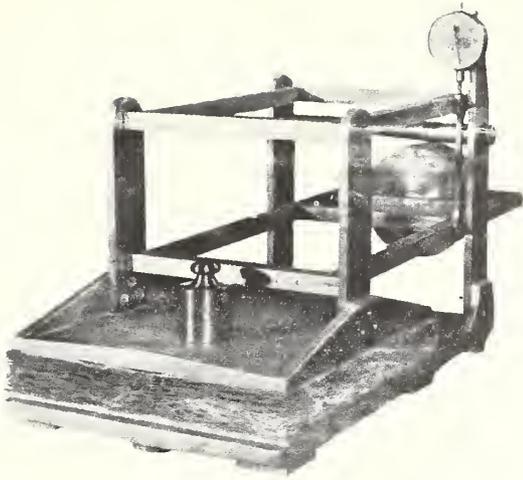


FIGURE 27. "Measure-Matic" gage to measure thickness of fibrous materials.

the material extending to or beyond the platen on all sides. Before the measurement is made, the platen is balanced by adjusting the counterweight on a threaded rod behind the uprights. The movement of the platen relative to the table is transmitted to a dial indicator giving the distance between table and platen with an accuracy of 10 mils up to 5 in. total distance.

Manufacturer: Gustin-Bacon Manufacturing Co., Kansas City, Mo.

1.145. *Special Designs* (figs. 28, 29). Many special designs of dial thickness gages have been developed. A few examples follow: The Federal Products Corp. portable dial gage (fig. 28) has wide-face spring-loaded upper and lower anvils to hold the gage perpendicular to the sheet surface. The ring thickness gage (fig. 29), of Federal Products Corp., has a movable gage system which aligns itself radially. The Guyer gage, used for rapidly checking the thickness of large sheets, clamps on the sheet just long enough to read the dial, and then is automatically released for removal to the next point to be measured. Up to 38 measurements per minute are possible.

(e) *Holt Gage for Rubber Specimens* [116a]. Screw micrometers combined with a very sensitive electrical-contact indicating device have been adapted to measuring the dimensions of soft rubber parts. Two vertical gages have been built for thickness measurements, and a horizontal gage for the diameter of cylindrical or spherical parts. The measurements agree closely with those computed from the volume because the specimens are compressed only very slightly in making the electrical contact.

1.146. *Roller Gages* [177] (figs. 30, 31). Roller gages are used for the continuous measurement of moving strips, as in drawing wires or in coating wires with insulating material. The anvils are replaced by balls or rollers. This can be done in various ways. In a "single roller gage" (fig. 30) (Federal Products Corp.) the

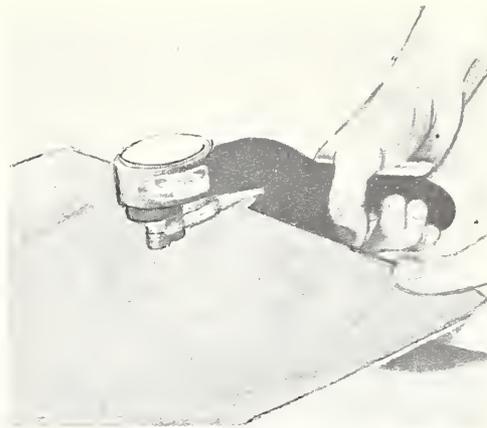


FIGURE 28. Sheet-metal dial micrometer.

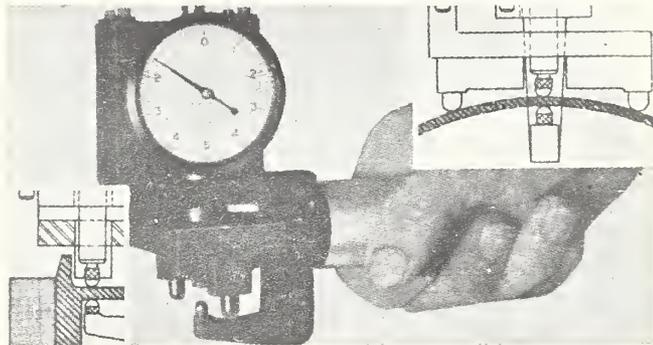


FIGURE 29. Ring-thickness dial micrometer.

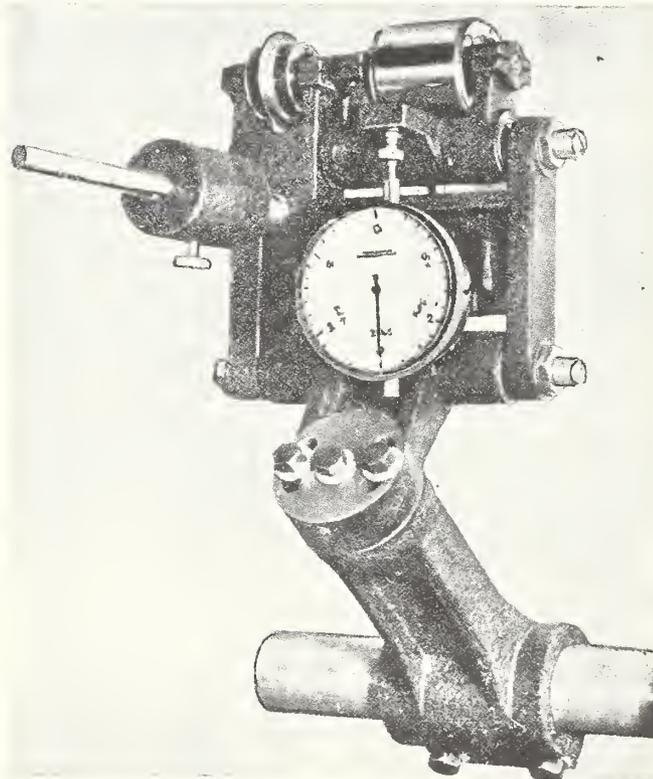


FIGURE 30. Single-roller gage.



FIGURE 31. Differential-roller gage.

roller on the gage bears against one side of the stock while the other side is in contact with a reference surface, a ground roller, or a platen over which the strip can be passed.

When the material is passing over a calender roll, a "differential roller gage" (fig. 31) is used, in which a cylindrical roller rides on the stock and a barrel-shaped roller rides on the calender roll. Such double roller gages carry their own reference roll as well as a measuring roll, and are particularly suited to measure the thickness of wet paper stock before it enters the calender rolls.

"Floating gages" permit the gaging head to follow variations in the pass line of the work while keeping the reference roll in contact with the underside. They are used to measure rods that are not perfectly straight.

Manufacturer: Federal Products Corp., Providence, R. I.

1.147. Averaging Gages. In many cases it is sufficient to maintain an average thickness. To measure the average, it is necessary to introduce a lag or damping into the indications (up to 100 seconds for slow moving material). This can be done most simply by using mechanical-electrical gages and damping the moving-coil indicator.

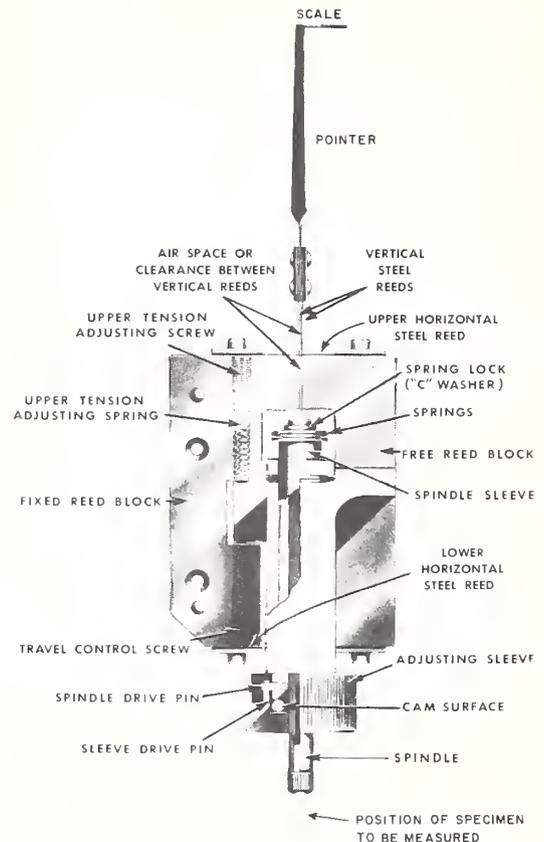


FIGURE 32. Reed gage for mechanical magnification of displacement.

1.15. Dial Gages, High Mechanical Magnifications.

1.151. Parallel Reeds [7] (fig. 32). Two vertical reeds of thin steel are used to measure and magnify displacement by mechanical means (fig. 32). They move parallel to one another, one being held by a fixed block, the other by a free-moving block. The relative movements of the reeds are magnified so that the pointer swings through a much wider arc. The amount of its swing is proportional to the distance the floating block is moved. These reed gages are ordinarily used with additional optical magnification (see 1.164).

1.152. Twisted Ribbon [1] (fig. 33). The basic design of the twisted ribbon gage for the mechanical magnification of displacement is shown in figure 33. It is generally known as the "Swedish Gage" and by the trade name "Mikro-kator." The pointer is mounted on the middle of a twisted strip of phosphor bronze. The twist of the strip is left-handed on one side of the pointer and right-handed on the other side. When such a strip is stretched, the center portion carrying the indicator pointer will rotate about an axis through the center line of the strip, i.e., unwind. The sensitivity is increased

by perforating the central portion of the strip. The diaphragm that supports the spindle at the lower end is cut out as shown in "A" to permit free vertical movement of the spindle. The upper end of the spindle is fastened to a plate spring. A coil spring, which is adjusted by a collar on a spindle, furnishes the required force between feeler and specimen.

The standard instrument uses a strip of phosphor bronze 0.06 by 0.0025 mm, 40 mm long, twisted 4,000 degrees, and has a tapered glass pointer 30 mm long. The magnification is about 5,200. Magnification about 10 times higher is obtained with the leverage of a "spring knee." The force required to produce a 90° rotation is as low as 0.3 gram.

Using only mechanical means, the manufacturer claims as much stable magnification as 200,000. Using a mirror and a light beam 900 mm long, the magnification can be brought up to 12 million. The least sensitive standard instrument has a measuring range of ± 3 mils, the most sensitive, a range of ± 0.1 mil (2.5μ). The latter is graduated to 0.1μ . Special designs have a range of $\pm 1 \mu$ and graduations of 0.01μ , with an operating force of 0.3 gram. The instruments are automatically protected against overload and possible damage by shoulders on the

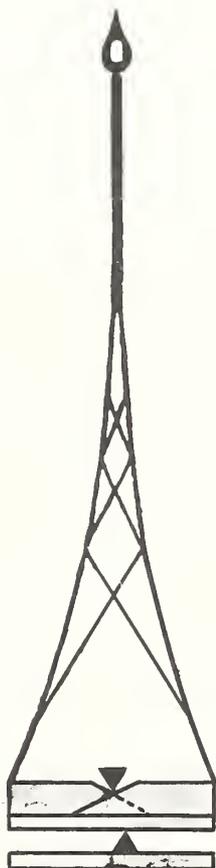


FIGURE 33. Twisted ribbon gage for mechanical magnification of displacement.

spindle which engage corresponding shoulders on the body.

Manufacturer: C. E. Johansson Gage Co., Eskilstuna, Sweden, and Swedish Gage Co. of America, Detroit, Mich.

1.153. *Tilting Block* (fig. 34). A lever construction for mechanical magnification is shown in figure 34. The "Comparitrol" (trade name for this design) uses a V-notch block, which is tilted by the feeler point, a steel ball, or a diamond or tungsten-carbide point (the lower pivot in the figure). The magnification obtained in this way is 500 for a standard model, 1,000 for special instruments. The scale length is ± 1.5 in. It was manufactured by George Scherr Co., New York, N. Y., but production has been discontinued.

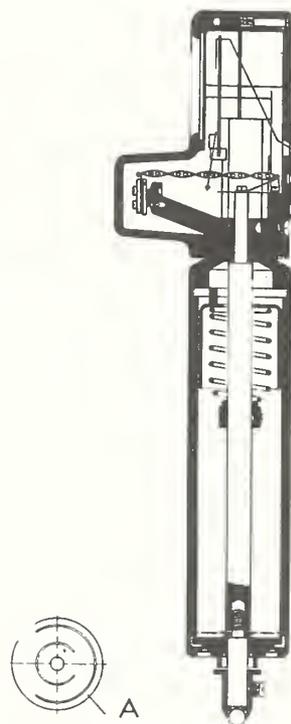


FIGURE 34. Lever magnification of movement.

1.16. Dial Gages, Optical Lever.

1.161. *Mirror Principle* (fig. 35). Optical magnification of angular movement caused by the displacement of an anvil brings a number of advantages:

(a) The reflecting mirror adds very little weight and moment of inertia to the moving system, and such systems generally withstand shocks and overload better than instruments with long pointers.

(b) Reflection on the mirror provides magnification of 2, which means that an optical system with 4-in. radius is effectively an 8-in. mechanical pointer.

(c) Very long scales can be obtained with several designs. Figure 35 shows the principle

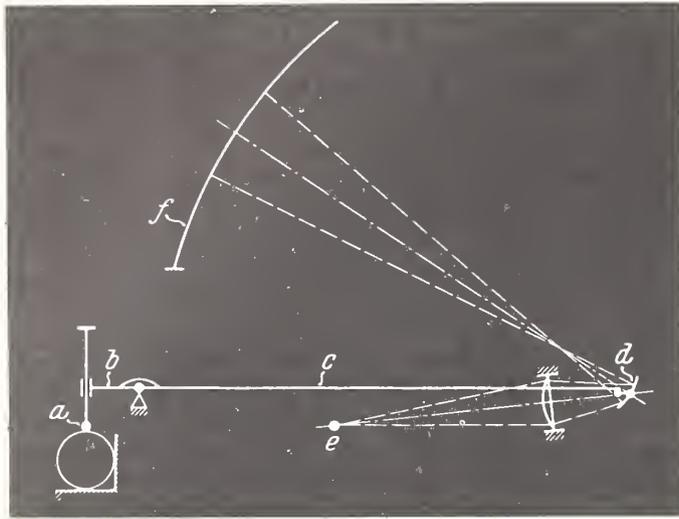


FIGURE 35. Optical magnification of displacements.

of optical magnification. The feeler (a) rests on the piece under test and is connected with the short end (b) of the two-arm lever (b-c). At the end of the long lever is the mirror (d) reflecting a light beam coming from the small lamp (e). Indication is given on the scale by a bright circle with a dark diameter line.

1.162. Roller Gage with Optical Magnification (fig. 36). Detail of the optical arrangement, used in a steel mill to indicate the movements of a roller gage, is shown in figure 36. The lower roll (it may be a drum) is fixed, and the upper roll swings in a fixed support, positioned according to the thickness of the material. The concave mirror is illuminated by a low-voltage, 50-watt lamp. The scale is at a distance of 10 ft and has a length of 40 in., which corresponds to a thickness of 90 mils. The magnification in this example is 450.

1.163. "Projectometer" (figs. 37, 38). A portable instrument for the measurement of thickness of individual test pieces uses a tilting mirror and an optical magnification system (figs. 37 and 38). An important measuring element of this instrument is a master glass scale of high accuracy. When the tested piece is put under the contact tip, a greatly magnified image of both scale and index is thrown against a screen so that a distance of 0.1 mil is enlarged to 125 mils, a magnification of 1,250. This image is further enlarged by a 1.5x magnifying lens. A 6v-5a lamp is used for illumination. The measuring accuracy is given as 0.005 mils.

A more recent development, known as the "Ultra-Projectometer," has a magnification of 10,000. The scale divisions are directly in 0.005 mil, which, with the 400 divisions of the standard projectometer scale, give a total range of ± 1 mil.

A direct-reading thickness meter, the "Leitz Vertical Measuring Machine," has a master scale 4 in. long and gives measurements in 0.05

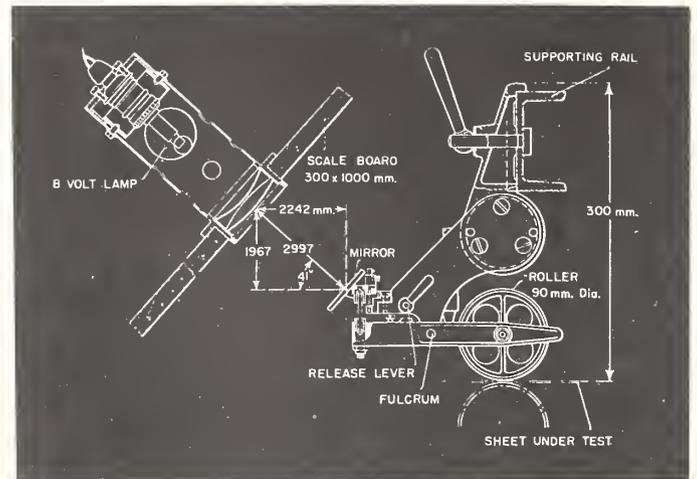


FIGURE 36. Optical magnification of roller movement.

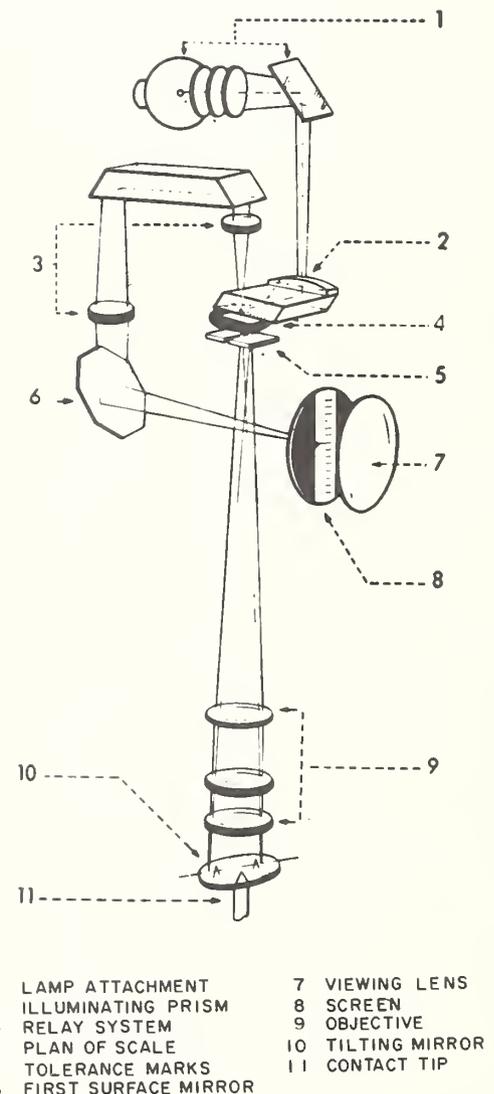


FIGURE 37. "Projectometer," optical magnification.

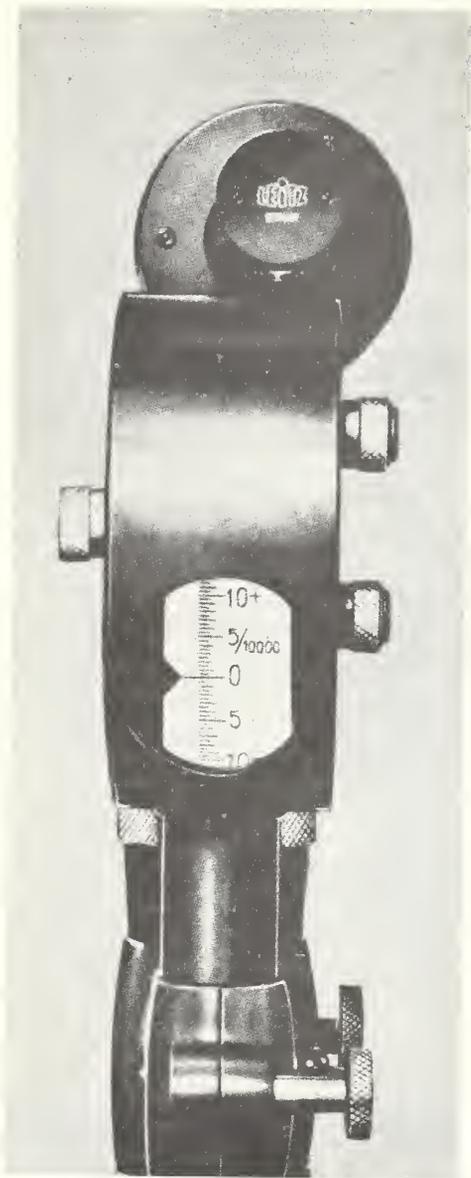


FIGURE 38. "Projectometer," reading scale and measuring head.

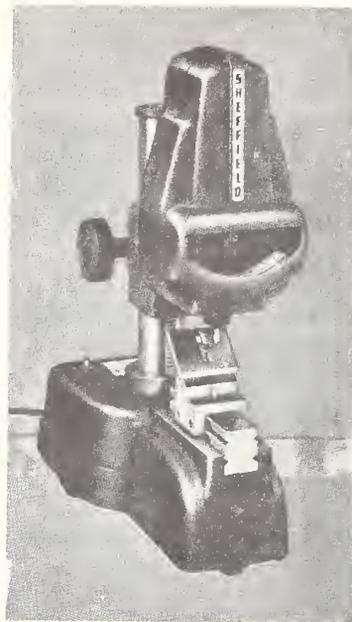


FIGURE 39. "Visual gage," using both mechanical and optical magnification.

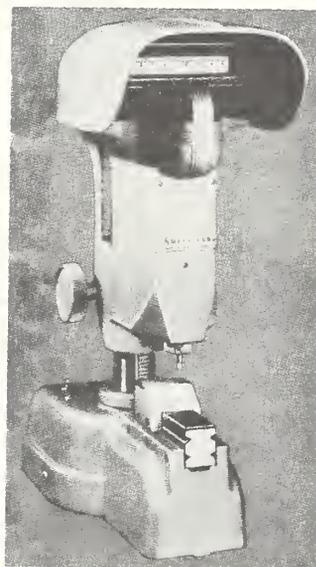


FIGURE 40. "Visual gage" for magnifications of 5,000 and 10,000.

mil. The magnification of this instrument is 2,700, and the tolerance is given as ± 0.008 mil.

Manufacturer: Leitz, Wetzlar (Germany).

1.164. "Visual Gage" (figs. 32, 39, 40). This instrument (fig. 39) uses the reed magnifier (fig. 32) (see 1.151) to get a pointer displacement proportional to the anvil movement. In one of the two designs, the end of the pointer bearing a flag is projected optically onto a curved scale, which has a length of 4 in. at the bottom of the opening in the gage housing. This meter is built for magnifications of 500, 1,000, and 2,000, the highest magnification showing 1 mil as 2 in. With a different design of the optical magnification (fig. 40) using a longer beam of light, magnifications of 5,000 and 10,000 are obtained on a scale 5 in. long. Here, 0.1 mil is represented by a deflection of 1 in.

Manufacturer: Sheffield Corp., Dayton, Ohio.

1.20. Pneumatic Gages [3, 4, 6, 7, 67, 85, 104, 123, 176, 187, 213] (pats. 3, 4).³ In pneumatic gages the flow of compressed air is influenced by the mechanical dimensions of the work under test. With increasing distance of the jets from the piece under test, the air flow to the gaging head increases and an indicator may be directly calibrated in units of thickness.

There are two basic types of pneumatic gages: Back-pressure and rate-of-flow gages.

³ A list of patents is given on page 75.

1.201. *Back-Pressure Gage* [4, 67, 104, 116] (figs. 41, 42, 43, 44). The fundamental form of the back-pressure gage for measuring thicknesses of individual pieces (fig. 41) consists of an air supply of constant pressure entering an orifice of predetermined size, with a pressure-regulator valve and an indicator on the constant pressure supply. A filter removes excess moisture and other foreign materials, which might spoil the parts tested or foul the small orifice through which all the air must pass. The pressure at the entrance of the restrictor orifice is identical with that in the large-bore tube whose open lower end is submerged in a tank of water. The constant pressure in the tube can be changed only by changing the water level in the tank. A small vertical sight-glass has one end

connected to the conduit of the gaging head, the other end to the lower portion of the water tank. When conduit and gaging head are open this indicator will show, under atmospheric pressure, the height of the water level in the tank. Increasing back pressure, with increasing resistance to the flow of air out of the gaging head, depresses the water level in the indicator tube. Because of the necessary height of the water tank, this type of gage seldom operates at more than 1 psi (1 lb/in.²) air pressure. Higher pressures can be used with mechanical pressure gages which measure the constant supply pressure and the working pressure below the restricting orifice.

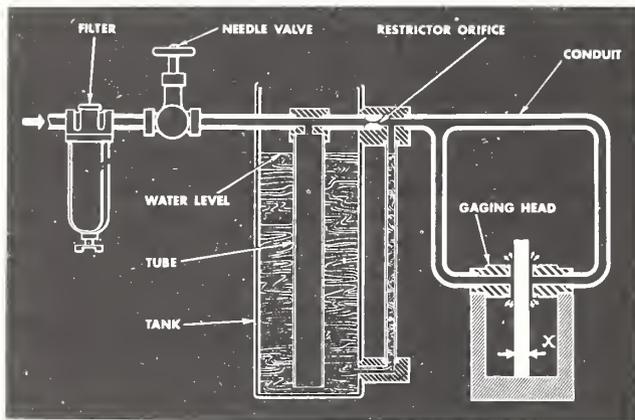


FIGURE 41. *Water-column back-pressure air gage.*

Figure 42 shows the principle of the "C" type of gage using an adjustable compensator. By means of its automatic orifice the compensator maintains a constant flow of air regardless of the gaging pressure. This allows approximately twice the nozzle clearance and twice the wear allowance of gages with a simple orifice. The maximum magnification of these gages is 8,200, which means that a deviation of 0.1 mil from the standard value is indicated by a pointer deflection of 0.82 in.

Manufacturer: Moore Products Co., Philadelphia, Pa.

Another type of back-pressure pneumatic thickness meter is the "Dimensionair" (figs. 43, 44). Pressure indication is given either by a dial manometer for high pressure (40 to 100 psi), or by a glass tube manometer for low

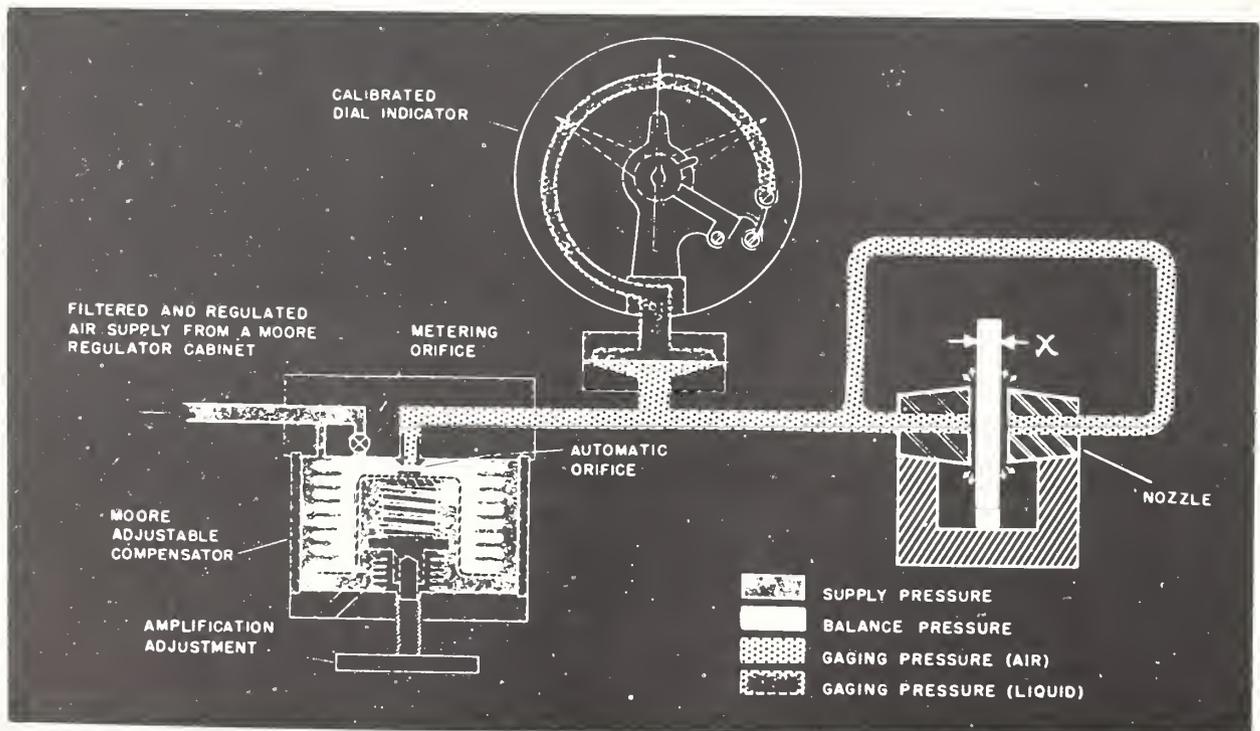


FIGURE 42. *Pneumatic back-pressure gage with adjustable compensator.*

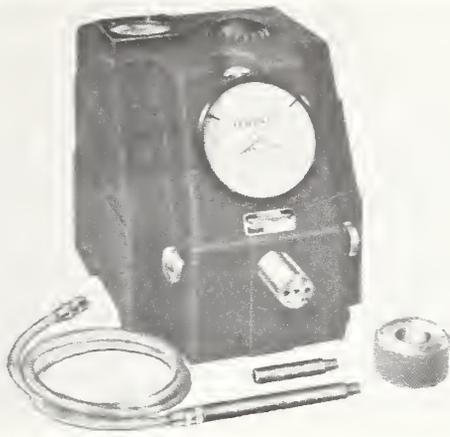
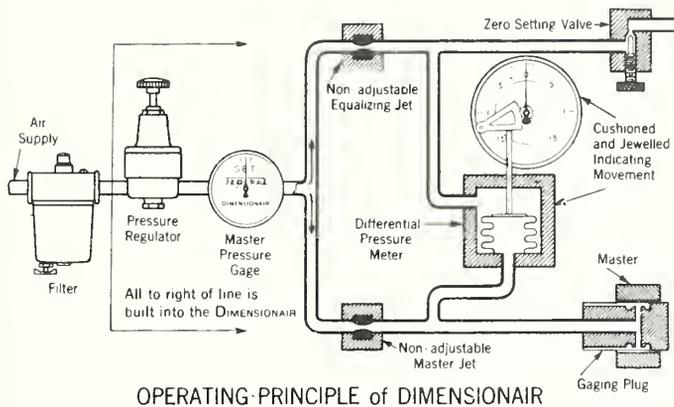


FIGURE 43. "Dimensionair" pneumatic gage.



OPERATING-PRINCIPLE of DIMENSIONAIR

FIGURE 44. "Dimensionair" pneumatic gage.

pressure (1 to 2 psi). The magnification is 2,500 with the high-pressure type, 4,000 with the low-pressure instruments. The range is ± 1.5 mils, the scale length 7.5 in.

Manufacturer: Federal Products Corp., Providence, R. I.

A pneumatic micrometer [176] gives a magnification of 32,000 with a measuring range of 10μ (0.4 mils). The 300° indicator scale has only 10 divisions, each 32 mm (1.25 in.), and is equipped with contacts for make-and-break control.

1.202. Continuous Gage for Threads and Wires [25, 85]. The National Physical Laboratory in London developed a pneumatic gage for the wool industry in England [25, 85] for measuring the diameter of threads or wires in the range 7 to 10 mils, with continuous indicating and recording. The measuring head consists of a small box with entrance and exit orifices through which the filament is run at speeds up to 1,000 ft/min. Air blown into the head through a tube at the top escapes through the two orifices. The material fills the orifices almost completely, and the closer the fit, the higher the sensitivity. The pressure measurement is made by a spring-loaded piston which displaces $\frac{1}{16}$ in. for 1 in. of water pressure.

The displacement is measured with a pneumatic amplifier so that pressures from 2 to 14 psi are obtained over the range of the instrument.

The indicating instrument has a 3-in. dial and the pointer makes one revolution for a piston movement of 100 mils. The scale has a length of 6 in., and because the piston has already magnified the change of the measuring jet 1,000 times, the total magnification is 60,000. One scale division of 10 mils represents jet displacement of 0.001 mil.

1.203. Continuous Gage for Moving Strips [67, 104] (pat. 3) (fig. 41). *Solex*. Instruments have been developed for use in rolling mills by the Societe d' Application de Metrologie Industrielle in Courbevoie (Seine), France, under the name "Solex." In the simplest design (fig. 41) compressed air flows under constant pressure from a tank into a chamber, from there through the measuring orifices. The back pressure behind the two orifices, one on each side of the strip, indicates the thickness. The differential pressure is read on a manometer. The magnification of this instrument is 4,800, so that a thickness change of 1μ is indicated by a scale deflection of 4.8 mm. For a total measuring gap of 35μ the range of the instrument is $\pm 5 \mu$ and the resolution 0.5μ .

The French company is represented in the United States by Arnolt Corp., Warsaw, Ill.

Etamic. Another continuous gage for moving strips, Etamic (pat. 5), uses air flowing at critical (sonic) speed in nozzles under high pressure (about 50 lb/in.²). The strip to be measured passes between two orifices located on either side of the strip. Both of these orifices are located at the downstream end of a measuring tube which has a calibrated nozzle at the upstream end. The change in absolute pressure in the region between the upstream nozzle and the gaging nozzle, formed by the orifice and the strip, is a linear function of the distance, d , if each orifice is circular, has a diameter D , and is a distance $d < D/4$ from the strip.

The gage can be set to operate at any standard capacity from 0 to 10 mm thickness. The accuracy is given as $\pm 1 \mu$ for a measuring range up to 80μ . The sensitivity is given as 0.1μ . Maximum and minimum tolerance limits can be set to any value between 1 and 100μ . The clearance between orifice and strip must not be less than 170μ .

Manufacturer: Ateliers de Normandie, Paris, France.

1.204. Rate-of-Flow Gage [6, 7] (figs. 45, 46, 47). In this type of gage, the restrictor orifice is completely eliminated. Air is supplied at constant pressure (through filter and regulator) and the rate of air flow escaping through the gage head is measured. The rate-of-flow meter used consists of an internally tapered transparent tube, vertically positioned, with the wide

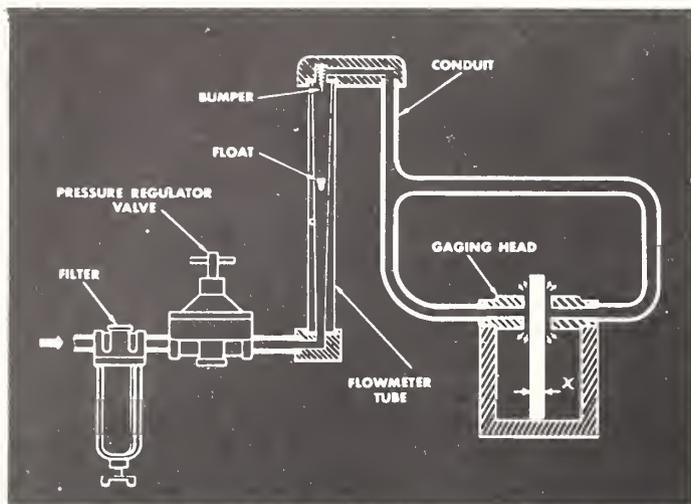


FIGURE 45. Pneumatic gage operating with constant pressure and indicating dimensions with a rate-of-flow air gage.

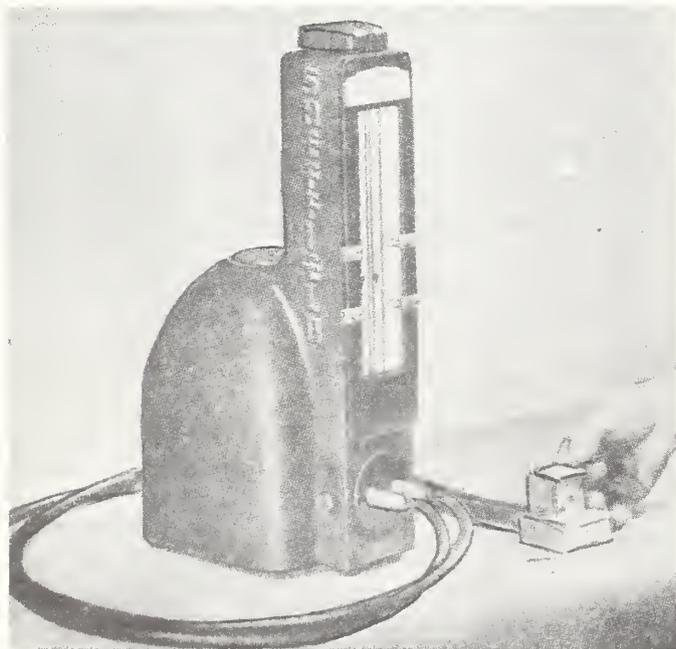


FIGURE 46. "Precisionaire" thickness meter and measuring head.

end upward, generally called a rotameter. A light-weight metal float positions itself in the taper according to the velocity of the air passing through the tube, so that the position of the float is a direct indicator of rate-of-flow and hence of thickness. Figure 46 shows a complete instrument, the "Precisionaire" of the Sheffield Corp. To measure thickness, special gages have been developed with two sets of directly opposed jets (fig. 47), so that perfect contact between the piece under test and the reference surface is not necessary.

The sensitivity of these pneumatic gages is very high. Standard magnifications range from 1,000 to 40,000, representing a dimensional difference of 1 mil as a float movement in the

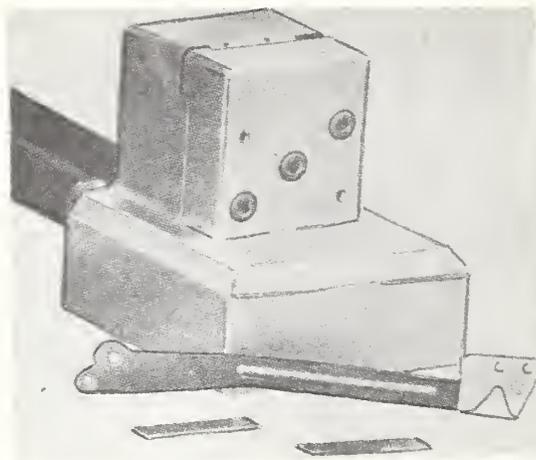


FIGURE 47. "Precisionaire" thickness meter and measuring head.

indicator tube of 1 to 40 in. For the maximum magnification, with a scale length of 8 in., a deviation of ± 0.1 mil is ± 4 in. As far as accuracy is concerned, it must be noted that all pneumatic gages, like most other mechanical gages, depend on calibration with a sample of known standard thickness, for which the float is set to zero by adjustment of a bleeder valve. In general the deviation from this standard thickness is not magnified more than 10,000 times. The indication of this deviation is affected by changes of the supply pressure, which, however, can be kept within very small limits.

Manufacturer: Sheffield Corp., Dayton, Ohio.

There are other ways of measuring the rate of flow to the gaging head. An electrically heated wire may be placed in a tube and the cooling effect of the escaping air measured in the conventional way, for instance with a Wheatstone bridge arrangement. The device described in [154] may be used for measuring the thickness of wires and threads. This introduces the advantages of electric indicating, controlling, and recording to pneumatic thickness meters.

To measure the fineness of textile fibers a fiber grader ("Micronaire") has been developed by the Sheffield Corp. Air is forced through a plug of fibers and the air flow is greater for coarse than for fine fibers. The repeatability for cotton fibers is given as $0.1 \mu\text{g}/\text{in.}$, or 0.36 mg per 100 yards.

1.21. Deflection. An instrument known as "Bondimeter" has been used to measure the thickness of metal sheets in airplane structures with only one side accessible. The gage consists of an inverted cup or shell with a top wall of transparent material and the bottom edge faced with a rubber gasket. Mounted inside the shell is a dial indicator, graduated in mils, equipped with a stem reaching to a spherical foot which, when the instrument is in use, rests against the test surface. A valve in the top of the case is

connected to an external pump so that the device can be partially or completely evacuated.

When in use, the gage is placed on the area to be inspected, the valve opened, and the air inside the instrument allowed to reach normal atmospheric pressure. A reading is then taken which serves as a reference standard for the test. With the valve again closed, the air is evacuated and the sheet metal deflected upward into the cup. A new reading of the dial is taken, and the difference of the two is compared with precalibrations on samples of the same metal, with the same mechanical properties and a known thickness.

Manufacturer: American Instrument Co., Silver Spring, Md.

1.22. Vibration [38, 50, 71, 79, 81, 82, 89, 94]. If the material under test is in the form of sheet or strip and if it has good elastic properties, the thickness may be determined by the vibration frequency of a reed of known width and length cut from the material. A thickness change of 1 percent causes a frequency change of 3 percent.

1.221. Sonic. The time of reflection of a sound signal from the bottom of the sea gives the thickness of the layer of water under the sound emitter mounted in the bottom of a ship. The speed of sound in water is 1,450 m/sec, hence a depth of 725 m is indicated by a time delay of 1 sec. In metals the speed of sound is several times higher. In a piece of steel, 0.32 in. thick, the echo will return in about 1 μ sec. Sound-reflection methods are also used in geophysical exploration to determine the thickness of different subsurface layers.

1.222. Ultrasonic [38, 39, 50, 71, 81, 82, 89] (figs. 48, 49, 50, 51, 52, 53). *Sonigage* (fig. 48). This is one of a number of industrial thickness meters using the reflection of ultrasonic waves. The measurement is made by bringing the work piece into resonant vibration in the test direction and measuring the resonance frequency. Driving energy is provided by an electronic oscillator and a small quartz crystal which is pressed against the work piece. The frequency of the oscillator is changed until the indicator gives a maximum deflection and, for any given material, the frequency dial may be calibrated in units of thickness. The frequencies employed for thickness measurement of steel are in the range of 1.4 to 2.8 megacycles per second for a thickness range from 0.125 to 12 in. By switching several inductance coils into the oscillating circuit and by interchanging crystals, a wider variety of measurements can be handled by a single instrument. One quartz crystal may be used over a frequency range of about 2:1. The natural frequency of the crystal is always much higher than the frequency used for the measurement.

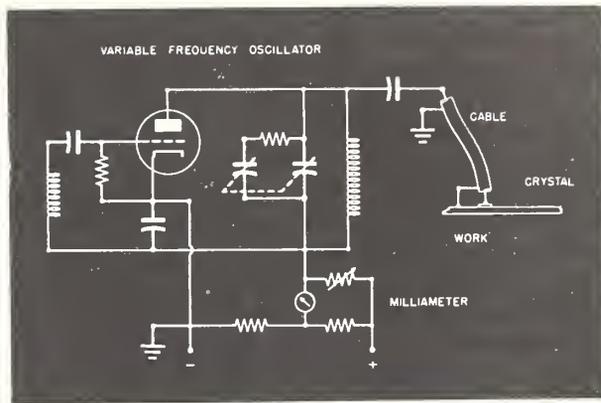


FIGURE 48. Circuit of the "Sonigage."

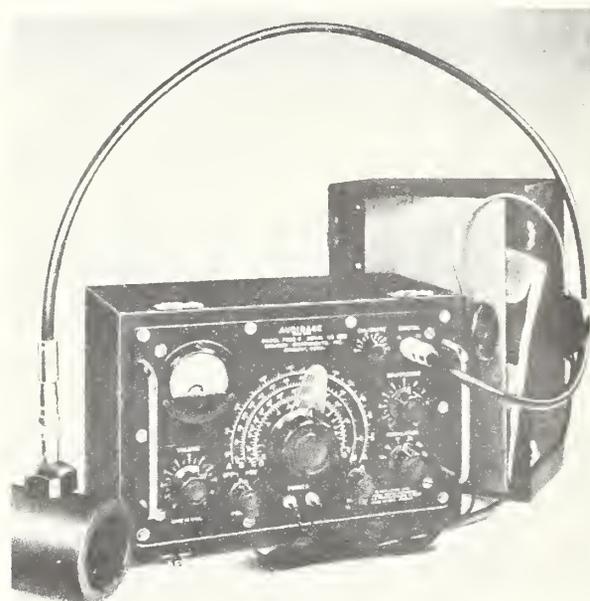


FIGURE 49. "Audigage" thickness meter.

Audigage. Another instrument of this type [38, 39], the "Audigage" (fig. 49), uses a headphones in place of the indicating meter, taking advantage of the fact that the ear is faster in response than sensitive moving-coil instruments. The operator is free to use his eyes for other duties, e.g., best placement of the transducer probe.

Manufacturer: Branson Instrument Co., Stamford, Conn.

In another design [81, 82], the tuning capacitor is rotated continuously by a small motor. The indicator is a cathode-ray oscilloscope. The horizontal deflection plates are connected with the sweep voltage, and the plates for the vertical deflection represent the output of the oscillator. When the oscillator frequency is equal to the resonance frequency of the object, a vertical deflection is obtained, as shown in figure 50. Thickness scales are printed on transparent slides and changed with the range of the instrument.

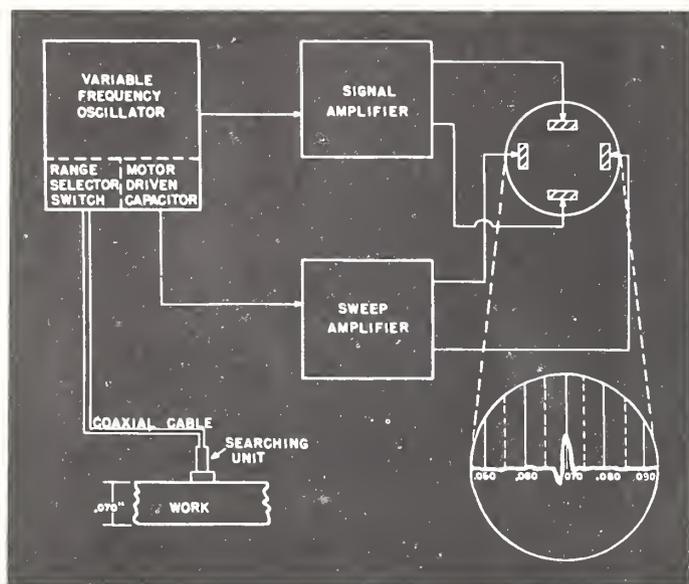


FIGURE 50. Block diagram showing the major components of the "Reflectogage."

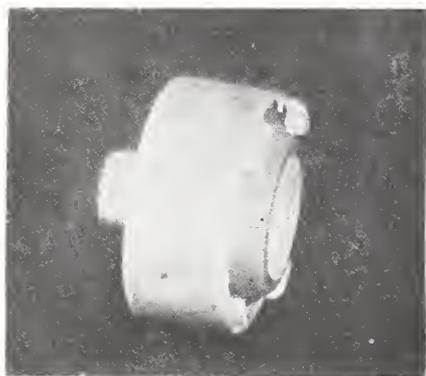


FIGURE 51. Crystal transducer searching unit.

Reflectogage. The design of the "Reflectogage" [50, 71, 89] (figs. 50, 51, 52) has the four ranges, 25 to 50 mils, 45 to 90 mils, 80 to 160 mils, and 150 to 300 mils.

Manufacturer: Sperry Products Corp., Danbury, Connecticut.

Sonizon. The "Sonizon" ultrasonic thickness gage, under license of General Motors Corp., has the ranges 12.5 to 25 mils, 25 to 50 mils, 45 to 90 mils, 80 to 160 mils, 150 to 300 mils, and 250 to 500 mils. It is possible to measure thickness above the nominal maximum by reading the indications from higher harmonics. If the range up to 90 mils is selected and a piece of 240 mils tested, there will be two readings of 80 and 60 mils on the scale. The general relation is

$$T = Rr / (R - r)$$

(eg., $80 \times 60 / 80 - 60 = 240$)

where T is the actual thickness, R the higher reading, and r the lower reading on the scale of the oscilloscope.

Manufacturer: Magnaflux Corp., Chicago, Ill.

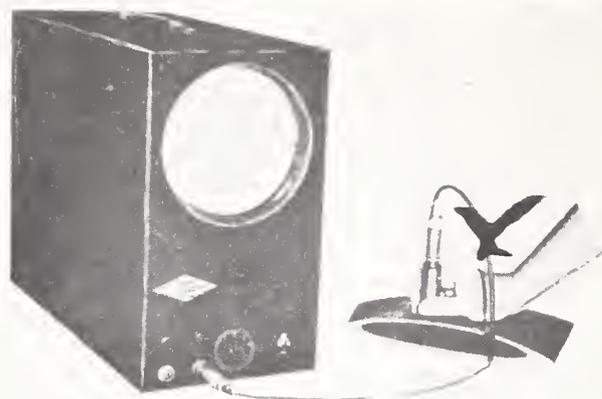


FIGURE 52. Typical application of the "Reflectogage." Testing a hollow propeller blade for thickness.



FIGURE 53. "Metroscope" thickness meter.

Metroscope. A similar instrument (fig. 53), based on the same patents, is manufactured under the trade name "Metroscope" by Photicon Research Products, Pasadena, Calif.

Mechanical-Electric Methods. The operating principle of all mechanical-electrical gages is a mechanical feeler element which is in contact with the work and follows all changes of thickness with a variable displacement of the feeler. This displacement is transformed by resistive, inductive, or capacitive devices into proportional electrical signals, which are used for indicating, recording, or controlling at a distance.

1.23. Displacement With Resistive Pickup.

1.231. Slide Contact [62]. Basically a dial-gage mechanism moves a slide contact on a resistive voltage divider, which is fed by a constant voltage supply, and operates a volt-

meter calibrated in displacement of the dial-gage spindle or thickness of the specimen under test. In a British design [62] a conventional dial micrometer was equipped with a bare resistance wire of 3 ohms resistance around the scale, and a small contact was mounted at the end of the pointer. The pointer contacts the resistance wire every 2 seconds (for recording), and is free to rotate normally for the rest of the time.

1.232. *Resistive Strain Gages* [99] (figs. 54, 55, 56). Small movements, representing thickness or change or difference of thickness, can be

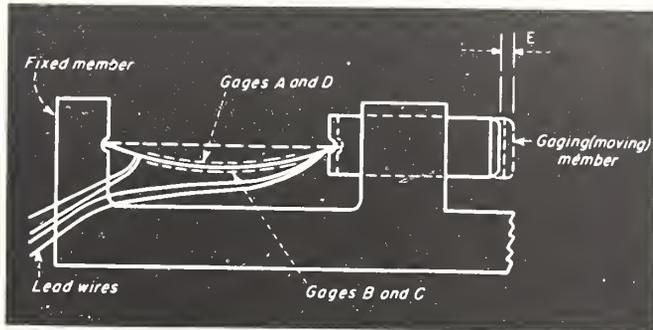


FIGURE 54. Bonded wire strain gage, as used for measuring thickness.

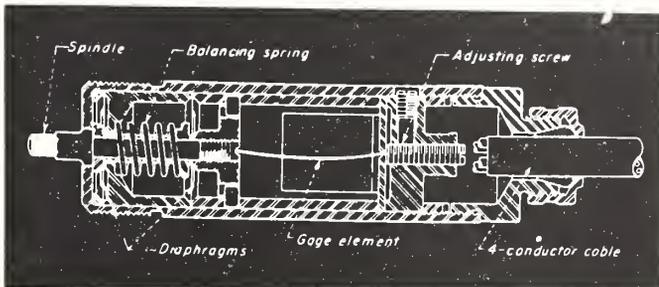


FIGURE 55. Cartridge-type displacement transducer with bonded wire strain gage.



FIGURE 56. Brown & Sharpe "electronic thickness gage."

The SR4 strain gages are in the head of the "external comparator" at right; the 1,000-cps oscillator, amplifier, and indicator are in the measuring unit at left.

transformed into resistance change by resistive strain gages. Wire strain gages, which are almost exclusively the type used in this country, may be of either the bonded or the unbonded type. The bonded SR4 strain gage (Baldwin Locomotive Works) consists of a fine resistance wire, with a diameter of 1 mil or less and generally a resistance of 120 ohms, wound in meander shape, pasted on paper and cemented to the test object. When the gage is stretched 0.1 percent of its length, the resistance of the wire increases 0.2 percent or more, depending on the gage factor of the material, defined as the proportional change of resistance to length. For a gage length of 10 mm we get an elongation of 0.01 mm (0.4 mils), and about 0.24 ohms resistance change, which, with a gage current of 10 ma, gives a signal of 2.4 mv. Usually four of these gages are connected in a d-c Wheatstone bridge in such a way that two gages are stretched, two others compressed, and the sensitivity (millivolts/elongation) is increased four-fold.

A commercial application of strain gages for measuring thickness is the Brown & Sharpe electronic thickness gage [99], built with SR4 gages which are mounted in a flat spring held at both ends (figs. 54, 55, 56). The power supply is 2.8 v, 1,000 cps ac. Motion of the feeler produced by the varying thickness of the specimen deflects the spring gage element and thereby changes the resistance of the attached resistance wire gages. The scale of the indicating instrument has a length of 3.6 in. and 20 divisions. At the highest sensitivity one division of 180 mils represents a displacement of 0.01 mil, giving a magnification of 18,000. The instrument indicator has five sensitivities, 0.01, 0.02, 0.025, 0.05, and 0.1 mil corresponding to the ranges 0.1, 0.2, 0.25, 0.5, and 1.0 mil.

Manufacturer: Brown & Sharpe Mfg. Co., Providence, R. I.

1.233. *Variable-Resistance Spring* [216]. The variable-resistance spring transducer can be used to measure small displacements. The active element of the device is a helical or conical spring with a corrosion-free surface, wound in such a way that the initial tension varies slightly along its length. Thus, when the ends of the spring are pulled apart, the turns separate one by one, not all at the same time as with an ordinary helix. The entirely closed spring has the resistance of a cylindrical tube, and the completely opened spring has the resistance of the total length of the coiled wire. Displacements (or thickness changes) of 0.01 mil can be measured without the use of amplifiers. The accuracy of this gage is given as ± 10 percent of the indicated deviation from the standard thickness. The "gage factor" may be 100 and even higher, which means that a displacement of 0.1 percent of the spring length may cause a resistance change of 10 percent or more.

1.234. *Linear-Motion Potentiometer.* Precision wire-wound linear-motion potentiometers are available with displacement ranges of 0.5 to 20 in. Maximum departure from linearity may be as low as ± 0.5 percent. Operating force may be as low as ± 0.5 percent. Operating force may be as low as 0.5 oz.

1.24. *Displacement With Inductive Pickup.* In this type of instrument the thickness of the sample, frequently a moving sheet, causes a mechanical displacement which in turn causes the unbalance of an electric-inductive circuit and generates a voltage in proportion to the relative movement of a coil.

1.241. *Mutual Inductance* [113, 114, 144]. One design of mutual-inductance gage, applied to measure the thickness of nonmagnetic moving sheets, consists of a standard head, a measuring head, and a 1,000-cps oscillator to energize both heads, which are continuously balanced against each other in a bridge circuit. Standard and measuring heads comprise the primary and secondary windings of special audiofrequency transformers. Changes in the spacing between primary and secondary vary the degree of coupling and hence the secondary voltage. The relationship between displacement and output of the bridge is linear for displacements between 125 and 1,000 mils. The sensitivity of the system is high enough to give full scale readings on the d-c meter with as little as 0.5-mil deviation from the standard thickness. The usable range of the instrument is approximately 1 to 1,000 mils. Production of this thickness meter has been discontinued as a more universal system (see 1.243) is now used.

The Foote-Pierson electronic gage [113] also operates by change of mutual inductance. The gage head contains two coils, one fixed and connected to a 100-kc oscillator, the other mounted on a spindle which is actuated by the work piece. The second coil is coaxial with the fixed coil and, as it moves in accordance with the thickness of the material, a varying voltage is induced which increases linearly with the spindle displacement. The output is rectified and indicated by a d-c instrument. Four magnifications are possible, the highest approximately 14,000, with a 0.2-mil movement of the spindle head producing full-scale deflection on the meter. Other ranges are 0.6 mil, 2 mils, and 6 mils, the corresponding magnifications 4,650, 1,400, and 465.

Manufacturer: Federal Products Corp., Providence, R. I.

1.242. *Double-Airgap Reactance Ratio* [45, 113, 130, 170] (figs. 57, 58, 59). This principle is used for many designs of electromagnetic thickness gages (fig. 58). Gages operating on the double-airgap reactance ratio consist of two electromagnets with a movable armature between them. The armature is made of a magnetic material. As its position shifts from

center, the airgap increases on one side and decreases on the other, introducing corresponding inductance changes in the two circuits. The inductance is increased as the armature is brought close to an electromagnet, decreased as it moves in the opposite direction. Compensation for the change is made with a voltage divider. The amount of compensation necessary to balance the circuit is directly measurable and can be calibrated in units of thickness. The circuit can also be adjusted with a standard so that deviation rather than actual thickness is shown on the indicator.

1.2421. "ELECTROLIMIT" GAGE. The "Electrolimit" gage (fig. 57) is representative of the double-airgap reactance ratio. The circuit is shown in figure 58. As the central core moves from the point of perfect balance, the output voltage is, within wide limits, proportional to the movement of the core. Resistor *R* adjusts the amplitude to compensate for change in supply voltage; voltage divider *S* is used to adjust the point of zero deflection to any desired value. With sensitive moving-coil instruments for indication, the sensitivity can be adjusted to almost any value. Gages of this type can be built to multiply the actual thickness or thickness change as much as 20,000:1, to read down to 0.005 mil on the dial. In one special design, the "Electrolimit Millionth Comparator," the full scale of 10-in. recorder is 0.1 mil, each of the 100 divisions is one millionth of an inch, and the magnification is 100,000.

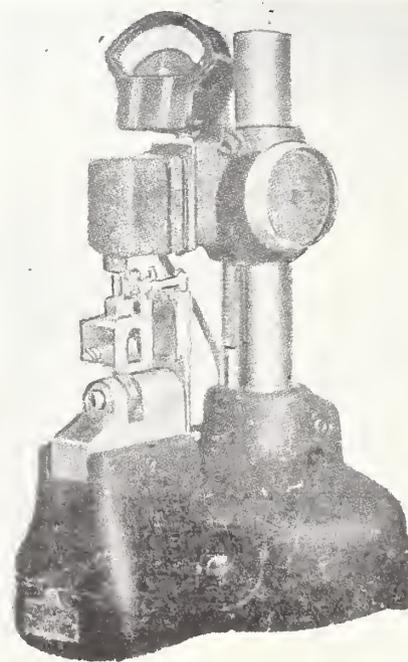


FIGURE 57. "Electrolimit" gage, equipped with a roller gage to measure thickness of 35-mm film.

There are several designs for production supervision (some with the trade name "Magnetic Gage") that use the same measuring principle. One of the designs has an indicator with a 3-in. scale on the top of the measuring head, giving magnifications of 1,000 to 5,000. Another instrument with a separate light beam gives a magnification up to 10,000 on a 5-in. scale.

Manufacturer: Pratt & Whitney, Inc., West Hartford, Conn.

It must be kept in mind that for all thickness meters that transform a displacement into an electrical signal, these figures mean sensitivity rather than accuracy. Accuracy must always be established by inserting dimensional standards into the measuring system and adjusting the electric circuit accordingly.

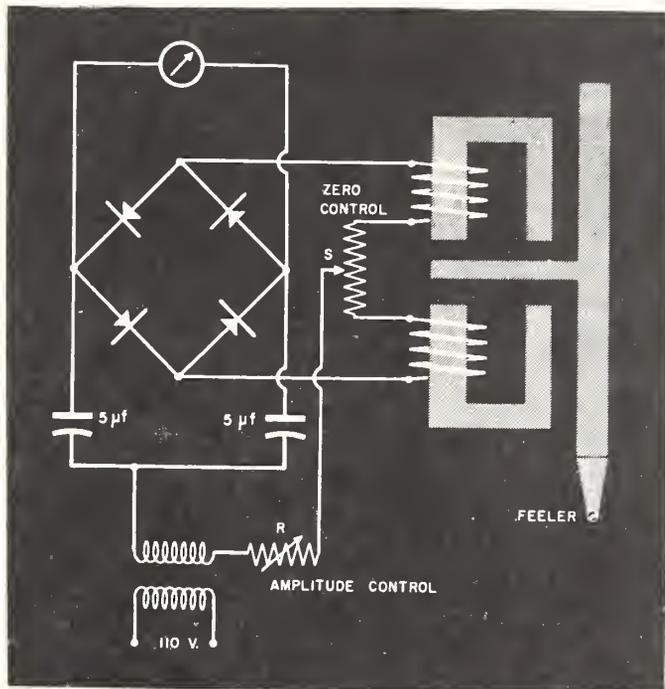


FIGURE 58. "Electrolimit" gage, equipped with a roller gage to measure thickness of 35-mm film.

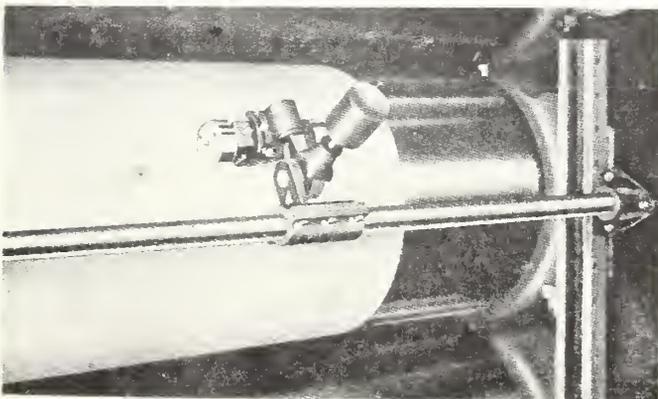


FIGURE 59. Transmitter assembly of a "Schuster gage" to measure the thickness of paper on a calender roll.

Schuster Gage (fig. 59). This is a special design of the "Electrolimit" gage to measure the thickness of sheet materials such as paper, rubber, film, or asbestos, during production. The gaging head consists of a roll carriage, accurately ground rolls, and the Electrolimit coil assembly.

Manufacturer: Pratt & Whitney, Inc., Hartford, Conn.

1.2422. "ELECTRIGAGE." This instrument also operates on the principle of the double-airgap reactance ratio. The range of the standard indicating instrument is 1.2 mils and the scale has 24 divisions of 0.05 mil each. The magnification is 2,500. Interpolated readings of about 0.01 mil are possible.

Manufacturer: The Sheffield Corp., Dayton, Ohio.

1.2423. "METRON" GAGE (fig. 60). Two sets of circular coils, which are connected to the indicating unit by a cable, are embedded and sealed into the core, L, of the measuring head (fig. 60). The coils with the core are inductances which produce a magnetic flux. The flux links the coils, and passes across two airgaps and through the iron armature on the spindle. The airgaps are only a few mils in length, so that with the displacement of the spindle by the thickness of the test object, the flux that links the coils changes by a relatively large percentage, as does the inductance of the coils. The four coils are part of a 60-cycle a-c bridge, fed with constant voltage, the output of which is measured with a sensitive moving-coil instrument across a dry-disk rectifier. The spindle assembly is held in the housing by two circular diaphragms so that there is no friction of the gage pin in its bearings.

In its most sensitive design, this instrument will give full-scale deflection for 0.02 mil. Each division is 0.001 mil, equivalent to a magnification of 100,000. The least sensitive instrument

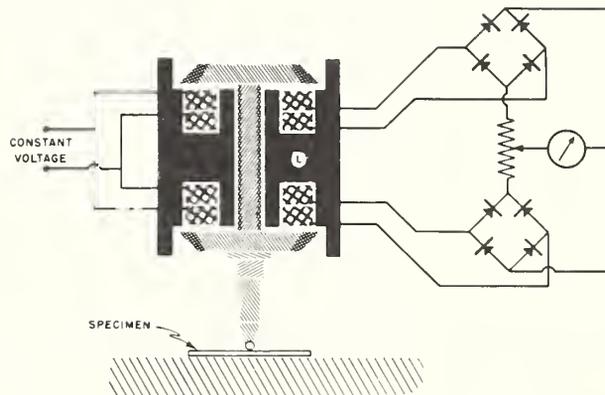


FIGURE 60. Measuring head and circuit of the "Metron" comparator gage (double airgap reactance ratio system.)

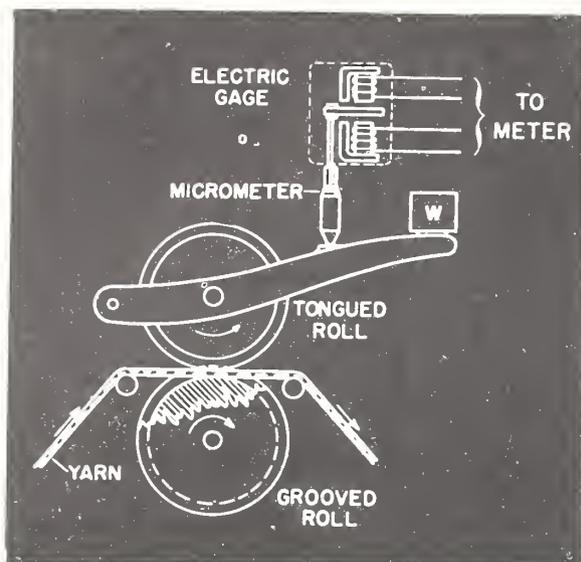


FIGURE 61. Measuring system of the "Pacific" evenness tester for yarn.

of this type has full scale with 20-mil displacement, 1 division equal to 1 mil, and a magnification of 100.

Manufacturer: Metron Instrument Co., Denver, Colo.

1.2424. "PACIFIC" EVENNESS TESTER [125, 136] (fig. 61). This instrument has been developed by Pacific Mills, Lawrence, Mass., and the General Electric Co., to measure the thickness of yarn and sliver in the textile industry. The twisted material, with uneven surface, is led through a guide with tension controls into the nip of a pair of tongued and grooved rolls, at a speed of 12 yards per minute. The groove is square, so that the material is compressed into a rectangular shape. The bottom roll establishes three fixed sides. Variations of the thickness cause the top roll, whose force is constant, to move up and down. This, in turn, changes the position of the middle core of a GE double-airgap-ratio meter (fig. 61). The output of this electric micrometer may drive the pen of a recording instrument, or may be used for analysis devices. For testing yarn the rolls have three sets of grooves, 6, 9, and 12 mils wide. The rolls for testing sliver have groove widths of 3, 125, and 312 mils. One head for extra heavy sliver has a groove 625 mils wide.

In the strict sense, this instrument is not a thickness meter, but a volume meter; for given density of the compressed sliver, it measures the weight of the material per unit length.

Manufacturer: Anderson Machine Shop, Inc., Needham Heights, Mass.

1.243. *Differential Transformers* [76, 138, 170, 178, 191, 195] (fig. 62). The arrangement of an assembly of one fixed-primary coil and two fixed-secondary coils with a moving iron core is being used more and more to measure

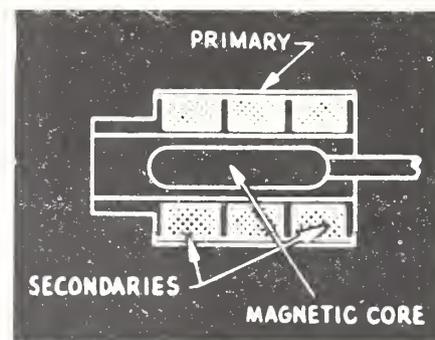


FIGURE 62. Differential transformer system to measure small displacements.

displacements. Figure 62 shows one of the many possible arrangements of the coils. With proper dimensions and proper spacing of the coils the voltage unbalance is, with high accuracy, proportional to the displacement of the moving core. The source of power may be a 60-cps supply line or it may be an oscillator operating at frequencies up to 100,000 cps. Operation at 60 cps makes it possible to avoid the necessity for amplification of the output signal. It is only necessary to rectify the output to operate a sensitive moving-coil indicator. Highest sensitivity is possible with the higher frequencies and amplified output. For portable thickness meters to be used where no line supply is available, battery-operated systems have been developed.

1.2431. "ATCOTRAN" AND SCHAEVITZ DESIGNS [138, 178] (figs. 63, 64, 65). These devices are used in many designs of displacement meters. A Schaevitz design gives, with an input of 2,500 mv and 21,000 cps, an output of 25 mv for a core displacement of 1.6 mils, with a linear characteristic. Standard designs of differential transformers of the "Atcotran" type have a full range of ± 10 mils. Without step-up transformer, the output is given as 0.25 mv per input volt per mil armature displacement. With a 22:1 step-up transformer the output is 5.2 mv per input volt per mil displacement. Deflection systems with indicating instruments are dependent on fluctuations of line voltage and frequency, at all points other than the balance point. With bridge circuits, it is possible to be independent of voltage fluctuations over the full range, but the output varies widely with the frequency. However, it is possible to select an operating frequency that allows wide frequency variations. The accuracy of the "Atcotran" thickness meters is given as ± 0.25 percent for the total system, including reasonable variations of temperature, voltage, and humidity.

Manufacturer: Automatic Temperature Control Co., Philadelphia, Pa.

A system that automatically balances and records is shown in figure 63. In this circuit, the primaries of two transformers, one in the

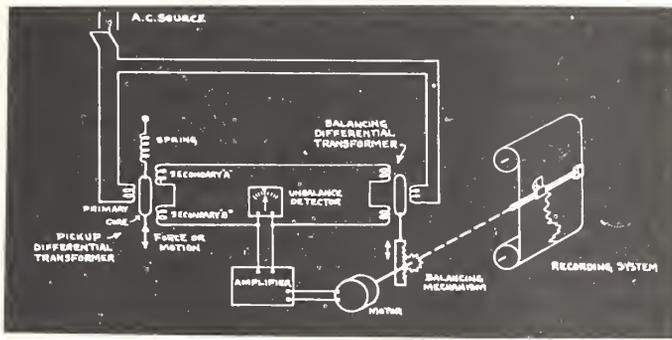


FIGURE 63. Automatic balancing and recording thickness meter with differential transformer.

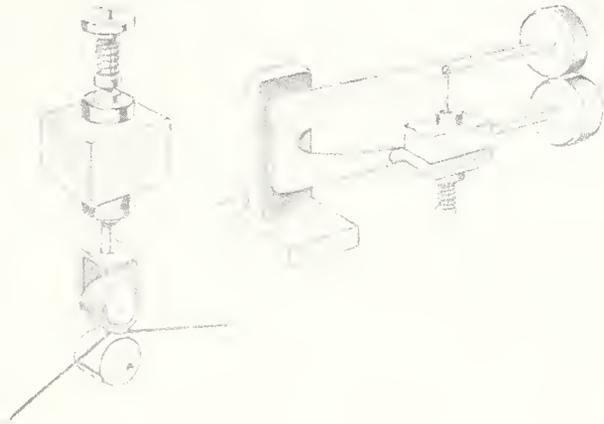


FIGURE 64. Application of differential transformer systems to measure thickness.

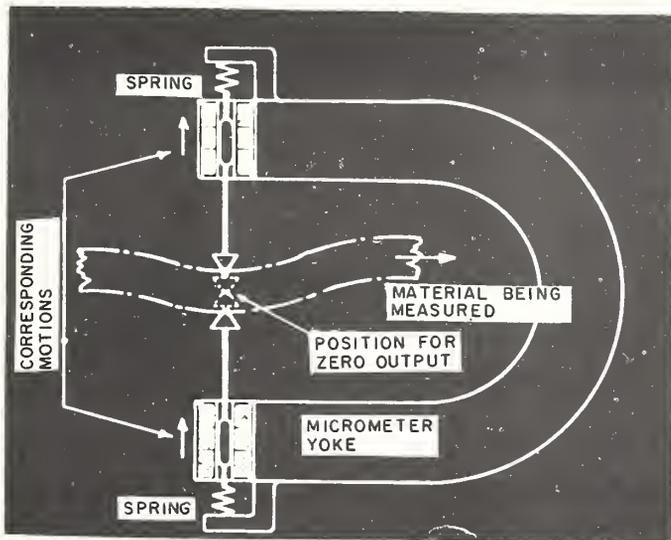


FIGURE 65. Micrometer with floating anvils and differential transformer.

pickup device, the other in the indicator or recorder, are connected in series to the source of voltage. The secondaries are also connected in series, but opposing. When the position of the core in the indicator is the same as the core in the pickup, the voltage fed to the detector is zero. If the pickup core is moved, voltage is fed

to the detector and the indicator shows a need for movement of the balance core to achieve correspondence between the two cores. This re-balancing motion is accomplished automatically, and a recording pen is positioned to indicate the thickness on a chart.

Manufacturer: Schaevitz Engineering Co., Camden, N. J.

There are many arrangements of the coils and the circuits used in differential transformers. Figure 64 shows the use of differential transformers to measure the thickness of wire or ribbon. A micrometer with floating anvils is shown in figure 65. The two transformers are connected so that no voltage output is present when the two anvils are together, even though they are in motion. However, should their position differ due to material between the anvils, the output will be a measure of the material thickness. Slight movements of the material in the vertical direction will not affect the accuracy.

"Dyna-Myke" Micrometers. These instruments, developed to measure the thickness of moving sheets, use a pickup with a differential transformer of the Schaevitz type 0100LC and replace an older device described in [96]. The Dyna-Myke is operated at 8,000 cps and the indicator has a plus-minus scale 4.5 in. long. If the material to be measured is flexible enough to be held in contact with a base which acts as a reference point, thickness can be measured with a single gage head. For more rigid materials, such as glass, sheet metals, or solid plastics, two gage heads are required. These are mounted on either side of the material and electrically connected in such a manner that any lateral motion of the material is without influence, the gages indicating only the relative distance between the two points of contact. The Dyna-Myke, used for indicating and recording, may have the following five ranges: ± 100 mils, 22.5 amplification; ± 10 mils, 225 amplification; ± 1 mil, 2,250 amplification; ± 0.1 mil, 22,500 amplification; ± 0.01 mil, 225,000 amplification. With the maximum sensitivity, one scale division of 112 mils corresponds to a thickness difference of $0.02 \mu\text{in}$. The over-all accuracy is given as ± 2 percent of the scale range.

A similar device with the same pickup, the "Myke-A-Trol," is used for control in ranges from 0.1 to 100 mils.

Manufacturer: Industrial Electronics, Inc., Detroit, Mich.

1.2432. STEVENS-ARNOLD PORTABLE INSTRUMENT. (fig. 66). For portable instruments, to be used in places where ac is not available, a battery and a dc-ac converter may be used to feed the coils. A device of this type is shown in figure 66. The plunger moving the iron core has a travel of ± 10 mils or 0 to 20 mils. The dc indicator has a scale length of 4 in., giving a



FIGURE 66. Transmitter for electric micrometer.

magnification of 200. The accuracy is given as 0.25 mil. The pressure on the plunger can be adjusted between 3.5 and 10 oz.

Manufacturer: Stevens-Arnold, Inc., South Boston, Mass.

1.2433. "LYN-A-SYN" TRANSDUCER. A recently announced transducer for sensing small motions is available in 32 models, with linear displacement ranges from 3 mils to 2,000 mils, under the trade name "Lyn-A-Syn." Operation is based on the linear change in coupling between the primary coil and secondary coils as a result of the displacement of the high permeability metal core. The smallest unit for the 3-mil range has an outer diameter of $\frac{1}{4}$ in. Total length of this unit is also about $\frac{1}{4}$ in.

Manufacturer: Minatron Corporation, Belle Mead, N. J.

1.244. "Metrisite" Transducer. The "Metrisite" is a position-sensitive transducer capable of measuring very small displacements. The basic structure of this device consists of three coils on the three legs of a laminated magnet. In the center leg of the three there is an airgap containing a loop of conducting material around the yoke. The loop is free to move along the base and mechanically connected with the feeler to indicate displacements.

The coil on the center core is connected with a constant 60-cps a-c voltage. With the short circuit loop exactly in the middle of the center leg the magnetic flux spreads in equal parts over the outer legs, which carry two coils so connected that the signal output is zero. If the loop is displaced, the balance of the two flux halves is disturbed. The side to which the loop moves has a higher magnetic resistance and the resulting output from the secondary coils is, within certain limits, in proportion to the dis-

placements of the loop. The output signal is in phase with the input on one side of zero and 180° out of phase on the other.

The total travel of the loop is 860 mils. For ± 200 mils the output is linear within 0.1 percent of the maximum, which is obtained with 300 mils movement.

The amplifier of standard instruments provides four ranges giving full scale on the indicator for 0.1, 1, 10, and 100 mils movement. Without amplifier the full range is obtained with 5 mils. With a special amplifier, full-scale response has been obtained with armature movements of only 0.001 mil ($1 \mu\text{in.}$) and the sensitivity of such an instrument (not accuracy) corresponds to $0.1 \mu\text{in.}$

Manufacturer: Graydon Smith Products Corp., Boston 14, Mass.

1.25. Displacement With Capacitive Pickup.

1.251. *Change of Capacitance* [5, 17, 61, 66, 70, 91, 96, 103, 110, 124, 133, 134, 135, 141, 149, 151, 154, 155, 169, 183, 191, 215]. The capacitance of an air capacitor consisting of two parallel plates changes inversely as the distance of separation of the plates following a hyperbolic law. Reducing the distance to one-half gives twice the capacitance. The dimensions for such capacitors are rather limited. The useful diameter is no more than 3 to 4 in., the initial distance not less than 10 to 20 mils, which gives an initial capacitance of about 100 to 200 $\mu\text{mf.}$ To keep the change of capacitance fairly linear with the change of distance, the change of distance can only be from 3 to 10 percent of the initial value, which gives deflection ranges from 0.3 to 2 mils and capacitance changes in the order of 2 to 20 $\mu\text{mf.}$ which have to be measured with a precision of better than 1 percent. None of these devices can be operated at line frequency, because the required insulation cannot be attained. They are all operated with frequencies from 1,000 to 100,000 cps.

Ultramicrometer. The "Ultramicrometer" is described in [215].

Dowling Micrometer. The Dowling micrometer [70, 154] obtained a maximum stable sensitivity of 1-mm galvanometer deflection per 20- $\mu\text{in.}$ displacement, or a magnification of 2,000.

Gerdien Micrometer. The Gerdien capacitive micrometer [103, 124, 149], used in connection with electromagnetic oscillographs to measure very high pressures by the deflection of a capacitor plate, gave magnifications of 10^7 and even 10^6 . A displacement of $1 \mu\text{in.}$ was represented by a deflection of 1 in. on the chart of the oscillograph. The accuracy of the best mechanical designs of capacitive micrometers is limited to a distance of about 0.2μ , or $8 \mu\text{in.}$

Manubel. A capacitive micrometer, the "Manubel" comparator [91], is manufactured in France and Switzerland. The fixed capacitor plate consists of a ground and metallized

steatite disk. The movable anvil is in the shape of a piston, whose upper face forms the second plate of the capacitor. The change of capacitance is measured by change of frequency, as with most capacitive micrometers. The indicating instrument, with a scale length of about 50 mm, has two ranges, ± 5 and $\pm 10 \mu$ (± 0.2 and ± 0.4 mil). This corresponds to a magnification of 10,000 for the lower range. The accuracy is given as 0.2μ (0.008 mil).

Manufacturer: Etablissements Edouard Berlin, Paris; also Manufacture de Machines du Haut-Rhin, Mulhouse.

Cornelius Co. Meter. Another mechanical-electrical capacitive meter [17] uses a quartz crystal as a frequency standard. The full-scale deflection of 100 mm on the indicator can be reached with a displacement of only 1μ , corresponding to an amplification of 100,000. The instrument is described as highly stable with constant temperature and full-scale deflection. The indicator deviation is less than 0.02μ or 2 percent of full scale.

Manufacturer: Cornelius Electronic Instrument Co., Ltd., England.

1.252. *Change of Capacitance Ratio* [135, 149, 185] (fig. 67). This method (fig. 67) resembles the double-airgap inductance ratio meter used, for instance, in the Electrolimit gage. The use of a single capacitor has, as already mentioned, the disadvantage that the capacitance change follows a hyperbolic law and is proportional to the thickness for only a small percentage of the initial value. Measuring the ratio of two capacitances, formed by two electrodes and one moving member, gives linearity over a wide range of displacement of the movable electrode. Here, too, the low capacitances

make it necessary to use higher frequencies. Magnifications up to 1 million are possible. The limit of sensitivity is given as 0.0025μ (0.1μ in).

There are other ways of obtaining a linear change of capacitance with the displacement of an electrode [149], such as the axial displacement of one cylinder into another. A thickness gage of this kind is described in British Patent 629,648 (G. Jacot and J. Monti), the scale reading in fractions of a micron.

Recently a much simpler thermal device has been developed, moving a vane in vertical position between two heated nickel helixes which are part of a Wheatstone bridge. No air current is used. Displacement of the vane causes heat-radiation losses of the helixes to change and a differential voltage is generated in the diagonal of the bridge. The sensitivity, measured in vane displacement and millivolt output, is about the same as with the previously described bolometer. With 0.1-mm displacement, 20 mv are generated in the diagonal.

Manufacturer: Ferngteuergeraete OHG, Berlin-Zehlendorf.

1.26. *Displacement With Thermal Converter* [160, 191] (fig. 68). A schematic diagram for an electric-thermal device for measuring thickness ("Bolometer gage") is shown in figure 68. The feeler, a, is positioned without friction by the reed springs, b. The movements of the feeler are transmitted by the spring, c, to the moving coil, d, swinging in the field of the magnet, h, part of a bolometric amplifier system. This consists of four heated nickel helixes. The a-c magnet, l, vibrates a reed, m, to produce a flow of air, going through the slots n past the two edges

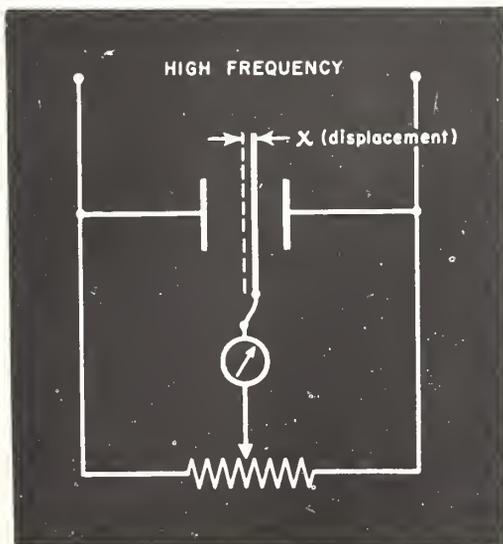


FIGURE 67. Displacement measuring system, using the principle of change of ratio of the capacitance of a double airgap capacitor.

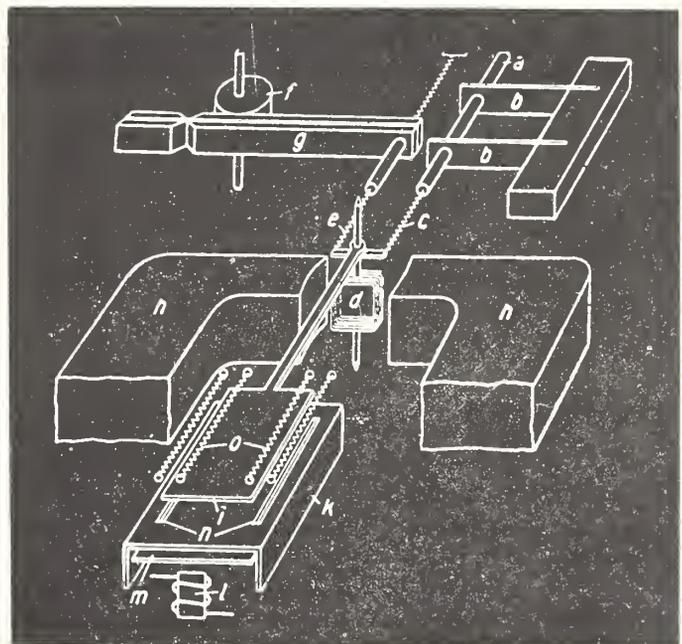


FIGURE 68. Electric bolometer system to measure small displacements.

of the vane, *i*, moved by the galvanometer coil. In the center position, the air flow on both sides of the vane is the same and the bridge with the four heated arms is balanced. When the feeler is moved, the flow of air strikes one pair of helixes more than the other and a current is generated in the diagonal of the bridge in proportion to the displacement. This unbalance controls current to coil *d* to maintain the position of vane *i* in the neutral or zero position, while the current necessary to do so is a measure of the thickness. The spring *e* balances the spring *c*, while *f* and *g* are used for the zero adjustment of the gage. With the range of 50 to 150 μ (2 or 6 mils) the output of the bridge is about 100 mw, sufficient to operate control elements or ink recorders. The accuracy is given as $\pm 1 \mu$ ($\frac{1}{25}$ mil). With a 120-mm scale width on a recorder chart, the magnification is $120,000/50=2,400$.

Manufacturer: Siemens & Halske, Berlin.

1.27. Displacement With Electronic Converter [108] (fig. 69). This micrometer employs a species of vacuum tube whose electrical constants are changed by a mechanical displacement applied to the end of a short rod. Figure 69 shows the tube in its simplest form. A hot filament provides a stream of electrons which flows to insulated plates 3 and 5. These plates are carried on a short rod, 13, with motion permitted through the deformation of the vacuum-tight diaphragm 25. In this manner the resistance of one plate-filament circuit is increased while the other is correspondingly decreased. Because of the instability of emission in vacuum tubes the two plate-filament circuits are incorporated in a bridge arrangement. Resistances 17 and 19 are nearly equal when the filament is centered between the plates. Thus, the reading of microammeter 31 normally measures the mechanical departure of the plate system from its midpoint and is only slightly dependent upon irregular emission or plate potential.

The sensitivity is given as approximately 6 ma/ μ (0.04 mil) deflection. Using a conventional portable microammeter, with a deflection of 4,000 mils per 5 ma, a magnification factor of 120,000 is obtained. The torque necessary to deflect the rod 1 μ is about 1 gram-centimeter.

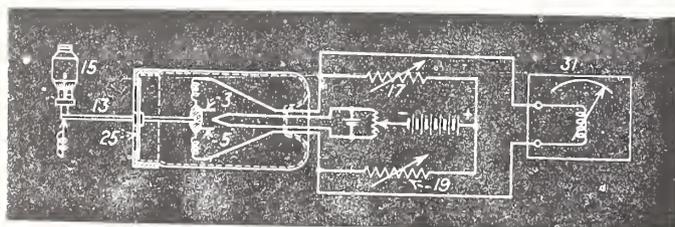


FIGURE 69. Principle of a mechanical-electronic micrometer.

1.28. Displacement With Photoelectric Pickup [73, 126, 181, 204, 207] (figs. 70, 71).

Principle: A beam of light (fig. 70) passes through a condenser lens, a slot, *c*, and a second lens system, to a photoelectric cell. Between the cell and the light source a knife edge, *b*, cuts off a part of the light beam going to the cell. This knife edge is moved by a mechanical feeler, *a*, in proportion to the thickness of a sheet or the diameter of a wire, thus changing the light quantity falling on the photocell. The current of the photocell varies in proportion to the movement of the knife edge, in other words with the thickness of the specimen. In this arrangement the indications are dependent on variation of lamp voltage as well as of photocell sensitivity, and frequent recalibration is necessary.

Figure 71 shows a more satisfactory device. Light source LS emits either two beams or one that is split. One is directed through the condenser lens system, CL, to the photocell, P_1 , just as in figure 70. The other beam is sent over the mirror, *M*, and the grey wedge, *W*, to a second photocell, P_2 , identical with P_1 . The difference of illumination of the photocells is in proportion to the movement of the knife edge of the micrometer head, which measures the sample of unknown thickness. The zero of the scale is adjusted by placing a standard thickness under the knife edge and adjusting the comparison beam by moving the grey wedge *W*. The output of the two photocells is amplified in a conventional way and the moving-coil indicator may give either thickness or deviation of thickness from a standard value (see also section 4.53 and fig. 123).

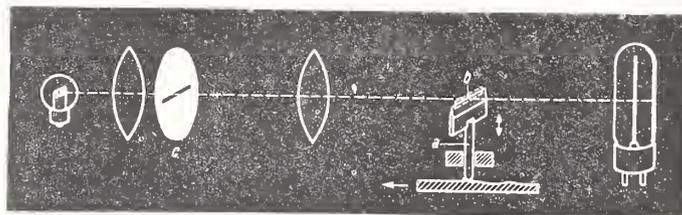


FIGURE 70. Mechanical-optical method of thickness measurement.

a, Feeler; b, knife edge; c, slot.

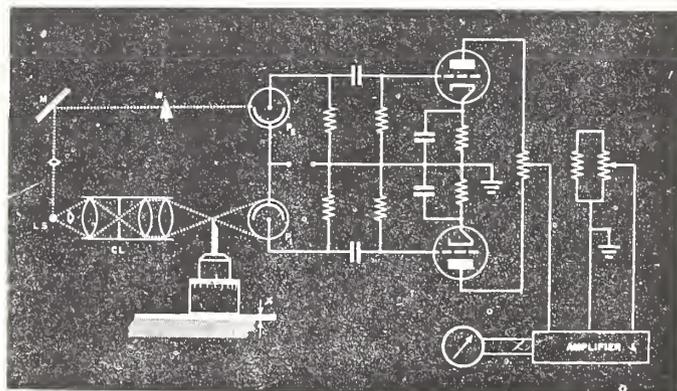


FIGURE 71. Thickness measurement with two photocells in differential arrangement.

2.00. Chemical Methods

2.1. Stripping and Weighing [16, 30, 36, 145].

Destructive stripping is often used to determine the thickness of the metallic coating of sheets or wires. A measured and weighed area of coated metal sheet (or length of wire) is put in a solution that dissolves the coating. The average thickness is calculated from the loss of weight. By dissolution of the base metal, the weight of the coating may be obtained directly. For measuring the thickness of the tin coating on sheet iron, specimens 4 by 8 in. are used and the iron is dissolved in hydrochloric acid.

The stripping method is also applied for the determination of the thickness of gold coatings on fine wires of silver, tungsten (wolfram), or molybdenum 3 to 10 mils in diameter. The base metal is dissolved, and the remaining metallic gold is weighed.

See ASTM Standard A90, Weight of coating on zinc-coated (galvanized) iron and steel articles.

2.2. Optical Projection With Stripping [80] (figs. 72, 73a, b). *Coating thickness of corrosion-protected iron screw threads:* The highly magnified contour of the finished screw is projected by means of an optical contour projector on photographic paper through a graphscreen which provides reference lines along with the contour of the thread. Next, the coating is chemically removed by the stripping solution, which is applied to the thread with a dropper, without disturbing its position. After this, the screw is carefully washed with distilled water and dried by a stream of hot air. A second exposure is made on the same photographic paper. The resulting picture shows the dissolved coating as a halftone, clearly demarcated from the base metal.

2.3. Spectrophotometry [54]. Applicable to metallic gold-plated sheet with a total thickness of 30 mils or less, this scheme uses a disk of 1 mm², cut out with a punch and treated with diluted nitric acid, which dissolves the base metal and leaves a small piece of gold. This is washed in water and then dissolved in a few drops of aqua regia, after which it is evaporated to dryness by impinging a jet of purified air on the surface of the solution. The quantity of gold is determined with a spectrophotometer from the intensity of yellow color produced when a measured volume of a solution of *o*-toluidine (3-3'-dimethylbenzidine), dissolved in normal sulfuric acid, is added to the air-dried residue. Quantities of gold up to 10 μg, corresponding to 0.5 μ (0.02 mil), of gold on 1 mm² of surface can be determined accurately.

2.4. Spectrochemical Analysis [145, 156]. To measure thickness of a metal coating by spectrochemical analysis, the coating is sparked. The actual thickness is determined in one of

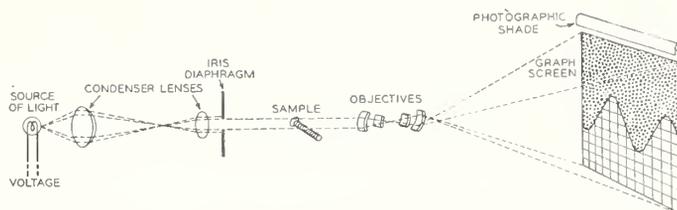


FIGURE 72. Optical contour projector, used to determine thickness of coating on screw threads.

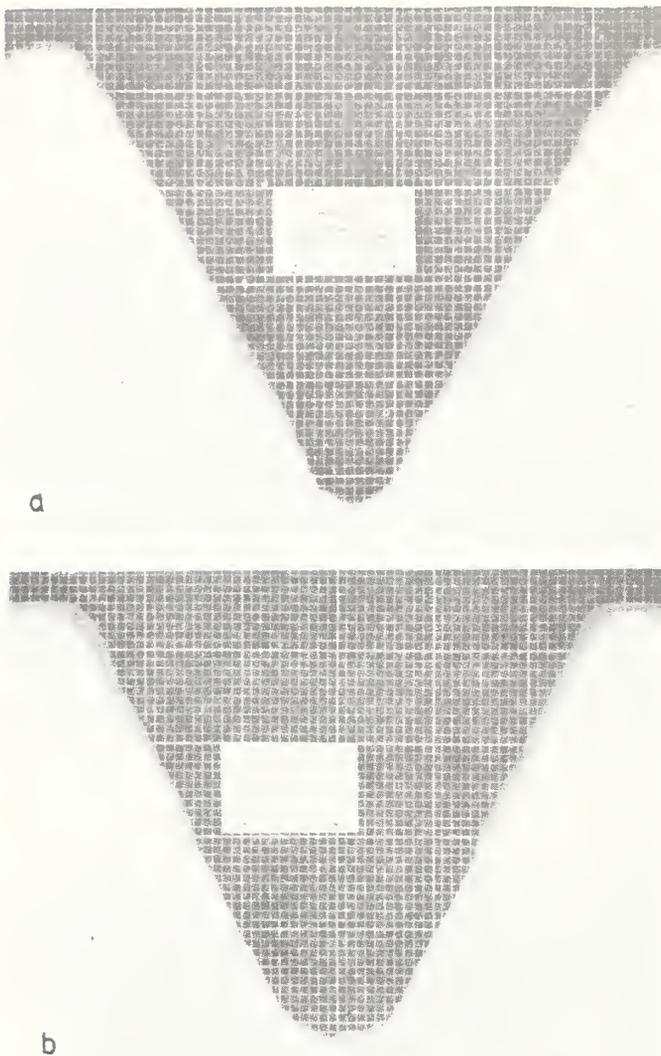


FIGURE 73. Shadowgraphs of plated screws.
a. A satisfactorily plated screw; b, an unsatisfactory plate—the deposit is almost entirely on the outside of the thread

two ways. The intensities of specific lines in the spark spectrum are compared with those obtained with standard specimens, or the time required for the spark to reach a given intensity is measured. This principle is not useful for metals with low melting points, such as tin. It is applicable, however, not only to sheet metals, but also to pipes, rods, and other such shapes.

2.5. Color Change [16]. The coating, generally of a different color than the base material,

is destroyed by the application of a chemical solvent, usually an acid, in a standardized intensity of application. The time needed to see a change of color is an indication of the thickness of the coating.

2.51. Spot Test (ASTM A219) [18] (fig. 74). This is used for chromium coatings 0.03 to 0.05 mil thick. One drop of hydrochloric acid of 1.180 specific gravity at 60°F is placed with a dropper within a wax ring 250 mils in diameter on the cleaned chromium surface. The time required from the beginning of gas evolution until the first appearance of nickel is measured to ± 0.5 second with a stop watch.

The temperature must be carefully observed. A change of $\pm 1^\circ\text{C}$ at 20°C changes the time (and apparent thickness) ± 3.7 percent (fig. 74).

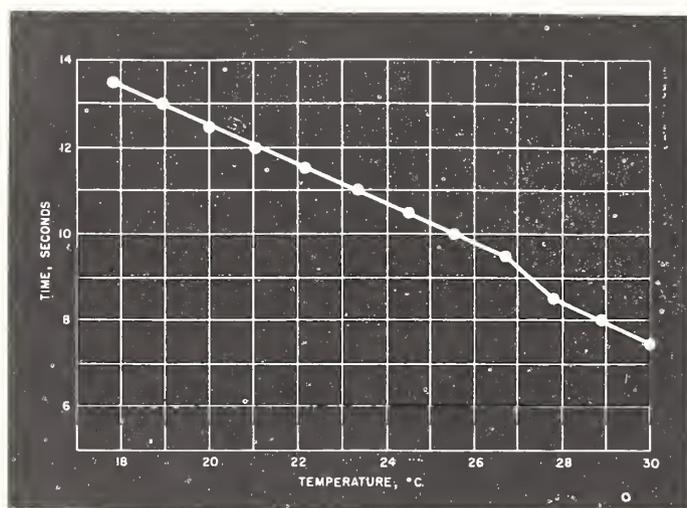


FIGURE 74. Time to dissolve a chromium coating 0.01 mil thick as a function of temperature. From ASTM Standard A219.

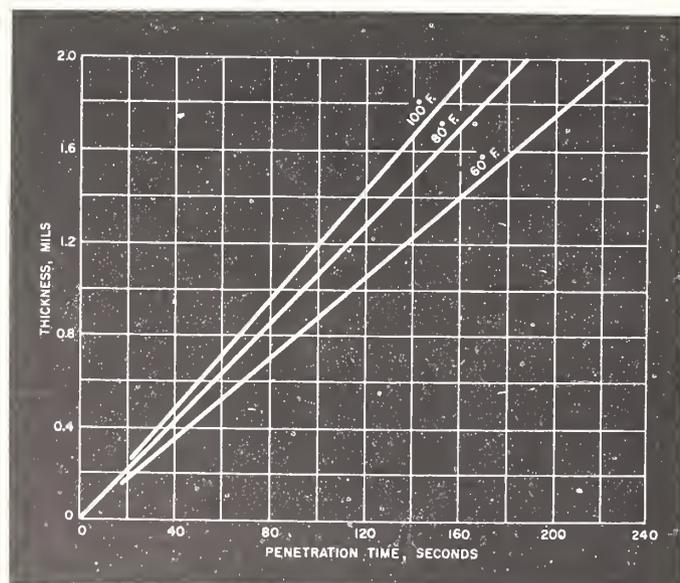


FIGURE 75. Thickness of zinc platings as a function of penetration time for three temperatures (dropping test).

2.52. Dropping Test [41, 46] (fig. 75). To determine thickness of coatings of lead, zinc, or cadmium on steel, plane or curved, the corrosive test solution is dropped from a funnel at a rate of 100 ± 5 drops per minute onto the specimen, which is held at a 45° angle about 0.5 in. below the tip. The time for the solution to penetrate through the coating is measured.

The test solutions for lead on steel are 3.5 percent by volume of glacial acetic acid, or 3.5 percent of 30-percent hydrogen peroxide; for zinc and cadmium on steel, chromic acid, 200 g/liter, or sulfuric acid, 50 g/liter. Figure 75 shows the thickness of zinc plating as determined with the standard corrosive solution at temperatures of 60, 80, and 100°F. The accuracy of the method is 5 to 10 percent, the temperature influence 1.5 to 2 percent per degree C.

2.53. BNF Jet Test [60]. The BNF (British Non-Ferrous Metals Research Association) Jet Test is similar in principle to the spot or dropping test, the difference being that here the corrosive liquid is delivered to the test surface in a stream or jet. The increased speed with which the liquid is delivered results in a corresponding decrease in testing time. The method is applicable for either plane or curved surfaces. The apparatus is easily portable and the accuracy is about ± 15 percent.

2.6. Electric Potential [117]. The following is a procedure for measuring the thickness of inhibiting films on glass electrode surfaces. The glass electrode of a pH meter is an extremely sensitive indicator of the presence of inhibiting films, thinner than 0.06μ , on the outer surface of the electrode.

The pH response (millivolts per pH) of glass electrodes is changed considerably by films of either conductive materials like silver, or non-conductive like petrolatum. It is possible to detect films with a thickness of only 0.003μ (30 A) by the voltage deviation from standard values.

3.00. Electrical Methods

3.1. Insulation Breakdown. The breakdown voltage of thin insulating coatings on metal is approximately proportional to the thickness. This method is generally applied to determine the thickness of electrochemically deposited oxide on pure aluminum.

3.11. Aluminum Oxide Testers [11, 19, 122] (fig. 76). *Bell Laboratory Testers.* The Bell Telephone Laboratories, Inc. [11], have used a chromium-plated steel sphere electrode of about $\frac{1}{8}$ -in. diameter under a spring force of 2 to 4 lb. The 60-cps voltage is increased in steps of 25 volts up to breakdown voltage, which is generally less than 1,500 volts. The thickness is proportional to the breakdown voltage within

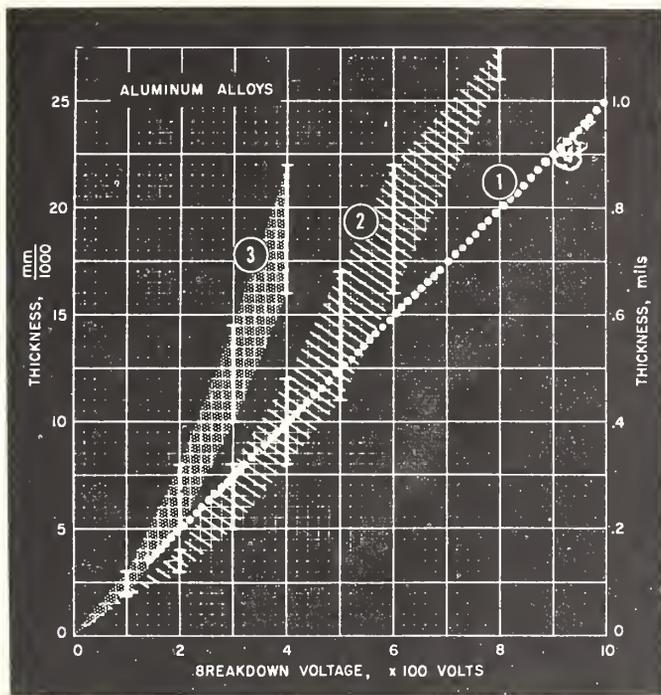


FIGURE 76. Thickness of aluminum oxide coatings as a function of breakdown voltage.

1, Tests of the Bell Laboratories, 1942; 2, tests by Herenguel and Segond, without Cu Si; 3, tests by Herenguel and Segond, with Cu and Si.

about ± 10 percent (about 1 mil for 1,000 volts) (fig. 76).

French Method. In a French study of this method [122], not only aluminum but aluminum alloys with copper, silicon, magnesium, and other metals were tested. These tests gave a lower breakdown voltage for alloys with copper and silicon than for pure aluminum and alloys with other metals, as shown in figure 76. The breakdown voltages for a given thickness are lower than the values given by the Bell Laboratories.

GE Tester. A commercial tester for anodized aluminum is manufactured by the General Electric Co. It is not called a thickness meter, and is used only to determine the dielectric strength (breakdown voltage) of anodized coatings. The instrument has three voltage ranges for the different types of coatings. The lowest range, with 0 to 20 volts 60 cps ac, is for chromic-acid-type coatings; the other ranges, 0 to 100 and 0 to 600 volts, are for sulphuric-acid-type coatings. The test voltage is gradually increased from zero until a drop in the voltmeter reading indicates breakdown. The maximum voltage is a measure of the quality of the coating as insulation (see GE Bul. 894, Anodized aluminum testers).

ASTM "Standard Method of Test." The "Standard Method of Test for Dielectric Strength of Anodically Coated Aluminum" (ASTM B110) gives only the test procedure.

Permissible breakdown values are left to agreement between purchaser and seller. If it is sufficient to test only the continuity of the oxide film, a solution of 2-percent crystalline copper sulfate and 2-percent hydrochloric acid of 22 deg Baume is spread over the coating. It leaves black copper deposits on any areas that are not covered by the oxide film. Size and number of such spots after 5-min reaction give the degree of continuity. Testing of rubber gloves by electrical breakdown is described in ASTM D120 (Book of Standards, part 6, p. 34-41, 1952).

3.12. Enamelled Wire [109, 150] (fig. 77). A method of counting electric breakdowns is used for production testing of film-insulated magnet wire to indicate thickness uniformity (fig. 77). Immediately after the last coat of enamel is applied, the wire passes through a mercury bath. A d-c voltage of approximately 50 volts is maintained between the mercury and the wire. The electrodes are a pair of 1-in.-diameter metal disks, bolted together with a semicircular 10-mil spacer between them. The central portion of each disk is hollowed somewhat to provide a reservoir for the mercury. Six of these elements are joined in a gang electrode, and two such gang electrodes are connected to a machine with 16 heads of wire.

The test voltage can be set to a minimum of 48 volts dc for singlecoated wire, up to 270 volts for multicoated wire. An electronic relay will trip when a fault of 0.2 megohms or less appears for the lowest setting, or 10 megohms for the highest setting. Whenever a fault occurs, a signal lamp flashes and the corresponding pen on a multipen recorder with 20 pens for 20 machines is deflected. A certain number of breaks per minute is allowed, depending on the type of wire.

This method does not provide a direct thickness measurement, but is a means of counting the spots with less than minimum thickness along the wire.

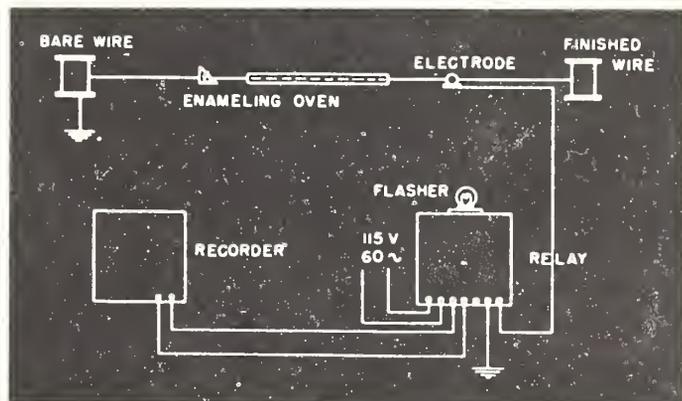


FIGURE 77. Layout of equipment for fine wire coating thickness-continuity test.

3.2. Resistance. The principle of all electric resistive methods is to pass an electric current, generally dc, through a known conducting length. Measurement of the resistance then permits calculation of the average cross section.

3.21. Resistance Method for Wires and Ribbons [63] (figs. 78, 79). For some applications

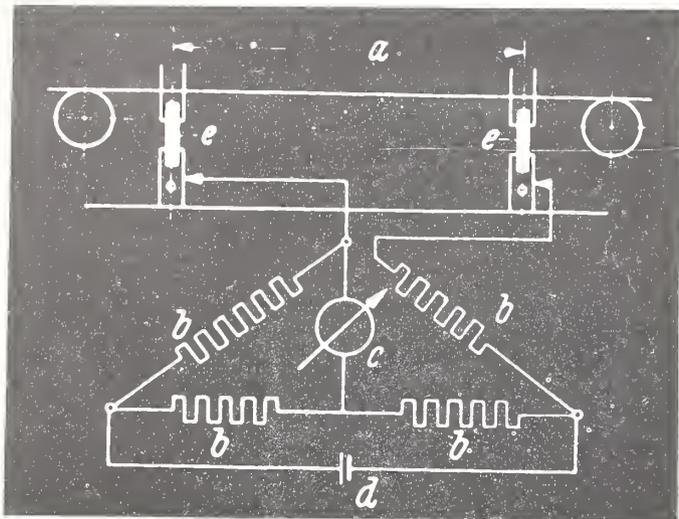


FIGURE 78. Testing of wire diameter by the electric-resistance method.

a, Wire length under test; b, bridge resistors; c, galvanometer; d, battery; e, mercury contacts.



FIGURE 79. Mercury contact for figure 78.

a, Glass tube; b, mercury; c, iron (or molybdenum) rod; d, brass sleeve; e, positioning screw.

the mechanical dimensions of a product must be maintained within very narrow limits, e.g., in suspension wires and ribbons for galvanometers. If the resistivity is known, the average cross-sectional area is given by the electrical resistance. In other cases, as in filaments for lamps, the electrical resistance per unit of length must be held within narrow limits, the actual thickness or diameter being of only secondary importance and not necessarily uniform. Generally, the average thickness over the length to be used in the finished product is desired. There, resistance should not be measured over a shorter length. Average thickness is often desirable in nonelectric objects, and again electrical resistance measurements can often be used to advantage.

One company has used the method shown in figure 78, with the electrode of figure 79. A glass tube, a, is placed on top of a rod of iron or molybdenum, c, to which the current connections are made. The glass tube has two diametrically opposed holes, b, of 0.5-mm diameter, through which the wire is pulled. The tube is filled with mercury. For lamp-filament wire a current of about 2 ma is sent through the wire, the measurement being made with a Wheatstone bridge. The distance between the electrodes is from 12 to 40 in., the wire diameter between 0.4 and 4 mils. The wire travels between 10 and 17 ft/min. The unbalance current is recorded with an ink recorder by means of a thermal amplifier; length of 1,000 ft. of wire is recorded on 1 ft of chart length. The chart range is ± 2.5 percent resistance change from normal, and changes of 0.1 percent in resistance are represented by a deflection of 10 mils. This corresponds to a thickness variation of 0.01μ for a wire with $1\text{-}\mu$ (0.04-mil) diameter.

3.22. Resistance Method for Metal Sheets From One Side [200, 211]. If a direct current from two distant points is passed through a sheet, and the voltage drop measured at two points in the path of the current, this voltage drop is an indication of the thickness of the sheet. Four point electrodes are used, two for current, two for voltage, preferably all in a straight line. For steel plates 1 in. thick, with potential points 6 in. apart, the resistance is about 8 microohms, and the potential drop with 10 amp is 80 microvolts. Up to about one-half in., the conductivity is about proportional to the thickness, but for thicker plates the conductivity increases more slowly. One type of low-range ohmmeter gives the resistance independent of current variations, with a linear scale of 0 to 100 microohms. It is recommended that the relation of resistance to thickness be determined by making measurements on sheets with known thickness of the same material. The accuracy of this method is given as ± 5 percent.

If the specific resistivity of the material is not known and if it is not possible to calibrate the instrument with objects of known thickness, another method can be used, arranging the four electrodes not in one line but in a square. Two sets of such electrodes have to be used for each measurement, both spaced at distances larger than the expected thickness. From the ratio of the two readings the thickness of the plate can be computed, independent of permeability and conductivity of the material under test. For all measurements, two readings with reversed polarity have to be made to exclude errors from parasitic thermoelectric currents.

An instrument of this type, the Electroflux thickness gage, was manufactured by the Scott Electroflux Co., which is no longer in business. The thickness range for this instrument was given as 0.125 to 2.25 in., the accuracy as 3 percent of the actual thickness. The instrument was battery operated.

3.23. Resistance Method for Intricate Castings [201] (fig. 80). This method is used as a high-speed test procedure in production. For intricate castings, current is applied to two fixed points of the casting. The potential drops are measured between contacts which are moved over the surface independently of the fixed current contacts. The movable contact points are about $1\frac{1}{16}$ in. apart, and the maximum current for this test is 300 amp. The instrument must be calibrated with a perfect test piece as a reference. It is essential that the electrodes for one type of casting always be placed in the same spot. To do this, templates of plastic material are used with holes for the different test positions (fig. 80). To avoid the use of a battery with high discharge current, the test can also be made with 60-cps ac. Anomalies are detected by the unbalanced emf in two opposed pickup coils connected to a dry-disk rectifier system.

Manufacturer: Record Electrical Co., Ltd., England.

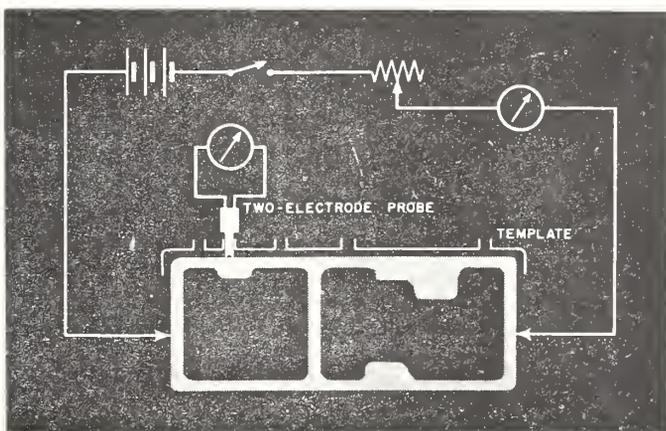


FIGURE 80. Measuring the thickness of intricate castings by the voltage-drop method.

3.24. Resistance Method for Silver Plating [9, 65]. A waveguide plating quantity indicator based on electrical conductance has been developed to measure the thickness of silver plate on the inside of a stainless-steel waveguide from the outside. A known amount of direct current is passed through a portion of the waveguide wall and the electric potential at two other points is measured. The potential is a function of thickness alone if the current is laminar throughout the material and if the linear dimensions of the material are several times greater than the probe spacing.

3.3. Heating (pat. 8). If an electric current is sent through a conductive sheet or tube, ferrous or nonferrous, in such intensity that the conductor heats up to 100 to 200° C, and the surface is coated with a substance having a melting point just above the temperature reached by a tube with normal thickness, thin spots or flaws that increase the resistance will show up because the substance will melt at such points. Painting the objects under test with a color-changing coating will give even better results.

3.4. Electrochemical Method.

3.41. Coulomb-Counting. In electroplating, the weight of the deposited material is proportional to the ampere-seconds (coulombs) used in the process. For a given test area on the plated object, the thickness of the coating is proportional to time, when the current is constant. This process can be reversed, taking the coating off by electrolysis.

3.42. Potential Change [8, 210]. A small area of a metallic coating, approximately $\frac{1}{8}$ in. in diameter, inside a small rubber gasket, is anodically deplated. This tester is a miniature reverse-current plating cell in which the article to be tested is the anode and the cell itself is the cathode.

At the start of the test and until the base metal is exposed, a voltage characteristic of the plating exists across the cell. When all of the plating has been removed from the test spot, and the liquid touches the base, this voltage changes sharply and assumes a new value which is now characteristic for the base metal. This sudden voltage change is the "end point" of the test and is amplified to operate a relay which turns off the instrument and the flow of liquid.

The time required to dissolve the plating on the test spot is proportional to the thickness of the coating. By correlating the area of the test spot with the current used to strip the plating, the counter can be made to read directly in units of thickness.

The solution, which is in contact with the coating, does not attack the plating chemically, but serves only to carry the current and to receive the dissolved metal. The method can be used for all metallic coating, either pure metals

or alloys, but not for insulating oxide and other coatings. The standard instrument has a range from 0.05 to 5 mils, except with chromium coatings, for which the range is 0.005 to 0.1 mil. If an accessory is used, the minimum chromium thickness measurable is $0.5 \mu\text{in.}$, for other metals $5 \mu\text{in.}$ The instrument is not designed for heavy chromium deposits, but for light, decorative chromium coatings. The average test requires about one minute at 115 v 60 cps and the accuracy is 5 to 10 percent, depending on the thickness.

3.5. Capacitance. If the material to be tested for thickness (sheets as well as coatings) has very high resistivity, the measurement of the capacitance of a well-defined area under metallic electrodes can be used to determine the thickness. There is wide variety of possible arrangements of the electrodes, and they may be of the contacting or noncontacting type. The instruments have to be calibrated with samples of known thickness and of the same dielectric constant as the material.

Capacitive methods cannot be used for materials with variable water content, because the high dielectric constant of water (10 to 50 times that of most materials) would cause enormous errors. For accurate measurements it is necessary that the water content either be kept constant by using a controlled atmosphere, or that the standard of thickness have the same water content as the test specimen. One percent moisture in the material may cause an error of 40 percent. Capacitance gages can be used with line frequency, but generally higher frequencies are used to get higher currents and to be less dependent on the insulation. There is a wide variety of methods for measuring capacitance, and a variety of electrodes have been used [100, 142, 143] (fig. 81).



FIGURE 81. Film-thickness gage using mercury electrodes.

Mercury electrodes [100] (fig. 81) have been used by the General Electric Co. to measure film thickness of a molecular order of magnitude. A mercury point electrode is used which covers an area of 0.003 in.^2 , corresponding to a circle 60 mils in diameter. The capacitance is measured between the electrode and the metallic base plate, and from this the film thickness can be calculated within an accuracy of two molecules.

Hand electrodes [142], to be placed on one side of the sheet to be measured, are either two side-by-side half circles, or two concentric electrodes, the diameters between 10 and 30 mm. They are made of metallized sponge rubber to assure good contact.

Liquid electrodes may be used to test objects like rubber gloves for thickness or dielectric strength. The objects are filled with a conductive liquid and immersed in the same liquid in a vessel.

The most straightforward method of measuring capacitance is to use ac of known frequency and voltage and measure the current per unit of area. Some instruments use beat-frequency circuits with either an earphone for tuning or a moving-coil instrument with a rectifier for direct indication of the thickness. Portable, battery-operated instruments are also available.

Very high sensitivities appear possible, especially with beat-frequency methods. Limits of sensitivity with capacitive methods are given as 0.01μ . The accuracy generally depends more on the uniformity of the object than on the measuring method. For very thin coatings or sheets, an uncertainty of ± 5 percent is usually permissible.

3.51. Fielden-Walker Evenness Tester. In this instrument the yarn or sliver is passed continuously at a speed of 5 to 50 fpm through an electric capacitor with an effective field length of 1 centimeter. Three different capacitor heads are available, to cover all materials from the heaviest jute sliver to a single strand of 15-denier nylon filament (15 denier means a weight of 15 grams for 9,000 meters of thread). The capacitor head is one leg of an electric bridge, operated with high frequency. The deviation from zero is proportional to the mass inserted into the capacitor. As already mentioned, moisture of the yarn influences the indication to a very considerable extent. If, however, the material has been stored for a reasonable length of time under constant temperature and humidity, the results indicate the irregularities of the material under test in a fully satisfactory way. The instrument generally works with fixed electrodes, but one special design has portable electrodes.

Manufacturer: Fielden Instrument Division, Philadelphia 33, Pa.

3.52. Noncontacting Capacitors [53] (figs. 82, 83). *Verigraph*. For sticky sheet materials,

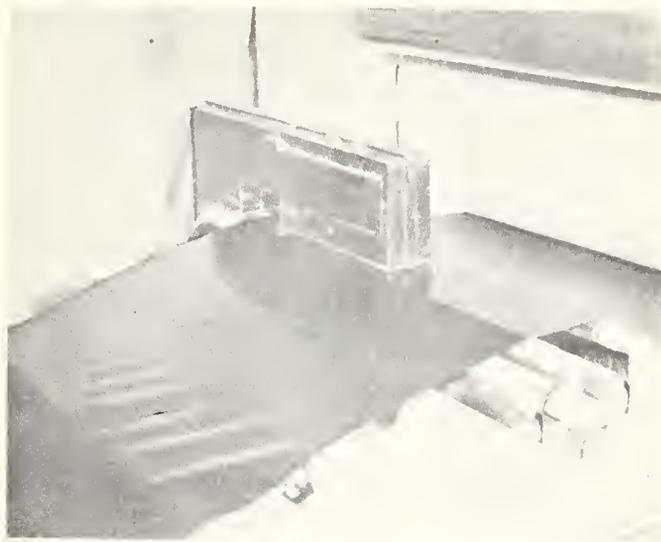


FIGURE 82. Installation of a "Verigraph" on the conveyor of a calender producing sheet stock for rubber footwear.

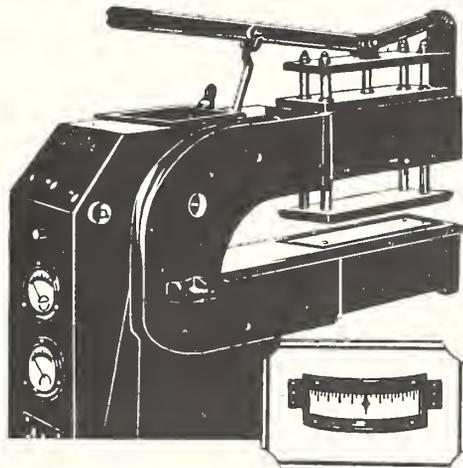


FIGURE 83. Measuring head of the Foxboro "Verigraph."

contact methods cannot be used. In this case the sheet is passed between two fixed air-capacitor plates (figs. 82, 83). If the dielectric constant of the sheet material is higher than unity, the capacitance between the plates will be increased. The deflection of the galvanometer in a bridge circuit can be calibrated in terms of thickness for material with a given dielectric constant. This method has been used for "weighing" rubber-coated sheets—cord fabrics for tires, gum stocks for aprons, raincoats, and other articles of rubber—with substantial increase in uniformity and saving of rubber. The capacitor plates are about 2 by 11 in. and spaced 150 mils apart. A frequency of approximately 1,600 to 1,900 kc is used. The diameter of the uncoated threads is about 20 mils. The rubber coating on each side is 10 mils, so that the total thickness is about 40 mils. The travel of the indicator

pointer is about 2 in. for a change of 1 mil, a magnification of about 2,000. A thickness change of 1 percent gives a deflection of 800 mils. The accuracy (constancy of calibration) is given as 0.1 mil.

Trade name: "Verigraph," Foxboro Co., Foxboro, Mass.

Idometer. The same basic method has been used in Europe under the trade name "Idometer" (Siemens & Halske, Berlin) [53].

3.53. Textile Uniformity Analyzer. A non-contact capacitive method is also used in the "Textile Uniformity Analyzer." The thread is passed between the plates of an air capacitor. The capacitance, measured in a high-frequency bridge, changes in proportion to the weight of the material per unit length. Indications are not affected by the shape of the material, whether it is twisted or not, whether compact or loose.

3.54. Decker Model 103 Comparator Micrometer [77]. In the Decker Model 103 Comparator Micrometer, capacitance variation is converted to electrical signal by means of a transducer element called the T-42 Ionization Transducer. This consists of a glass envelope containing two internal probe electrodes and filled with stable gases under reduced pressure. Under these conditions, a d-c output appears across the internal electrodes. Capacitance changes of 10^{-15} farad or motions of 10^{-6} in. can be measured. The d-c signal developed by the transducer is applied to a differential circuit, which provides an output to the indicating meter of $400 \mu\text{a}$ per volt of input signal. Sensitivities from 0.02 to 0.2 mil for full-scale deflection are given.

Manufacturer: Decker Aviation Corp., Philadelphia 25, Pa.

3.6. Thermoelectric.

3.61. Thermoelectric Thickness Gage [115]. The thermoelectric thickness gage is useful for the measurement of the thickness of electrodeposited or other thin metallic coatings. It consists essentially of two probes, one hot and one cold, a power source, a voltage stabilizer to insure uniform heating, a d-c amplifier, and a milliammeter or other indicator of the amplifier output. In the simplest case where the coating and probe tips are of the same metal, heat flows from the hot probe as a result of contact and raises the temperature of the area under it. A thermoelectric potential difference then exists between the base and the coating metals, causing small electric currents to circulate between them. There is a consequent potential difference between any two points on the coating surface, which can be measured by suitable means. This potential difference is dependent on the nature of the two metals, the temperature difference between the hot probe and the work, the pressure with which the hot probe is applied, and

the shape of the probe. When these conditions are controlled, the measured potential is related to the coating thickness. Using standard samples, calibration curves are made for each combination of metals that is of interest. The standard deviation of the instrument is about 10 percent for coatings that are 0.5 mil and thicker; it is slightly higher for coatings on the order of 0.2 mil. (General Motors Corp. is thought to be doing some work on an instrument similar to the one described here.)

4.00. Magnetic Methods

4.1. Attractive Force.

4.11. *Magnetic Gages* [18, 31, 37, 40] (figs. 84, 85, 86). One type of magnetic gage depends on the attraction between a small permanent magnet and the magnetic base under the nonmagnetic coating (figs. 84, 85). The maximum force is obtained when the coating thickness is zero. Any material between the base and the magnet decreases the attraction, which of course is also dependent on the magnetic properties of the base metal. The choice of the magnet, which has to be constant over long periods of time, depends on the problem. For testing plated coatings, for example, a magnet of 36 percent cobalt steel, 30 mm long and 1 mm in diameter, is used.

The attractive force is measured with a spring balance. To make a measurement, the magnet is brought in contact with the coating and the dial is turned until the magnet is detached. The force necessary to do this indicates the thickness. The principle can be applied to measure the thickness of a magnetic coating like nickel on nonmagnetic base metals such as copper and brass.

The following instruments which operate on this method are manufactured by American Instrument Company under the trade name "Magne-Gage":

Application	Range Mils	Magnet
Type A: Nonmagnetic material backed by iron or steel	0 to 2	M-1
	2 to 7	M-2
	7 to 25	M-3
	25 to 80	M-4
Nickel, which is or can be backed by iron and steel	0 to 0.75	M-1
	0.5 to 2	M-2
Nickel, which is or can be backed by a nonmagnetic material	0 to 1	M-3

There are also special designs of "Magne-Gages," e.g., with extended arm to measure coating thickness on inside surface of objects.

The British Standards Association has developed a pocket-sized thickness gage based on magnetic attraction. It consists of a light mag-



FIGURE 84. Brenner magnetic-attraction thickness gage. Spring balance with cover and false bottom removed.

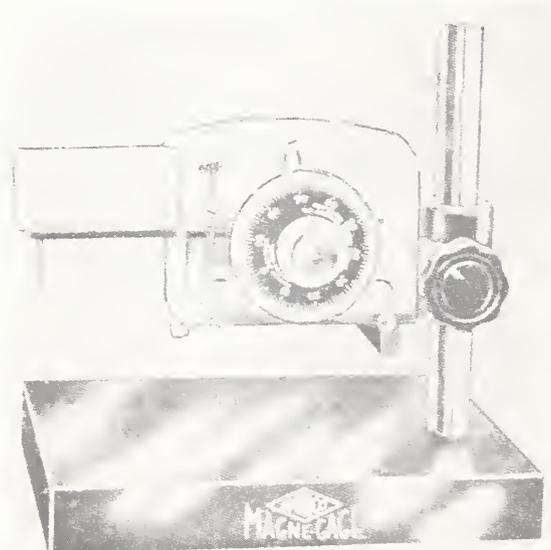


FIGURE 85. Aminco-Brenner "Magnegage," commercial design, with extended arm.

net attached to a spring contained within a pencil-shaped tube (fig. 86). To make a measurement, the magnet is placed on the test surface and the body of the gage is drawn away, thus stretching the spring. The spring extension is observed on the scale and is proportional to the force required to detach the magnet from the surface, providing a measure of the thickness of the nonmagnetic layer. The range of the instrument is from 0.2 to 10 mils; accuracy is given as ± 15 percent.

An instrument working with the same principle is offered in this country by Platers Research Corp., New York City. The range is given as 0.1 to 15 mils.

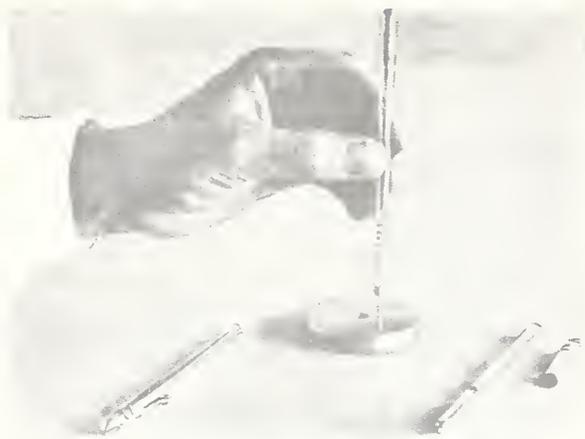


FIGURE 86. BSA/Tinsley pencil thickness gage.

4.12. Magnetic Gages for Glass Bulbs [68].

To measure the wall thickness of a bulb by the magnetic force method, it is necessary to provide a magnetic base. This is accomplished by filling the bulb with carbonyl iron powder. A mechanical vibrator must be run up and down the bulb to settle the powder. A particle size of $20\ \mu$ (0.8 mil) requires about 1.5 minutes. A modified "Magne-Gage" is used and calibrated through aluminum foil cemented over some holes in a test bulb. This is necessary because the readings will depend on the nature and degree of compaction of the iron powder. Sensitivity decreases with the thickness (distance) and also with the size of the particles. With a $3\text{-}\mu$ diameter the sensitivity is about twice as high as with a $20\text{-}\mu$ diameter, but the necessary time of vibration is about 6 minutes.

The test magnet of the Magne-Gage has to be selected for maximum sensitivity. For a thickness range from 0.4 to 7 mils, magnet M-2 is preferred; for the range 4 to 16 mils, M-3 is better. The accuracy obtained is 2 to 3 percent for thicknesses down to 1.2 mils. For thinner walls the accuracy increases, and measurements accurate to $1\ \mu$ (0.04 mil) are easily obtainable.

4.13. *Modified Magnetic Gage for Nickel and Copper on Steel* [40, 43] (fig. 87). A modification of the original Brenner gage [40] is used to measure the attractive force between the specimen and two permanent magnets of different strength. In order to compensate for the difference in the attractive force of the two magnets, they are hung at different distances from the pivot on the balance arm. The gage is calibrated for each magnet and for all-nickel, for 50-Ni-50-Cu, and for all-copper. The percentage of copper or nickel is defined by the point where the thickness readings for the two magnets coincide. A graphical method is used, as indicated in figure 87. Here the thickness falls on the calibration curve for half copper and half nickel, and no interpolation is necessary. Using an interpolation method, the gage is found to be accurate within 10 percent of the

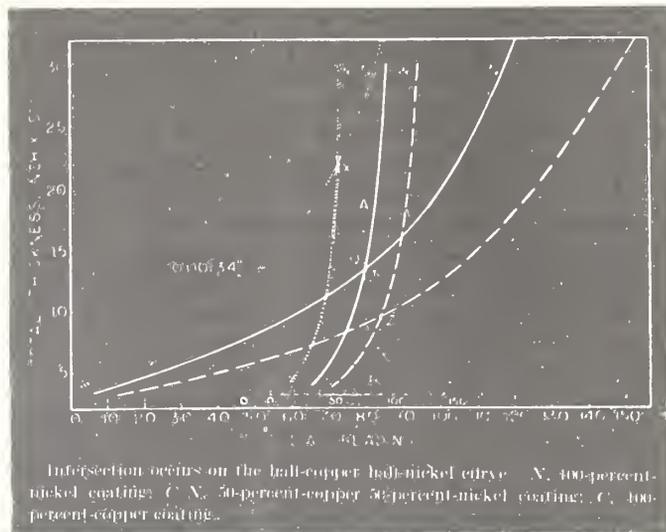


FIGURE 87. Graph to determine ratio of copper and nickel in a composite coating of steel plate.

total thickness and 15 percent of the copper percentage, for a total coating thickness of 0.5 to 3 mils.

Manufacturer: American Instrument Co., Silver Spring, Md.

4.14. Solenoid Pull Gage [133] (figs. 88, a, b).

This instrument for the measurement of thickness of nonmagnetic coatings on steel consists of a solenoid about 4 in. long with a central hole to admit a $\frac{3}{16}$ -in.-diameter glass tube, on which a scale is engraved. A core of iron wire fits loosely inside the glass tube and carries on one end a cellulose acetate rod to act as a mark for the scale. In the equilibrium position, with the coil energized, the location of the core can be noted by reading the position of the indicator with reference to the scale. To use the instrument, the coil is energized by a-c voltage and held vertically over a coated steel article, and lowered, so that the lower end of the core in the glass tube contacts the coated surface. Then the solenoid is raised slowly. At first the core will stick to the magnetic surface. When chat-

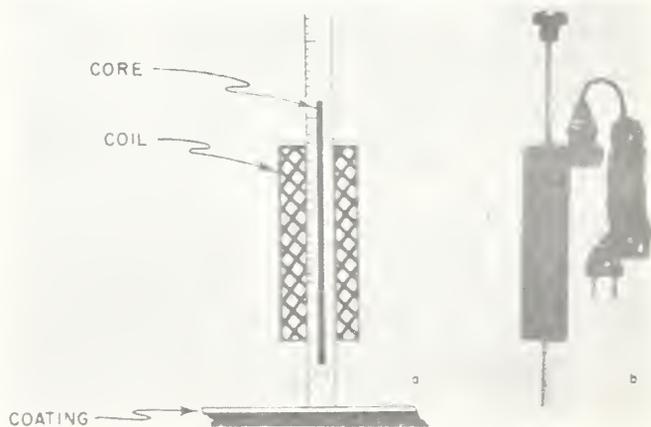


FIGURE 88. Lea "Lectromag."

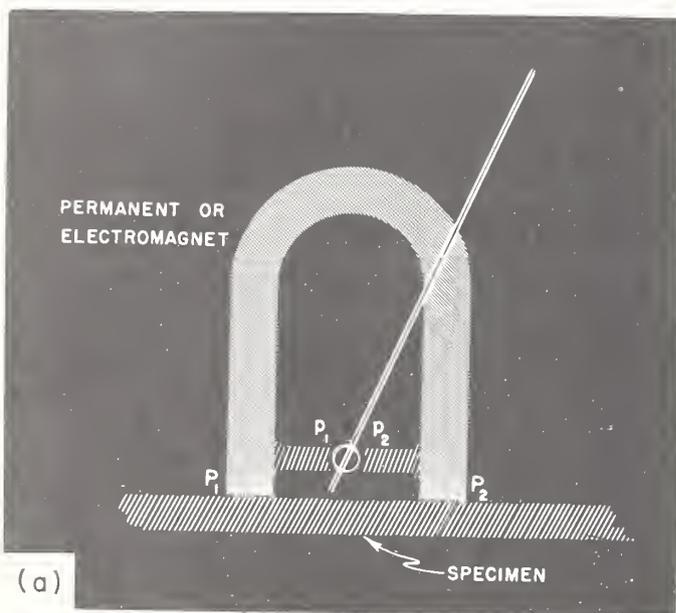
tering can be heard, the core is about to separate from the steel base and finally will go up in the glass tube. When it has come to rest inside the solenoid, the position of the indicator on the scale is read.

The reproducibility of the instrument ranges from ± 5 percent for films 0.2 mil thick to ± 3 percent for coatings exceeding 2 mils.

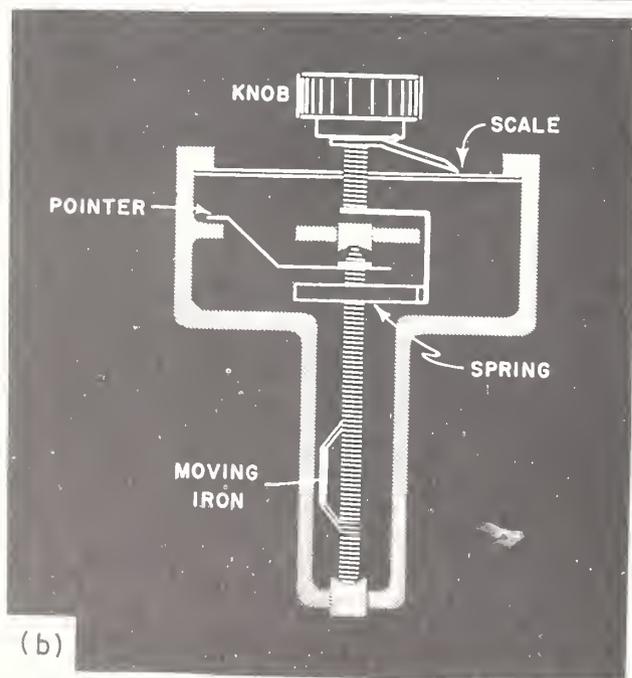
Manufacturer: Lea Manufacturing Co., Waterbury, Conn. Trade name: "Lectromag."

4.2. Parallel Magnetic Flux. These devices measure the flux in a path parallel to the object under test.

4.21. Moore-Williamson Instrument (pat. 2) (figs. 89a, b). A strong magnet of U-shape,



(a)



(b)

FIGURE 89. Magnetic thickness gages measuring the flux in the object under test.

with large pole ends, P_1 and P_2 , is placed on the surface of the iron or steel plate under test (fig. 89a). Two smaller poles, p_1 and p_2 , are so arranged that they carry a stray flux parallel to the sheet. In the gap between p_1 and p_2 is placed a magnetic moving element, eccentric to a non-magnetic spindle. The stray flux between poles p_1 and p_2 will depend on the thickness and permeability of the material between the large poles, P_1 and P_2 . Thin materials will draw less flux than thick materials. Thickness is indicated either by the deflection of the moving-magnet indicator, or the torque (adjusted by hand) to move the pointer back to zero. The instrument has to be calibrated with material of known quality, identical with the plates to be measured. The method can be used for materials up to $1/2$ in. thick. Figure 89b gives details of a torque meter which can be used as an inductor to be placed between poles p_1 and p_2 of figure 89a.

4.22. "Elcometer" Dry-Film Thickness Gage (pat. 7) (figs. 90, 91, 92). This instrument, described in (pat. 7), consists of a permanent magnet with two soft iron pieces, extending into two soft iron contact spheres. This fixed magnet FM (fig. 91) keeps a moving magnet MM deflected against the force of an opposing spring. When the contact spheres C and C are put on a ferromagnetic base, the flux-path of the fixed magnet is in part closed by the base and the pointer takes a new position, equivalent to zero coating thickness. If, now, a sheet of nonmagnetic material such as paper is placed between the contact spheres and the base, the pointer moves nearer the original position. The pointer can be locked after it takes its position and before it is read.

The scale is nonlinear and is calibrated in thickness. Ranges are from zero to 2, 3, 5, 10, 20, 250, and 750 mils, with half the range taking approximately three-fourths of the scale length; suppressed zero scales, 15 to 40 and 40 to 80 mils, are also available. The scale length from zero to infinite thickness is about 32 mm, the



FIGURE 90. "Elcometer" magnetic thickness gage.

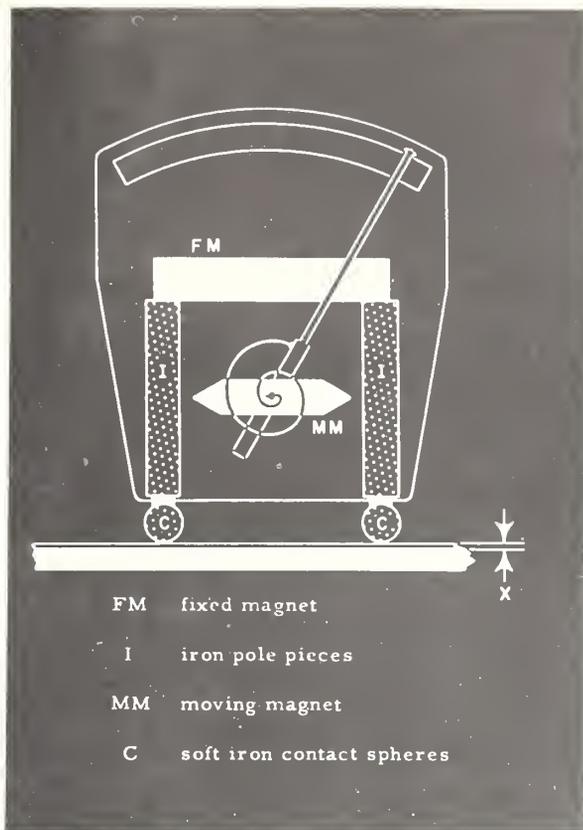


FIGURE 91. "Elcometer" magnetic thickness gage.



FIGURE 92. "Elcometer" magnetic thickness gage.

useful part of it about 28 mm. The accuracy is given as ± 5 percent or ± 0.1 mil, whichever is greater.

Manufacturer: Henry A. Gardner, Bethesda, Md.

4.23. Magnaflux Corporation Meter. This instrument is a small portable meter consisting of a permanent magnet and a galvanometer. The permanent magnet probe is placed on the sample to be measured and the thickness read directly in thousandths of an inch on the meter.

Accuracy is within ± 0.0005 in. when used on vitreous enameled steel, and hot-rolled, cold-rolled, and deep-drawing steel sheet. Typical applications include the measurement of thickness prior to deep-drawing so that the dies may be set for the proper thickness of material, and measurement of thickness of tin-can stock to assure the proper bending to form good seams.

This instrument is designed to measure sheet thicknesses from 0 to 0.050 in. Its advantages are its speed, its lack of any power supply such as line voltages or batteries, and its ability to measure sheet from one side only at any location.

Manufacturer: Magnaflux Corp., Chicago 31, Ill.

4.3. Magnetic Saturation. The saturation magnetic flux density (maximum number of flux lines per unit area) of a ferromagnetic material is a property characteristic of the material. When material of fixed composition is included in a magnetic field, strong enough to magnetize the material to saturation flux density, the total flux producing this flux density is a function of the thickness at the area under observation.

Mechanical working, and temperature to a lesser extent, affect the magnetic properties of some materials. For this reason, the reliability of the instrument chosen is somewhat dependent on calibration for the conditions under which measurements are to be made, as well as for the composition of the material.

4.31. GE Magnetic Saturation Gage [52, 188] (figs. 93, 94). A gage based on magnetic saturation has been developed by the General Electric Co. especially to measure the thickness of common grades of sheet steel from 10 to 45 mils thick, measuring from one side. The principle is the following: The steel sheet with known magnetic properties is magnetized by means of a permanent magnet to saturation. A strong ring-shaped Alnico magnet is used in the measuring head, together with a central pole of soft iron (fig. 94). The flux from the Alnico magnet enters the sheet and flows radially to the central pole, which, together with the yoke (also of soft iron), completes the flux path. The sheet itself must have higher reluctance than any other part of the magnetic circuit; it must be the "bottleneck" for the flux.

The flux in the center pole is determined by measuring incremental permeability. To do this, a laminated transformer core with primary and secondary windings is placed in the central pole (fig. 93). The primary winding carries alternating current, which produces an alternating flux which flows in the laminated core, as shown by the dotted lines. The alternating flux linking the secondary coil is a function of the magnitude of the steady flux supplied by the permanent magnet through the center pole. The

average secondary voltage is measured by the voltmeter, V, and is proportional to the peak a-c flux. This flux is a measure of the d-c flux and therefore of the thickness of the sheet.

The bucking transformer, T, suppresses the voltage induced in the secondary winding, S, when the gage is placed on a sheet of infinite effective thickness. The instrument has two ranges, one from 10 to 25 mils for magnetic sheets of medium silicon content; the other from 20 to 45 mils, for low-carbon steel either hot- or cold-rolled. The accuracy is given as 0.5

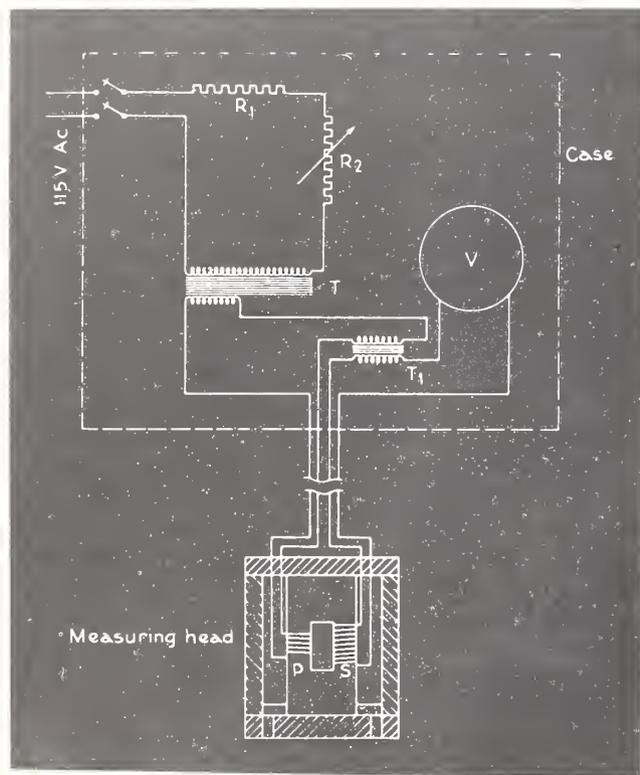


FIGURE 93. Thickness meter for sheet iron, using the magnetic saturation method.

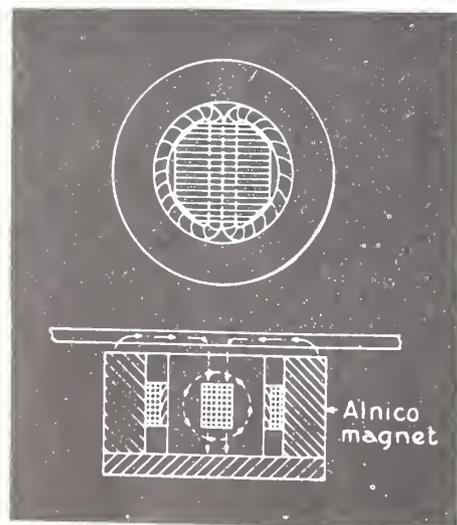


FIGURE 94. Thickness meter for sheet iron, using the magnetic saturation method.

mil when comparing one part of a sheet with another part of the same sheet, or in comparing sheets of the same grade, provided that the gage is more than one-half in. from the edge of the sheet and that the sheet is not backed up by other magnetic material. The measuring head must make close contact with the sheet.

Manufacturer: General Electric Co., Schenectady, N. Y.

Another steel plate thickness meter operating with dc has been developed by the British Iron and Steel Research Association [52]. It can be used for steel plates up to 250 mils thick. On normally machined surfaces the error is given as less than 3.5 percent or 3 mils, whichever is greater.

4.4. Coil Reactance [5, 102, 129, 174, 199].

Principle: The ferromagnetic material to be tested, or the ferromagnetic base on which a sheet of the material under test is placed, is part of an otherwise fixed magnetic circuit of soft iron, magnetized by an a-c current of known constant frequency supplied from a known and preferably constant a-c voltage. The total magnetic reluctance, and therefore the reactance of the circuit and the current in the coil, is changed by changes of thickness of the ferromagnetic base, or of the nonmagnetic material acting as an air gap between the ferromagnetic base and the rest of the magnetic system. This principle can be applied in many ways. A large percentage of electromagnetic thickness meters for nonmagnetic sheets or coatings use it.

4.41. GE Reactance Gage for Nonmagnetic Coatings on Magnetic Base (figs. 95, 96, 97). In using this gage, the sheet under test is placed under a measuring head (fig. 95) on a flat iron plate at least 1.4 in. thick, with negligible magnetic reluctance. There is, for zero thickness, a maximum of reactance and a minimum of cur-

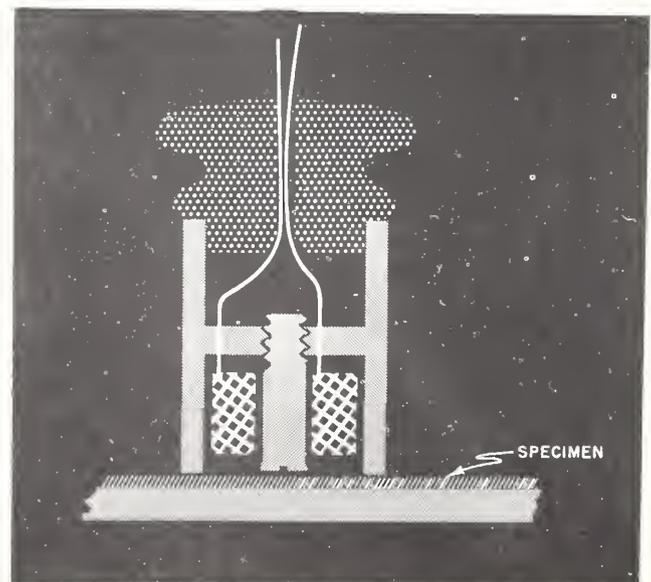


FIGURE 95. Measuring head of a thickness meter of the reactance type.

scale length of the ammeter. A better method is to place the a-c magnet system in one arm of an a-c bridge (fig. 96). This compares the variable reactance with a constant reactance with about the same power factor in the opposite arm, which is balanced so that for average thickness there is zero current in the diagonal. The diagonal current is rectified with dry-disk rectifiers.

Use of the bridge method has the following advantages: (a) Independence of voltage variations, (b) independence of frequency variations and waveform of the voltage, (c) an approximately linear scale for thickness, and (d) much higher sensitivity than is obtainable by simply measuring the current with an ammeter. Figure 96 shows the circuit of one of these thickness meters. Such instruments in portable form can be used to measure sheets and coatings down to 0.1 mil thick, and up to 750 mils. To suit typical rent. A nonmagnetic coating or sheet increases the current. In principle, any a-c ammeter can be calibrated in thickness, but it requires constant voltage, and the thickness scale is either very nonuniform or covers only part of the total

application problems, GE manufactures three types of such electromagnetic gages to measure the thickness of nonmagnetic sheets and coatings. Type A has the smallest measuring area, about 1.8 in.² Next is type B, with 3 in.²; type C has an area of about 7 in.² Figure 97 gives the ranges for these three types and that of a fourth instrument, described in 4.44, the "Coatingage."

All these gages are generally used to check deviations from a standard thickness. To obtain highest accuracy, it is usual to calibrate the instruments with thickness standards, and if a magnetic base is necessary to make the measurement, to calibrate with the base actually used. The accuracy obtained with this type of gage is generally 5 to 10 percent of the thickness of the standard. The error is a minimum for equal thickness, and increases with the deviation. If a null method is used the accuracy is better, around 1.5 percent of the thickness of the reference standard.

Manufacturer: General Electric Co., Schenectady, N. Y.

Another instrument in this group is the "Magnatest FT-300" of the Magnaflux Corp. It is operated from a 60-cps power supply. A small electromagnet in the detector induces a flux in the part under test. The indicator responds to the reluctance of the path offered to the magnetic flux between the detector pole pieces. This instrument is generally used to measure the thickness of paint or plating. The indicator has a dual range of 0 to 4 and 0 to 20 mils. The accuracy is given as ± 2 percent.

Manufacturer: Magnaflux Corp., Chicago 31, Ill.

4.42. *Haskins Turner Co. Gage for Scale Inside Boiler Tubes* [112]. This gage measures the thickness of scale inside boiler tubes. For no scale deposit, the inductance of the search-magnet is highest and the current is a minimum, marked as zero scale thickness on the instrument dial. The current increases rapidly with scale thickness, so that even a scale thickness of 1 mil gives a deflection of about 7 percent of the length of the meter scale. The search unit consists of a cylindrical electromagnet which is held against the inside wall of the tube by means of a spring and guide wheels. The head is simultaneously rotated and pulled through the tube. The scale deposit on the inner side of the tube represents an airgap in the magnetic circuit formed by the electromagnet and the tube. The range of the meter is 0 to 100 mils, with 20 mils in the middle of the scale. The head is connected with a 50-ft graduated steel tape. One operator lowers the probe head down the boiler tube from the upper header or drum; the other operator notes the scale thickness for each foot of travel. Two sizes of probe heads are used, one for tubes with less than 2-in. di-

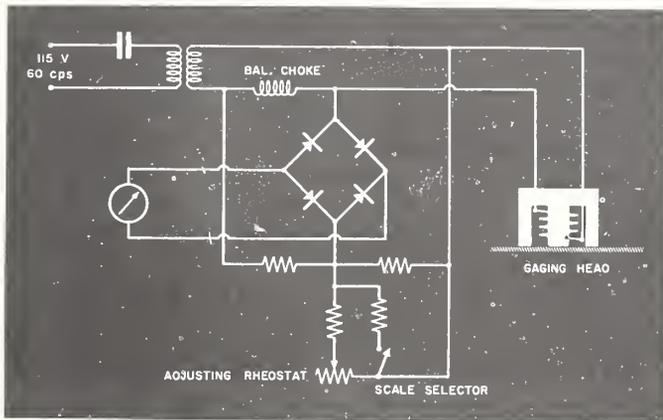


FIGURE 96. Basic circuit of the GE reactance-type thickness meter.

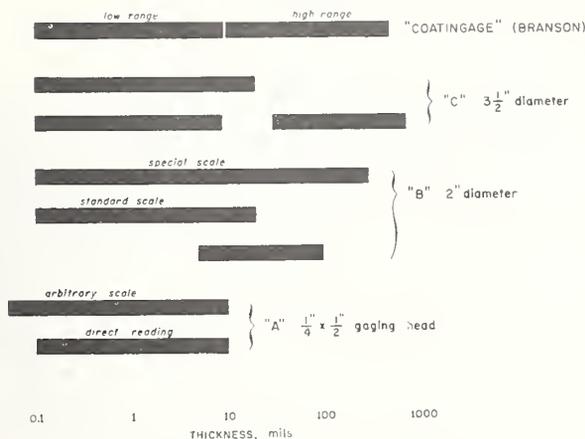


FIGURE 97. Measuring ranges of different types of GE and Branson thickness gages.

ameter, the other for tubes over 2-in. diameter. The thickness of the ferrous tube wall has no bearing on the indications. The instrument is operated from a 115-v 60-cps power supply.

Manufacturer: Haskins Turner Co., Jackson, Mich.

4.43. "Hurletron" for Paper Production [75] (figs. 98, 99). Figure 98 shows the gaging head of the "Hurletron" gage, a roller gage of the reactance type which has been developed to measure the thickness of paper board and heavy-weight paper as it comes from the calender. The wet paper is passed over a dryer or idler roll, and the gage measures the distance between the roll and the idler. Measurement is not distorted by the water content of the paper. The current represents the thickness of the



FIGURE 98. Closeup of a "Hurletron" gaging head.



FIGURE 99. Typical installation showing three gaging heads in operating position.

paper. The accuracy is given as ± 3 percent. Figure 99 shows an installation with three gaging heads.

Manufacturer: Electric Eye & Equipment Co., Danville, Ill.

4.44. "Coatingage" (figs. 100, 101). The instruments described so far operate on 60 cps from the power line. To use thickness meters of this type, where no a-c power is available, the "Coatingage" has been developed. It operates from a built-in battery and a low-frequency oscillator. The a-c unbalance current of the bridge is rectified and indicated by a microammeter. The gage coil of the instrument is wound on a chromium-plated steel spool, the flanges of which are placed in contact with the surface being tested. Figures 100 and 101 show circuit and scales of the two-range instrument. The low range is from 0 to 12 mils, usable between 1 and 12 mils, the high range from 10 mils to 500 mils. The tolerance is given as 0.1 mil up to 1 mil thickness of the coating. Above 1 mil the tolerance is 10 percent, provided the magnetic base is at least 125 mils thick and the instrument head is not placed closer than 1 in. from the edge of the sheet.

Manufacturer: Branson Instrument Co., Stamford, Conn.

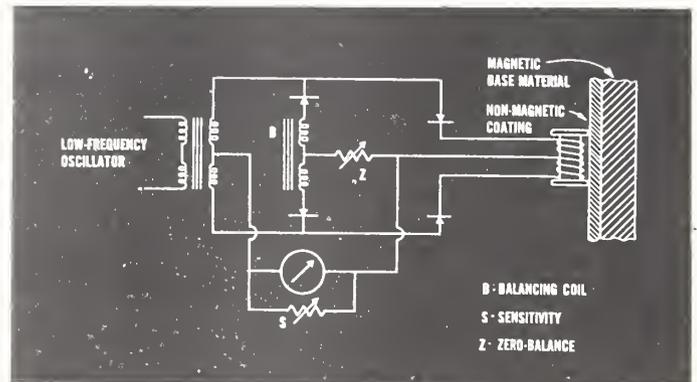


FIGURE 100. Circuit of the "Coatingage" thickness meter.

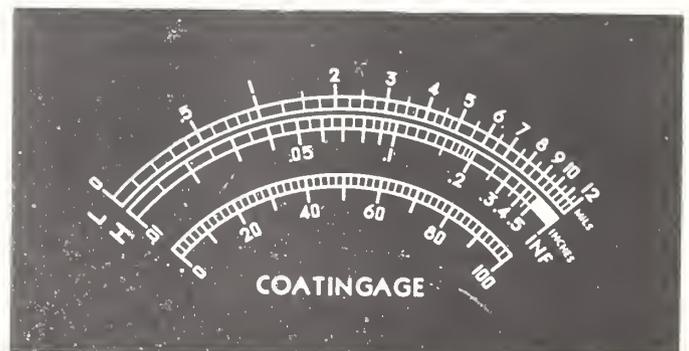


FIGURE 101. Scale character of the "Coatingage."

4.5. Transformers.

4.51. *Transformer Method for Nonmagnetic Coatings on Steel (One Side)* (fig. 102). In its simplest form, figure 102, a small U-shaped iron core is used, and the primary winding is

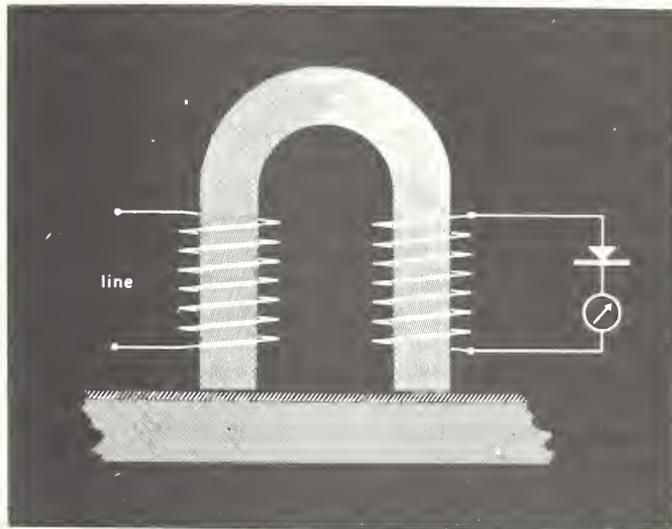


FIGURE 102. Simplest transformer.

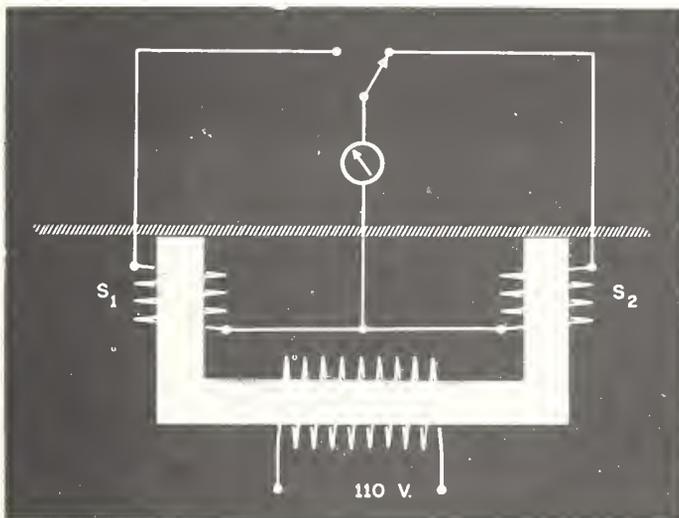


FIGURE 103. Cylinder-wall thickness meter.



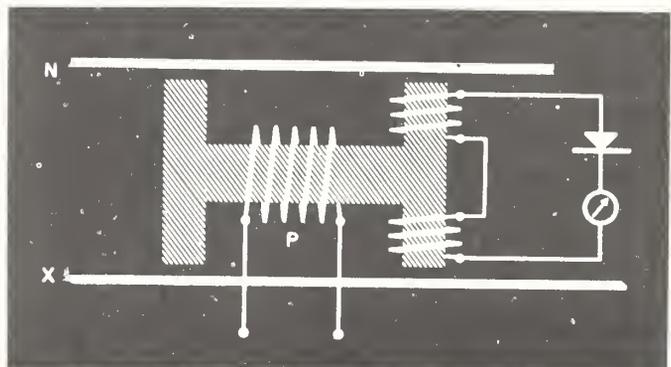
FIGURE 104. Cylinder-wall thickness meter.

connected with the 60-cps power line. Both pole ends touch the coated surface, the magnetic circuit being closed by ferromagnetic base metal of sufficient thickness and high permeability. The voltage of the secondary coil is measured with a rectifier voltmeter. Increasing the thickness of the nonmagnetic coating decreases the secondary voltage. The scale is reversed; maximum deflection corresponds to minimum thickness. The readings are sensitive to voltage and frequency variations.

4.52. *Transformer Method for Ferromagnetic Materials (One Side)* (figs. 102, 103, 104). When this principle is applied to measuring thickness of ferromagnetic materials without coatings, the magnetic reluctance of the material is measured. With the arrangement of figure 102, without a coating, a minimum voltage is produced when the instrument is not in contact with ferromagnetic material. The voltage, measured with a moving-coil rectifier instrument, increases approximately in proportion to thickness until saturation of the yoke is reached. This principle is used in a gage designed to measure the wall thickness of automobile cylinders after rough boring. Two measurements are made, one at the top, the other at the bottom of the cylinder. The magnetic system (fig. 103) is a long, U-shaped magnet with a primary coil connected to the line and with secondary coils on each leg. The whole magnet system is built into a cylinder which fits into the cylinders to be tested. Figure 104 shows the entire assembly. The adjustment of the instrument is made so that with the normal minimum thickness the pointer deflects to 50 percent of the scale length. A thickness change of 10 percent changes the deflection about 5 percent of the scale length. The magnetic properties of the alloy influence the deflection less than about 10 percent.

Manufacturer: Commercial Engineering Laboratories, Detroit, Mich.

4.53. *Sheet-Iron Tester, d-c Operated* [161] (fig. 105). This device measures the thickness of sheet iron by comparing it with a standard thickness. The transformer core, figure 105,



has the shape of an H, with one primary winding, P, and two secondary windings in opposition. On one side is the sheet N of standard thickness, on the other the material X under test. One practical design has a core of about 2.4 by 2.4 in., the yokes being 0.8 by 0.8 in. and the poles 0.8 by 0.4 in. Because it is not possible with any sizable pole area to make perfect contact with the magnetic body, this instrument does not attempt full contact, but provides air-gaps 1 mil thick under each head. The core rests on three hard points of nonmagnetic material, which penetrate layers of rust or paint.

This device can be operated with ac as well as dc. In using d-c excitation the primary voltage furnished by a battery is reversed and the ballistic deflection of the galvanometer observed. This instrument has been used for measuring ferromagnetic boiler tubes, using curved pole ends.

An instrument based on this principle is manufactured by the Magnaflux Corp., under the trade name "Magnaflux Series FT-100." The thickness range is 0 to 80 mils; the accuracy is given as ± 2 percent. One design has a portable measuring head with a small indicator for "go" and "not-go" limits.

4.54. *Nonmagnetic Coatings, Differential Voltage* [76] (fig. 106). This instrument measures the thickness of nonmagnetic coatings on a ferromagnetic base, using the differential voltage in the two secondary coils of figure 106. The deflection is zero for zero thickness, the difference increasing roughly in proportion to the thickness of the coating. Here the test specimen is clamped between and is a common part of two magnetic circuits, each having a primary

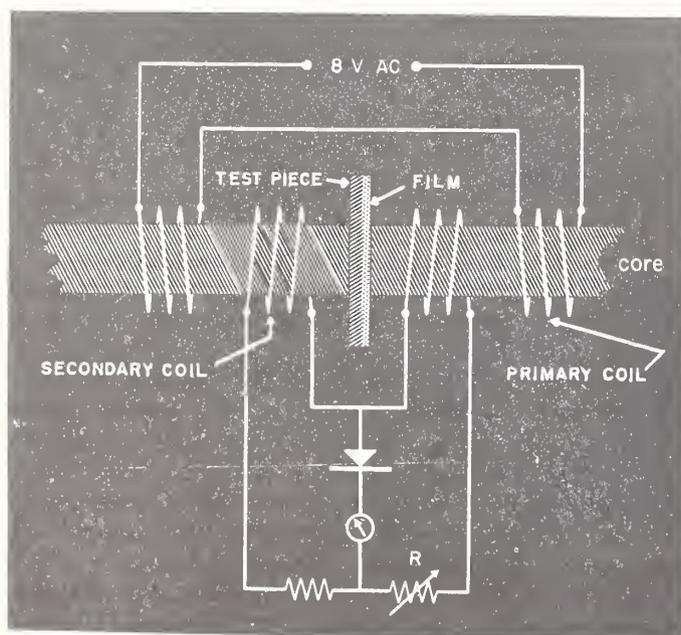


FIGURE 106. Coating-thickness meter.

and a secondary. The nonmagnetic coating introduces a gap in one of these and produces an unbalance. The output may be indicated directly with a rectifier voltmeter of sufficient sensitivity, or a null galvanometer may be brought to zero reading by restoring balance with adjustment of the resistor R.

Such devices can be made very sensitive and indicate thickness changes down to 0.01 mil.

4.6. *Eddy Currents, Inductance Change*. Principle: The inductance of a coil fed with a-c current decreases when the magnetic field of the coil induces eddy currents in a nonferrous conductor near the coil. Generally, two coils are used, connected in such a way that they form two legs of an electrical bridge circuit. The unbalance of the bridge is a measure of the thickness of the material in the vicinity of the coil, provided its distance from the coil is fixed. To measure the thickness of nonconducting material, a thick conducting base is provided, and eddy currents are induced in it. Under these conditions the intensity of the eddy currents depends on the distance of the coil from the base metal. The minimum base thickness necessary depends on frequency and conductivity; the higher the frequency, the thinner the base may be.

Any of the meter designs and circuits used to measure a change of inductance may be used. The frequencies used vary from 60 cps up to 1,000 kc; the design of the equipment depends upon frequency. The inductance-change effect depends very much on the frequency; for each object thickness there is a frequency that gives maximum sensitivity.

To measure the thickness of coatings or sheets from one side, two inductance coils are generally arranged in one head, the search coil being small and so built that it can be brought very close to the surface to be tested. Proximity to the metal base decreases the inductance of the test coil. To measure the thickness of metallic foils with both sides accessible, the search inductance is split into two halves and the metallic foil is passed between them, increasing the eddy-current loss and decreasing the inductance with increasing thickness of the material. When used to determine the thickness of nonconductive, nonmetallic coating on a nonmagnetic metal base, the zero position of the scale must be determined first by placing the search coil on a part of the metal free of any coating and perfectly smooth. In this condition the meter is adjusted, generally by a variable inductance. The calibration is established by placing sheets of nonconducting material of known thickness between the search coil and the base.

Magnaflux FT-400. The Magnaflux Corp manufactures an instrument of this kind, the "Magnaflux FT-400," with a bridge circuit and

a hand detector. Scale ranges are 0 to 2, 0 to 4, and 0 to 20 mils. The instrument operates at 60 cps and the accuracy is given as ± 2 percent of the coating thickness. The measured area has to be flat, with a diameter of more than $\frac{3}{4}$ in.

4.61. "*Filmeter*" [142] (fig. 107). This portable battery-operated instrument, "*Filmeter*," developed to measure the thickness of oxide coatings on aluminum, contains two oscillators, one of them with a constant standard frequency, the other with adjustable frequency. The search coil is at the end of an insulated piston which can be moved against the pressure of a spring in an aluminum cylinder. The eddy-currents in the specimen, under the coating, change the frequency. The frequency is readjusted to the frequency of the standard oscillator by turning the dial of a variable air capacitor until the earphone detector finds frequency balance. The indications are practically independent of the conductivity of the aluminum base, but the base has to have a thickness of at least 11 mils. Surface roughness, under practical conditions, changes the indications less than 2 percent. The method could, of course, also be used to get a pointer indication of the thickness with a rectifier and a moving-coil instrument.

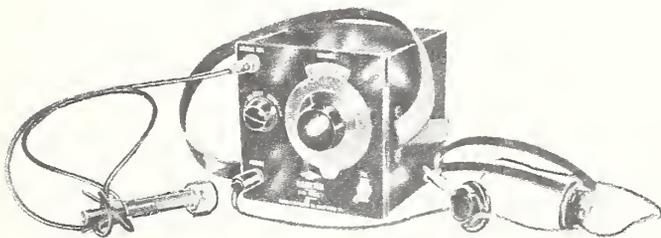


FIGURE 107. "*Filmeter*" with measuring head and earphone.

The accuracy of the *Filmeter* is given as ± 3 percent of full scale for the range 0 to 5 mils and ± 5 percent for the range 0 to 3 mils.

Manufacturer: American Instrument Co., Silver Spring, Md.

4.62. *Nash & Thompson Eddy-Current Gage* [120]. A British company has applied the eddy-current principle to the measurement of nonconducting coatings (paint, varnish, etc.) on nonferrous bases. The instrument consists of a coil which in conjunction with a variable capacitor forms the tuned circuit of an oscillator. The frequency of this is compared with that of an incorporated reference oscillator by means of a mixer circuit. The test coil, mounted in a housing and supported on three ball feet, is placed on the specimen. The variable capacitor

is adjusted until the null point is indicated. The coating thickness can be read from a dial fitted to the shaft of the capacitor. Variations in the base metal are compensated by the initial adjustment on an uncoated sample. A flat area on the test piece of $1\frac{1}{4}$ -in. diameter is required for the head. The range of coating thickness covered is 0 to 0.03 in.

Manufacturer: Nash & Thompson, Ltd., England.

4.63. *GE Eddy-Current Gage for Hollow Propellers*. The General Electric Co. has used the eddy-current method to measure the thickness of propeller walls. The search coil is calibrated by holding it in contact with walls of the same material and of known thickness. The measuring range decreases with increasing conductivity of the material under test. While the range is 0.25 in. for copper, it is 1.5 in. for brass. The accuracy was given as 5 percent. Production has been discontinued.

4.64. *Eddy-Current Gages for Moving Metal Sheets* (figs. 108, 109, 110). For this type of gage the material runs freely through an airgap between high-frequency coils. A meter, manufactured by GE but now discontinued, had an airgap of 375 mils for the material and was accurate to 0.01 mil.

Salford Gage. An instrument made by Salford Electrical Co., Ltd., England, is built for the following ranges; 1 to 5 mils, 2 to 10 mils, 5 to 25 mils, and 10 to 50 mils. The calibration, as with all these instruments, has to be made for the specific metal. Figure 108 shows the measuring head of the Salford foil-thickness meter.

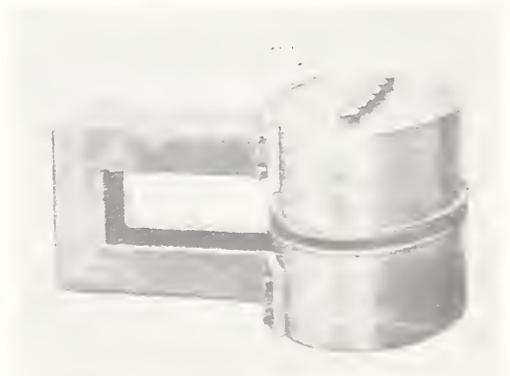
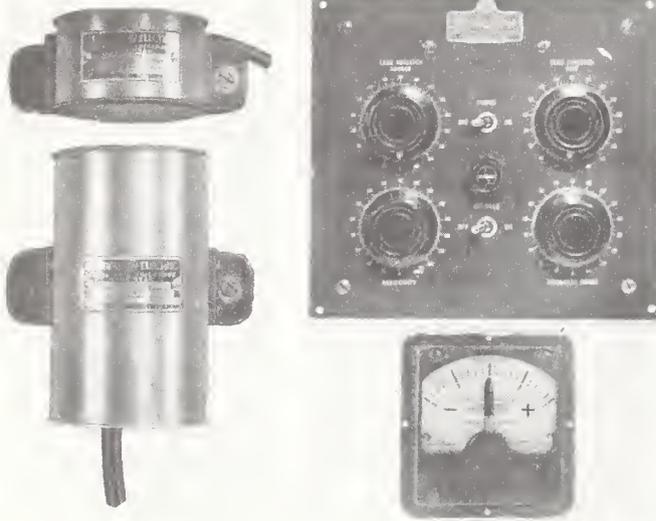


FIGURE 108. Measuring head of the Salford foil thickness gage.

Pratt & Whitney Gage. The foil gage of Pratt & Whitney, to be used directly with rolling mills, is shown in figures 109, 110. The gaging head consists of two parts: The lower part is under the coil in a fixed position, the upper part swings up out of the way while the mill is being threaded. The material passes between the upper and lower parts of the gage head. This gage can be used to measure foils from 0.25 to



FIGURES 109 and 110. *Pratt & Whitney foil gage.*
Gaging head. Power unit (adjustment of zero, coarse and fine sensitivity, thickness range). Indicator.

10 mils thick. The indicating meter, calibrated for the deviation from standard thickness, has two scales, 0 to 0.2 mil and 0 to 2 mils.

Magnatest FT-200. Another eddy-current gage to measure thickness of moving sheets and foils of copper, brass, or aluminum in continuous production, is manufactured by the Magnaflux Corp. under the trade name "Magnatest FT-200." It is operated on 60 cps. The thickness range may be from 30 to 80 mils. The range of the zero-centered indicator can be made ± 10 percent of any nominal value between these limits. The measuring area has a diameter of 4 in.; the accuracy is given as ± 0.1 percent of the nominal value.

4.65. *Gages for Lead Sheath on a Cable* [131]. Eddy-currents have been used for measuring the thickness of the lead sheath applied to cables by an extrusion process in the British Callender Cable & Construction Works in London. The method was later adopted by the Okonite-Callender Cable Company in Passaic, N. J. Here the influence of frequency is important. At 50 cps, it was found that the magnetic flux passed freely through the lead and little change of inductance took place. Up to about 500 cps a linear change of inductance took place, approximately 2.5 percent per 100 cps. The maximum change of inductance was at 1,400 cps; for higher frequencies the effect decreased and became almost negligible for more than 2,200 cps. For this reason the measurements were made at 1,400 cps. The flux was induced with four electromagnet coils mounted around the cable sheath, as it came from the press, using roller bearings to contact the sheath surface. The output after rectification was measured with a d-c instrument. Thickness measurement in this application corresponds to

determination of concentricity. The trade specifications allow 18 percent eccentricity. With the gaging device it was possible to keep eccentricity within about 5 percent, or 7.5 mils for 150-mil average thickness.

4.66. "*Probolog*" (fig. 111). Corrosion of condenser tubing presents a problem to many power plants. The tubes have outer diameters from 0.625 to 1.250 in. and a wall thickness from 28 to 165 mils. They are of nonmagnetic metals (copper, brass, stainless steel) and they are used in bundles up to 5,000 tubes. Test equipment for this problem, called a "Probolog," has been developed by the Shell Development Co., Emeryville, Calif. The probes, with diameters of 0.313 to 1.005 in., have interchangeable Mumetal cores with an axial length of 900 mils, figure 111. Two search coils are mounted on the core, each about 300 mils long, the coil centers about 370 mils apart. The clearance between probe and tube ranges from 40 to 152 mils. The probe is first driven by compressed air to the far end of the tube, and then slowly withdrawn with a power winch at a speed of 1 or 2 fpm through the tube length, which is generally about 20 ft. As long as the tube under test is uniform in thickness, the eddy-currents induced in the tube by the two coils (the tube acting like a short-circuited winding over the coil) are identical and the balance of the bridge circuit is not disturbed. If there is, however, a thinned area ("pool") caused by corrosion, on either inner or outer surface of the tube, the bridge becomes unbalanced. The unbalance voltage is converted into a current which energizes a solenoid to rebalance the circuit. The rebalance current is recorded on a strip chart which moves in synchronism with the pulling of the probe. With proper interpretation and calibration with test pieces of the same tubes having thickness differences along their length and also holes of known diameter, the size and depth of corrosion pools and other defects can be determined with fair accuracy, reaching ± 5 percent as far as the wall thickness is concerned.

For the different tube diameters, seven probe diameters are available. Recommended practice is not to test all tubes, but to make a hydrostatic test first to find defective tubes that fail under

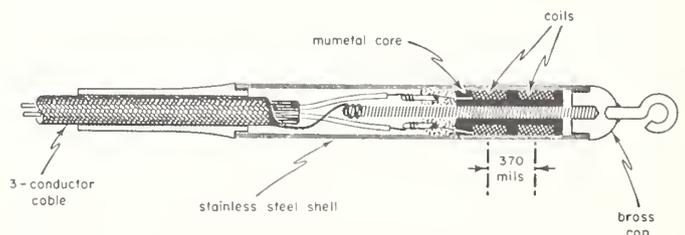


FIGURE 111. *Search-coil assembly for the "Probolog" condenser-tube tester.*

the test pressure, and after this to test all tubes in the vicinity with the electromagnetic method, as described.

Manufacturer: Shell Development Co., Emeryville, Calif.

4.67. Gage for Ceramic Coatings on Metal [106]. A ceramic-coating thickness gage has been developed to measure the thickness of protective coatings of metals and alloys in high temperature service. The range of this instrument is from 20 to 100 mils, with an accuracy of about ± 0.2 mil for 20 mils and ± 1 mil for 100 mils, i.e., about 1 percent of the indication of the moving-coil meter within this range. For this application a frequency of 500 kc is used.

4.68. Gages for Metal Coatings on Metal [44, 49, 209]. Conductive metallic coatings on a base of different electric conductivity can be measured by using the eddy-current principle.

One device employing this principle [44] uses a commercial type of oscillator with a high-frequency output. Two germanium diodes in opposite phase cause current to flow alternatively through the two legs of a bridge circuit, one leg including the test probe coil, the other, a variable resistance. A direct-current microammeter in series with the bridge circuit receives first the current through the test probe, then the current through the variable resistor, and registers the current difference.

The probe is first placed on a reference (uncoated) surface and the meter adjusted to read zero. It is then placed on the surface to be tested and the current change noted. In practice, this current change is a direct indication of the thickness of the coating in question. The magnitude of the induced eddy current, dependent on both difference in conductivity between the base and coating metals and the thickness of the coating, in turn determines the strength of its own magnetic field. The field of the eddy current opposes the field of the inducing current, thus lowering the impedance of the probe coil and allowing proportionately more current to flow through the probe. The recorded test currents are interpreted by comparison with a calibration curve prepared by measuring samples of known thickness.

The sensitivity range and accuracy of the eddy-current instrument are dependent on such variables as the frequency used (e.g., a frequency of 2 Mc is found effective for coatings up to 1.5 mils thick, and 100 kc for coatings up to 6 mils), circuit resonance, and the differences in conductivity of the two metals.

The Boonton Radio Corp. Gage, manufactured by the Boonton Radio Corp., Boonton, N. J., and the "Dermitron" gage, manufactured by Unit Process Assemblies, N. Y., are two examples of this type of gage.

Magnatest FT-600. Magnaflux Corp. has a commercial type [49] "Magnatest FT-600 Se-

ries" with a very small contact head which forms one arm of a bridge circuit. A typical unit has a scale graduated in mils to 10 mils.

4.7. Mutual Inductance With Air Core [42, 107, 141] (fig. 112). Principle: The mutual inductance transducer consists of a primary and a secondary coil wound on the same coil form, but at separate positions on the form. A metal surface lies near the secondary coil (fig. 112). When the primary coil is excited with rf, eddy-currents are induced in the metal surface. If the metal were an ideal conductor the field of the primary coil at the surface would be just canceled by the field of the eddy-currents. When the mutual inductance transducer is placed so that the secondary coil is adjacent to the metal surface, there will be no voltage induced in it. As the transducer is moved away from the metal an increasing voltage will be picked up by the secondary coil.

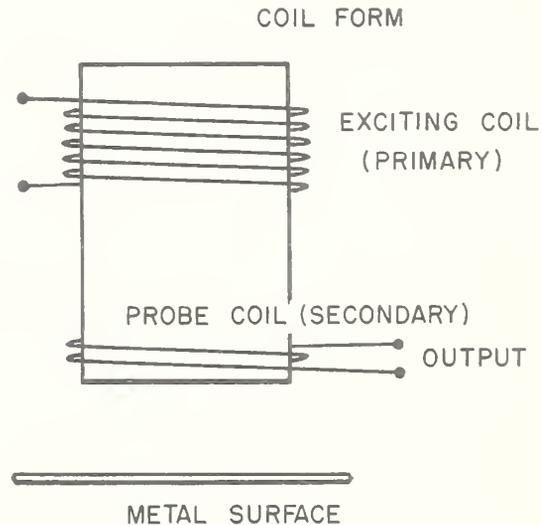


FIGURE 112. Basic structure of distance-measuring element.

Mutual inductance between two coils is varied by proximity of metallic surface.

4.71. Measurement of Bearing Eccentricity [107, 141, 185] (fig. 113). This instrument measures the displacement of a turbine shaft in its journal bearing. The problem is to measure the thickness of the oil film in a 6-in.-diameter bearing with a radial clearance of only about 6 mils, while running at more than 10,000 rpm submerged in lubricating oil at 200°F.

Four distance-sensitive elements are used, mounted around the shaft, immediately beyond the bearing in positions 90° apart. Each consists of two concentric coplanar coils, wound on a single bakelite form, 1 in. in diameter (fig. 113), the inner coil having a diameter of 70 percent of that of the outer coil. For such a coil system the output is linear up to a distance of about 50 mils. The shaft is electroplated with

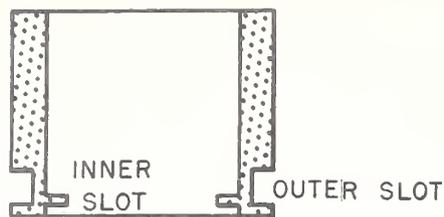


FIGURE 113. Probe coil form used in oil-film thickness indicator.

copper, 1 mil thick, in a band immediately under the distance-sensing coil assemblies. A frequency of 2,500 kc is used. The output from the four pick-ups is connected with the four plates of a cathode-ray oscillograph and in this way a picture of the eccentricity of the shaft in the bearing is obtained. The range is ± 13 mils. One mil is represented by two lines 150 mils apart, giving a magnification of the eccentricity of 150. The over-all accuracy of the method is estimated as better than 0.5 mil.

The same principle has been used to measure and indicate the longitudinal movement of high-speed rotating shafts, such as might be caused by failure or wear of a thrust bearing [185]. A brass disk, which forms part of the electrical system, is fastened on the end of the turbine shaft. Motion of the disk towards or away from the probe changes the mutual inductance between two windings. The output voltage, indicated on a meter, shows shaft position in units of mils on a d-c instrument.

4.72. Phase-Angle Thickness Gage for Silver Coating on Stainless Steel [9, 220]. The phase-angle thickness gage is useful only when the resistivities of the base material differ materially from that of the coatings. The probe consists of a mutual inductance transducer which is held in proximity to the sample. A buckout transformer shielded from the sample is also located in probe head. The two elements are electrically connected and excited with rf current in opposition, producing output voltages whose phase difference is responsive to the conductance of the sample. A phasemeter is calibrated for the desired frequency ranges. The application of this device given in the references is for silver on steel. For direct reading of thickness, the phasemeter output is indicated on a meter calibrated in mils of silver which has been applied to a base of 37 mils of stainless steel. The probe is brought within $\frac{1}{8}$ in. of the specimen in order to make a reading.

4.8. Wattage Absorbed. This method has been used to measure the thickness of ferromagnetic sheets or tubes (boiler tubes) from one side. Devices of this type have a pole area of 1 by 0.5 in.; they are connected to the power line, and the wattage absorbed in eddy-current losses is measured by a wattmeter. The method is reportedly satisfactory with larger tubes of considerable wall thickness. For small tubes

with thin walls, the eddy-current energy at times is lower than the power consumed in the potential circuit of the wattmeter used. Changes of permeability, conductivity, or thermal history of the tubes have little influence. The average error for boiler tubes is reported as not exceeding ± 5 mils.

5.00. Optical Methods

5.1. Measuring Microscope [18, 167, 190]. The standard ASTM method of determining the thickness of coatings requires the use of the measuring microscope. The chemical dropping test and magnetic tests are alternatives. In ASTM 219, rules for use of the measuring microscope have been established by the American Electroplating Society cooperating with the National Bureau of Standards and the American Society for Testing Materials. This test is used for electrodeposited coatings of copper, nickel, chromium, lead, zinc, or cadmium thicker than 0.05 mil. The surface of the specimen is protected either by mounting in a plastic material such as phenolic or acrylic resin, or by electroplating it with a relatively thick coating of copper. A section produced by cutting perpendicular to the flat surface is ground and polished by regular metallographic methods, treated chemically, and/or illuminated in such a manner as to obtain maximum contrast between the coating and the adjacent materials. The thickness is measured with a filar micrometer ocular that has been selected and calibrated so that the error will not exceed 2 percent. As an example, the scale may be graduated to 0.01 mm (0.4 mil).

Alternatively, the image of the specimen may be projected at a known magnification on a ground-glass focusing plane in the camera of a metallographic microscope, where the width of the projected edge of the coating is measured with a graduated linear scale. Magnifications of 500 are feasible.

When measuring the thickness of soft materials, like paper, with a micrometer microscope, the material must be embedded in resin or a similar substance [190, p. 4, 5]. Although the micrometer microscope discloses details of the cross section, it is very difficult to get accurate results because only the edge is measured, and not anything like the average thickness or the local thickness of a well-defined area of the sheet.

5.2. Change of Focus [64, 196, 208] (figs. 114, 115, 116, 117, 118, 119, 120). This method is used to measure the thickness of large sheets of transparent material at the middle of the sheet, where mechanical micrometers cannot be used, or to measure the wall thickness of glass bulbs, accessible from only one side. Some transparent materials are so soft that the pres-

sure of the anvil of the conventional mechanical micrometer deforms or damages them. The method is also used to measure the thickness of transparent coatings on an opaque base.

A microscope is used that can be adjusted with a micrometer screw to focus successively on the bottom and top surface of the sheet under test. With this method a focus discrimination of 0.2 mil is possible. The lower end of the microscope tube is held to the surface of the sheet. This is the zero mark on the micrometer scale. The transparent sheet is marked on the side away from the microscope with a soft pencil which leaves graphite particles that can be focused sharply in the microscope.

The readings on the barrel and thimble of this optical micrometer give the thickness of the sheet directly if the scale is calibrated for a certain refractive index n of the material under test. If the displacement of the microscope lens is h , the thickness is $x=n \cdot h$. Because most optical materials in common use have refractive indices of 1.520 ± 0.015 (a variation of about 1 percent), such instruments are generally calibrated for this value. Figures 114 and 115 show micrometers of this type.



FIGURE 114. Aireon optical micrometer.



FIGURE 115. "Opti-Mike."
Instrument Development Labs.

"Optimike." The "Optimike," figure 115, has a built-in lamp, connected with two flashlight cells. The measuring range of this instrument is 400 mils; the accuracy is given as ± 1 mil.

Manufacturer: Instrument Development Labs., Needham Heights, Mass.

Schneider instrument. The principle is used in an instrument built in Germany to measure the wall thickness of inaccessible but hollow transparent bodies. For measuring rough and optically bad surfaces, an immersion attachment is supplied with the instrument.

Manufacturer: Dr. Schneider Instrument Works, Bad Kreuznach, Germany.

The method has also been used to measure the thickness of transparent coatings of aluminum oxide, or to determine the thickness of glass mirrors. In this case the upper surface is marked with a graphite or grease pencil; the base metal of the mirror or the coating is easily recognizable from its texture by using a magnification of 500 to 1,000.

Thicknesses of very thin metal coatings on glass have also been determined in this way (fig. 116). The metal film is removed from a part of the glass plate. A beam of light from the lamp, B, (fig. 118) going through the lenses L_1 and L_2 and the filter F, projects a virtual image G of the grid G very near to the surface O_1 under test. The virtual reflections G'_1 and G'_2 are photographed through the microscope M_1 . The method has been used to determine

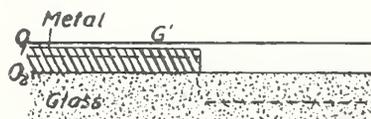


FIGURE 116. Measuring thickness of transparent films.

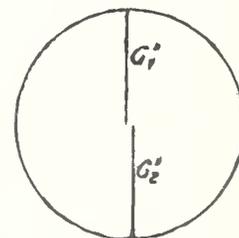


FIGURE 117. Mirror images of a line.

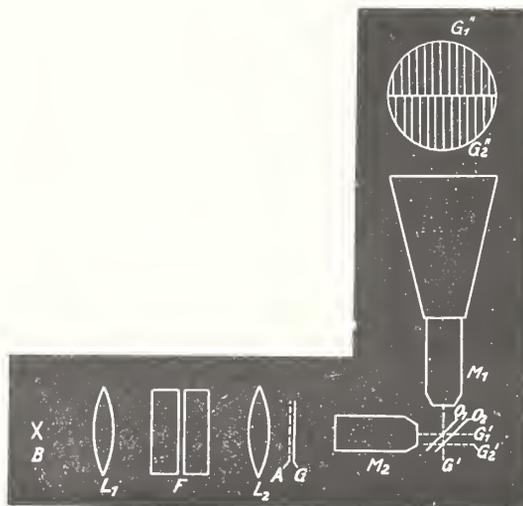


FIGURE 118. Arrangement to measure film thickness.

silver coatings 0.10 to 0.15 μ (4 to 6 μ in.) thick. The average error of 160 measurements was $\pm 0.008 \mu$.

Bausch & Lomb instrument. Bausch & Lomb Optical Co. has also used this principle in a thickness meter for glass (figs. 119, 120). The light source L projects an image of the graduated glass scale by means of the lens M on the first surface A' of the object (such as the glass bulb shown) when held against the small elongated aperture O, which is at an angle of 45° to the beam of light. The outer surface A' will reflect the image of the graduated scale S through the lens B and the prism P to the frosted screen Z. Only part of the light is reflected by the outer surface. The balance is refracted into the transparent material and will fall on the second surface A², be partially re-



FIGURE 119. The two scales as reflected in the eyepiece when measuring glass approximately 18 mils thick.

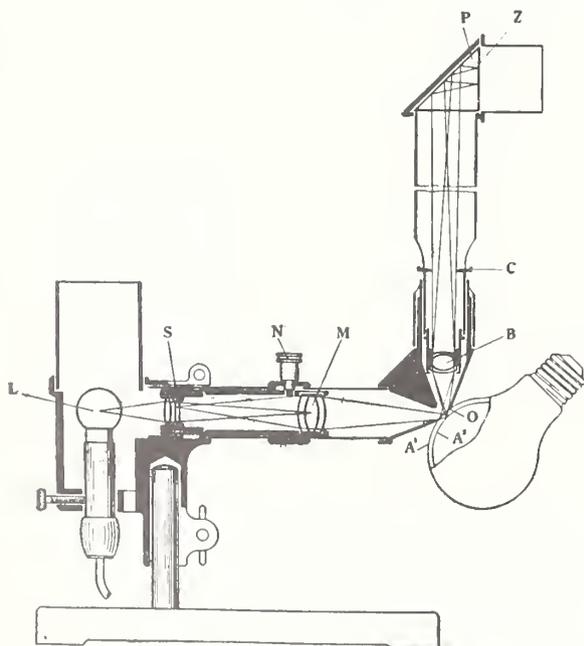


FIGURE 120. Optical system of Bausch & Lomb thickness meter.

flected to A and from there refracted into the air, giving finally a second picture of the scale S on the screen Z. The pictures are of almost equal light intensity, but the separation of the two images is proportional to the separation of the two glass surfaces, and can be measured directly by counting the divisions displacement. In the illustration the thickness is 0.018 in. The instrument is calibrated for the standard refraction index of glass, $N=1.522$. Readings on other glasses have to be corrected according to the refractive index.

5.3. Interference [128, 159]. The thickness of extremely thin reflecting metallic films is difficult to measure. The older methods of determining film thickness of the order of 1 to 0.01 wavelength of light have usually involved weighing or chemical determination of the quantity of material on a known area. These methods are difficult to apply because of the small quantities involved, and they suffer from the defect that the density of tenuous films (of atomic or molecular dimension) must be assumed to be that of solid metal. The interference methods described in this section provide measurements free from assumptions of density with precision of the order of 10 A (approximately 0.04 μ in.).

5.31. GE Spectrophotometer [2] (fig. 121). Spectrophotometers are used to measure the thickness of transparent films, e.g., electrolytic oxide coatings on aluminum. The recording spectrophotometer (General Electric Co.) projects progressively a series of monochromatic polarized beams of light, ranging from 400 to 700 $m\mu$, onto a sample coated with the film under test, placed over an aperture of 1 in. diameter in a small sphere. The reflectance or transmission of the sample is automatically recorded as a function of the wavelength. The reflected light obeys the laws of interference (fringes of equal chromatic order), and the curve traced by the recording spectrophotometer has variations related to the thickness and refractive index of the film (fig. 121). The refractive index must be

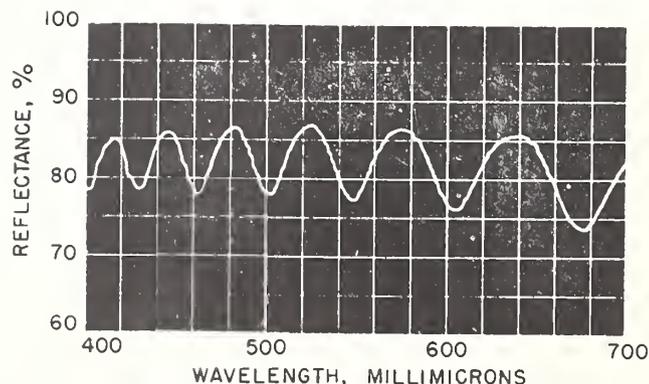


FIGURE 121. Reflectance curve for "Alzak" reflector sheet with commercially thin oxide coating.

determined separately. The thickness of the film is determined by the number of maxima between two wavelengths.

5.32. Michelson Interferometer [58]. This well-known instrument can be used to measure thickness of thin metallic films. One of the total reflecting plates of the interferometer is first coated with an opaque film of the metal by the usual evaporation process. Then, a central region, approximately one-third of the total reflecting area, is covered with a mica strip and the remaining unobstructed area is given an additional deposit of the same metal, when the film is evaporated in production. The mica strip has to be thin and in very close contact with the interferometer plate. The plate, with the mica strip removed, is placed in the interferometer and the instrument adjusted for white light fringes. A monochromatic source is then substituted, and the two fringe patterns compared. In the central region the fringes are displaced relative to the fringes in the outer portions. For a displacement of one fringe the thickness of the film deposited during the second evaporation is half the wavelength of the light used; for fractional displacements, corresponding fractions of the half wavelength. When the customary green mercury line is used for measurements, the fringe displacements for a film $20\text{ m}\mu$ thick is less than one-thirteenth of a fringe. The fringes are sharp enough, however, for comparator measurements of displacement, or even for projection on a screen with subsequent measurement. Both methods have been used with considerable accuracy.

5.33. Interference Microscope [198]. The interference microscope has been found useful in measuring the thickness of decorative chromium plating. In order to use this method, a part of the coating must be stripped from the piece to be tested by suitable means leaving a distinct boundary between the coated and stripped portions. It is then placed under the interference microscope and brought into focus. Focusing in the interference microscope is much like an ordinary microscope. When the enlarged, focused image of the surface is visible in the eyepiece, a shutter is opened which allows a series of parallel light and dark bands to be superimposed on the image of the surface. The spacing and direction of the fringes can be adjusted according to need. In boundary measurements such as the one under consideration, the fringes should be perpendicular to the boundary. A fringe shift results from any surface irregularity, the amount of the shift being determined by the depth of the irregularity. A shift of one bandwidth is equal to a depth of one-half the wavelength of the light used, usually the 5460-A green line of mercury. The range of the instrument is from 2 to $100\text{ }\mu\text{in.}$ The method is considered to be extremely accurate.



FIGURE 122. Step gage to measure thickness of molecular films.

5.34. Color Comparison [33, 34] (fig. 122). This method is based on the comparison of interference colors reflected from white light by the film under test [33] and colors reflected from a glass surface coated with films of various thickness.

The GE step gage [34] is a plate of special glass enclosed in a plastic case (fig. 122). A film of barium stearate is deposited on the glass in a series of monomolecular layers, deposited in such a way as to build a series of steps each $1\text{ }\mu\text{in.}$ thicker than the preceding one. When illuminated by white light, each step reflects a color determined by its thickness. The steps appear as a series of colored strips. The range is from 2 to $16\text{ }\mu\text{in.}$ The readings have to be corrected for the refractive index of the plate on which the unknown film is deposited. The procedure of correction is different from materials with lower or higher refractive index than the film.

The many uses of the step gage are described in the GE instruction book, GEJ-2329 "Barium Stearate Stepgauge."

5.35. Donaldson-Khamsavi Method [69, 202] (fig. 123). (The material presented here is taken from the book, "Multiple beam interferometry," by S. Tolansky [202]). In the multiple-beam method of W. K. Donaldson and A. Khamsavi, the thin film to be measured (AB in fig. 123) is deposited on part of a flat glass surface, ABC. A fairly thick opaque coating of silver, PQRS, is then deposited by evaporation over both film and flat. The height, QR, then equals that of the silver film AB. A silvered flat, DE (reflectivity=0.90), is brought close to the combination of AC. When illuminated from

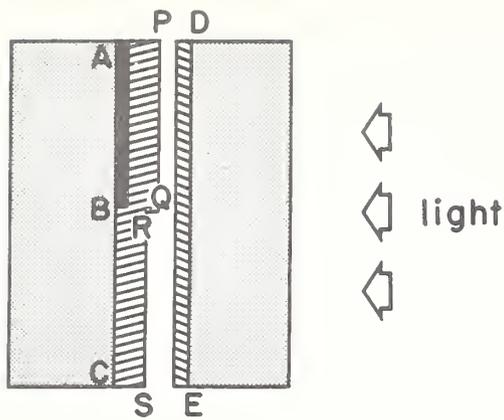


FIGURE 123. Donaldson-Khamsavi method for very thin reflecting metallic film.

the right, back-reflected Fizeau fringes are formed which exhibit the sharpness characteristic of multiple beam interference. The step displacement, RQ, can thus be determined with a precision down to the order of 10 \AA (10^{-6} mm).

It should be noted that the true metrical thickness of the film is obtained. The opacity of the silver coating PQRS automatically eliminates phase-change effects that would vitiate results if transmission fringes were used. It is not sound practice to make the coating film PQRS sufficiently thin to permit of transmission observations, for then the phase-change effect at the combination PQ may differ in an unknown manner from that at the simple film RS.

The film AB must be capable of withstanding the vacuum necessary for evaporation of the superposed coating of silver.

5.36. Fizeau Method [101]. This method, devised by Fizeau, is used to determine the thickness of silver film on glass, e.g., on mirrors. The film in question is supported 1 or 2 mm above a small iodine crystal. After a few minutes, the iodine vapor will react with the silver, thus forming a spot of transparent silver iodide. The area surrounding the transparent spot will be partially converted to silver iodide and will appear, when viewed by reflected light, as a series of concentric, colored rings. The number of these rings is a measure of the thickness of the silver iodide, hence of the silver film. A table is available which correlates the number of rings with the film thickness [101]. It is essential that the film be exposed to the iodine until transparency results. A longer exposure will enlarge the pattern, but will not increase the number of rings. The range of thickness usually varies from about 0.03μ to approximately 0.2μ .

5.4. Diffraction [126, 171, 173, 143a]. This method has been used to measure the diameter of fine wires. If a narrow beam of light is directed against an obstacle, the dimensions of which are not much more than the wave length of the light, interference fringes develop in the

space behind the object. For example, diffraction patterns from a needle are parallel bright and dark lines, visible under magnification, which broaden towards the tip of the needle. The smaller the object, the wider the spacing of these lines. On this principle, a "diffraction micrometer" has been developed by the Dr. Carl Leiss Co. in Berlin-Steglitz in cooperation with the Osram Lamp Co. The method can be used for wires from 0.01 to 0.08 mm in diameter; the accuracy is given as about ± 0.3 percent of the actual diameter (not of the maximum range).

The diffraction of light by a bundle of parallel fibers was employed by Thomas Young in 1824 in a simple, ingenious device (the eriometer) for the rapid, direct measurement of average diameter. McNicholas and Curtis [138a] made a new construction of Young's instrument and applied the method to textile fibers. The eriometer average diameter is in excellent agreement with comparable data obtained with the microscope.

5.5. Light Beam to Photocell [88, 118, 165, 181, 194, 218] (figs. 121, 124, 125). Optical-photoelectric thickness meters do not directly contact the object to be measured. Instead, a thickness or diameter influences the light quantity falling on a photocell. The method is not ordinarily used to measure the thickness of sheets during the process of production, but rather to measure the thickness of fine wires or, in the textile industry, fibers, threads, yarns, and slivers. In all such devices care is taken to see that variations in the light source do not influence the reading.

5.51. "Evenometer" [88]. This instrument is used primarily to determine unevenness (variation of diameter) of raw silk thread which is wound at a spacing of 100 threads per inch around a slotted board. A beam of light is directed to the space where the silk is over the slot and its intensity is measured in a differential arrangement. Two recordings are made, one of the average diameter, the other (by means of an integration circuit) of the deviation from the standard diameter.

5.52. "Filometer" [194]. This instrument developed by the Deering Milliken Research Trust in Pendleton, S. C., is similar in principle to the Evenometer, but does not use the shadow method, which may give erroneous results when the material has a tendency to band, ribbon, or twist, causing an apparent change of diameter. The Filometer employs a high-intensity beam of light, so arranged that light is actually transmitted *through* the material. In this way the change of light is a measure of the density of the material and not of its thickness alone. Different standards are required for colored yarn. The recorder has two pens, one recording the momentary thickness, the other recording long-term variations of average thickness.

5.53. "Serc" Evenness Tester [192], *Electron Micrometer*, and "Diamatrol" (fig. 124). These instruments are all operated on the optical-photoelectric principle. A beam of light from one source is split into two balanced equal halves, M and R (fig. 124). These beams fall on two matched photocells, P_1 and P_2 , whose amplified outputs are opposed to each other, so that the total output will be zero. The yarn or wire of known diameter, say 20 mils, is placed in beam R and the corresponding photocell P_2 will receive less light. An indicating meter connected to the difference voltage will now register a certain unbalance. With a knife edge, adjustable with a micrometer screw, the amount of light in the second beam M is reduced until it equals the intensity of the beam obstructed by the 20-mil yarn and the indicator or recorder reads zero again. This position of the micrometer is marked "20 mils."

When production yarn uniformly 20 mils in diameter runs through the meter the indicator will read zero. It will show a plus indication if the yarn is larger than 20 mils, minus for thinner yarn. The electrical meter can be calibrated in mils deviation, plus or minus, from 20 mils normal diameter. The scale of the micrometer, which adjusts the knife edge in beam M, measures the base diameter, while the electric indicator indicates the deviation from the base diameter. The scale of the indicator has a length of ± 4 in. and can be calibrated to read ± 4 mils, to give a magnification of 1,000. Special designs of the instrument have full scale deflection for ± 0.5 mils, equivalent to a magnification of 8,000. To prolong its age, the 6-v lamp is operated at only 3.2 v. Change of the light intensity caused by voltage fluctuations will cause the same percentage change of reading of deviation from base diameter. A method of compensating for changes in either amplifier or photocell is to use only one amplifier and one photocell to which both light intensities are

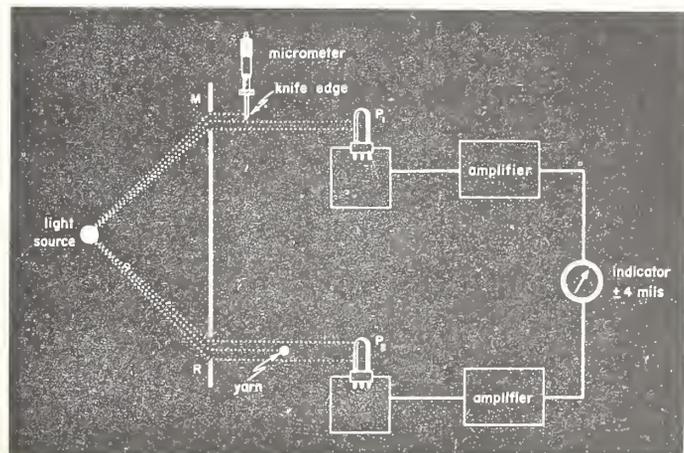


FIGURE 124. Principle of the yarn-diameter indicator of Standard Electronics Research Corp.

alternately directed. A rotating disk scans the two light beams alternately and generates two alternating voltages with 180 degrees phase displacement. The difference of these voltages is indicated by the meter.

Line-voltage fluctuations up to ± 5 percent, which cause light fluctuations of about ± 15 percent, can be compensated by another electronic circuit. For wider line changes, constant voltage transformers have to be used. The readings of these optical-electrical thickness meters are unaffected by the composition or specific gravity of the filament under test, or by its water content.

Manufacturer: Standard Electronic Research Corp., New York City.

5.54. "Visi-Limit" Gage [118, 218] (fig. 125). This instrument operates with a differential system, using only one beam of light, which compares the photoelectric effects generated by the sample with the effects of specimens representing high and low limits.

Figure 125 shows the basic principle, applied to measuring the thickness of a wire. There is one light source, a scanning disk of special design, a frame with three apertures, a photocell, and an amplifier. The three apertures are illuminated in sequence by the rotating disk. The wire under test is behind the middle aperture. The light coming through the middle aperture is reduced with increasing thickness of the wire, the illuminated area of the photocell becomes smaller and with it the photocurrent. The other two apertures are adjusted to reproduce production limits, the high-limit aperture corresponding to a test wire of maximum diameter, the low limit aperture to one of minimum diameter.

The scanning disk illuminates the three apertures in such a way that the total area uncovered at any time is constant. Three a-c voltages are generated, fed to an a-c amplifier, and from there to the vertical deflection plates of a 5-in. cathode-ray oscilloscope. All three voltages appear at once in the oscilloscope as three horizontal lines. Although the voltage is connected only for one-third of each revolution of the scanning disk, the picture is persistent for the observer. The thickness indicated by the length of the middle line has to be within the

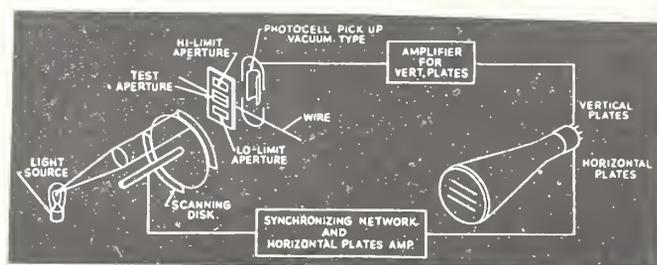


FIGURE 125a. Schematic diagram of the "Visi-limit" gage.

6.00. X-ray Methods

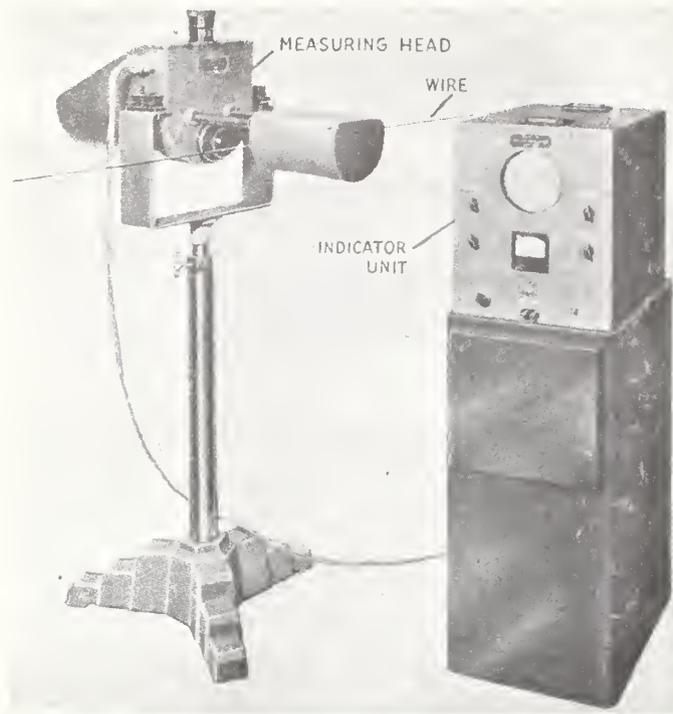


FIGURE 125b. "Visi-limit" micrometer, measuring head, and indicator unit, as used to measure wire thickness.

limits given by the lengths of top and bottom lines.

The magnification of the deviation from standard thickness can be adjusted up to 4,000, so that one-half mil is represented by 2 in. The resolution is constant and is given as 0.1 mil, independent of the total dimensions of the test object. The wire may vibrate as much as 400 mils. In the absence of vibration the accuracy is given as ± 0.1 mil.

An adaptation of the "Visi-Limit" gage to measure the thickness of sheets belongs with the mechanical-optical-photoelectric methods. The sheet is passed over a roller, and under a straightedge placed at a known distance above the roller. The light beam passes through the space between the top of the sheet and the straightedge, forming the test aperture. The limit apertures are adjusted to the allowed tolerances.

Manufacturer: Raymond Wilmotte Co., Washington, D. C.

5.6. Electron Microscope [139]. The electron microscope has been widely used since 1939, to measure the thickness of very fine fibers or films of the order of 0.1μ ($4 \mu\text{in.}$). New methods of measurement and of calibration with objects of known diameter have been developed here and abroad. Mahl [139] gives 36 literature references. By way of stating the limits of the electron microscope, it may be said that an object 0.02μ thick ($0.8 \mu\text{in.}$) can be measured with an accuracy of about 0.005μ ($0.2 \mu\text{in.}$).

6.1. Shadow Geometry [90] (pat. 1) (fig. 126). This method is used to determine the thickness of the walls of large vessels, especially the hulls of ships, where both sides are accessible but not in such a way that a gaging tool could be used. One of several test procedures is the following [90] (fig. 126): Two straight tungsten wires are cemented on the outside of the hull, about 4 in. apart. An X-ray tube with a very sharp focus is located on the same side about 8 in. from the surface. On the inside of the hull a photographic film is placed in close contact with the wall, the correct location being determined by means of an electromagnet on the outside and a compass needle on the inside. When the focal distance a and the spacing b of the wires are known, the unknown thickness of the wall may be calculated as $d = (ac/b) - a$, where c = the spacing of the shadows. Instead of using two wires at spacing b , two images of one wire may be secured by moving the X-ray tube parallel to the surface for two exposures.

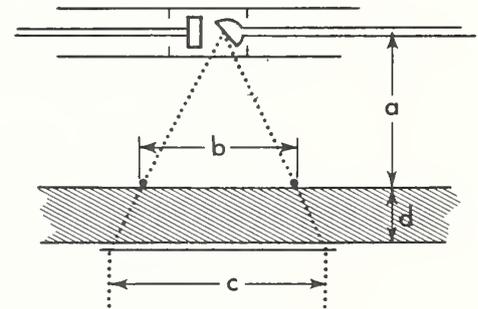


FIGURE 126. X-ray shadow method, using two tungsten wires in the distance "b".

6.2. Absorption, Photographic Comparison [148]. A set of blocks of known thickness of the same material as the test object, ground in steps of 20 mils over the full range of thickness likely to be encountered, is photographed simultaneously with the test object on X-ray film with fine grain and high contrast. In order to get the maximum contrast in the film, the minimum voltage capable of penetrating the object should be used. One application is the measurement of the thickness of hollow steel propeller blades. One-half of a film 7 by 17 in. is pressed by an inflated rubber bladder inside the propeller, the other half is placed under the test blocks.

6.3. Absorption, Radiation Detectors [55, 57, 66, 213, 219] (fig. 127, 128). This type of industrial thickness meter has received increasing use in the past 10 years, especially for measuring the thickness of continuously running sheets. With the advent of radioactive isotopes,

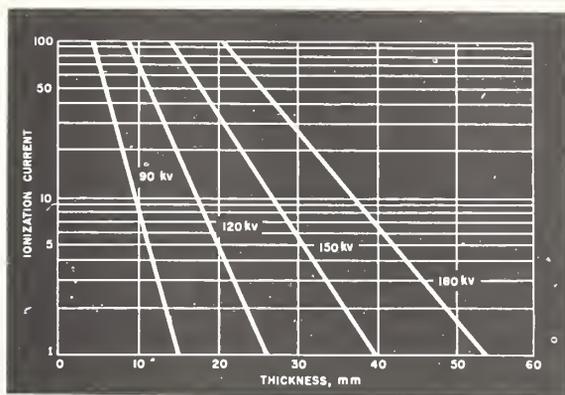


FIGURE 127. Ionization current as a function of material thickness and X-ray voltage.

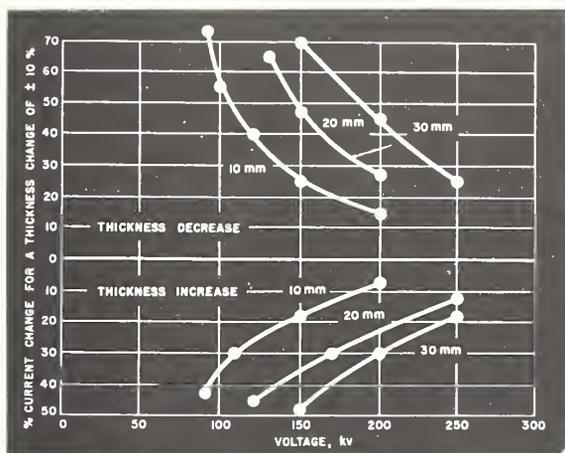


FIGURE 128. Current change for a thickness change of ± 10 percent for steel plates 10, 20, and 30 mm thick, as a function of voltage.

however, it has lost some ground. The basic principle is the following: When material of unknown thickness (of any kind, but generally metallic) is placed between an X-ray tube and a radiation indicator, a part of the radiation is absorbed. The decrease of intensity is an indication of the energy absorbed and depends on both material and thickness.

Two different effects are used for measuring radiation: (a) gas ionization in ionization chambers or Geiger-Mueller counters, (b) fluorescence of a screen, measured by a light-intensity meter. The first X-ray thickness meters used ionization chambers of old design, measuring intensity with slow moving-coil instruments of high sensitivity. When suitable amplifiers were developed it became possible to use for low-intensity radiation the newer ionization chamber and Geiger counters with high-speed response needed for measurements on sheets moving with a speed up to 30 mph.

When a fluorescent screen is exposed to X-rays, high-speed photoelectrons are liberated. Since the invention of the photomultiplier tube, this detector is used more and more. Its glass envelope is mounted near a fluorescent screen of zinc-cadmium sulfide or other materials with

high response speed, and is covered with black paper to exclude undesired light.

Radiation thickness meters have the great advantage that the material is not in contact with any mechanical member. They can be used when the strips are not only moving fast in a longitudinal direction, but also vibrating transversely. One design allows a transverse movement of the strip up to 18 in. In contrast to absorption meters using visible light, X-ray meters are not affected by steam or dust developed in rolling mills. Even layers of cooling water 1/16 in. thick give not more than ± 1 percent error. These meters do not give absolute measurements; they have to be calibrated with standard samples. The purpose of the different designs is to eliminate as far as possible the effects of variable voltage and of changes in the emission of the X-ray tube.

In principle, X-ray thickness gages are very sensitive, 1-percent thickness change corresponding to 5- to 10-percent change of transmitted energy. It is necessary, however, to use the proper type of X-rays for each test: Soft rays for thin or slightly absorbent material like paper, plastics, or foils, and hard rays generated with high-voltage tubes for thick material. Figure 127 shows ionization current in the detector as a function of material thickness and X-ray voltage. If we plot from this the sensitivity (percentage current change for 10-percent thickness change) we get figure 128. From this it is evident that the lower the voltage the higher the sensitivity, but of course there are limits to the sensitivity of the detectors and the stability of the circuits. Low X-ray voltage is also desirable for reasons of safety and cost.

It is important to bring both the tube and the radiation receiver as close as possible to the object under test, though it may be desirable to allow the material, if coming from a rolling mill, to vibrate transversely as already mentioned. When testing unevenness of the wall thickness of containers, tubes, or bottles, it is of the greatest importance to keep the area under test as small as possible, in order to find minor flaws at the same time. On the other hand, for measuring thickness of sheets in production, an averaging effect is desirable.

6.31. Single Tube and Beam [203, 213]. In an early use of this method [203] steel sheets 2 in. thick were tested with 200 kv in a 2-in.² area, using a Geiger-Mueller counter as the pickup. At the maximum sensitivity the response time was 0.1 sec for an instrument used with an active area of about 0.3 in.²

The modern "Measuray" [213] also uses only one tube and one beam. The X-ray tube is contained within a grounded housing in the lower part of the unit, and the pickup, a photocell, may be as much as 30 in. above the base.

Manufacturer: Sheffield Corp., Dayton, Ohio.

6.32. *Absorption Differential* [83, 84, 98, 137, 140, 219] (fig. 129, 130). The purpose of all differential methods is to obtain higher accuracy, independent of fluctuations of the supply voltage. This is done by comparing the absorption of two beams of X-rays in a specimen of standard thickness and in the sample under test. Three schemes are possible: (a) one tube alternately irradiating the standard and the unknown, (b) one tube with a double window to split the emission into two parts, one directed to a standard thickness, the other to the test object, and (c) two tubes, operating under identical conditions. All these instruments apply mainly to the measurement of the deviation from the standard value. The accuracy at the center of the scale is almost the same as the accuracy to which the thickness of the standard has been determined.

For the first method [137] one X-ray tube is used with two ports. Two fluorescent screens are mounted so as to receive the transmitted radiation from the test and the reference object. Visible light from the fluorescent material

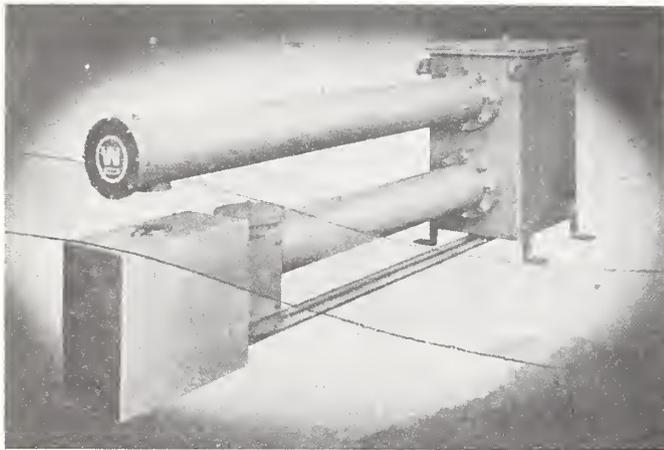


FIGURE 129. *Westinghouse X-ray thickness gage for cold-rolled steel strip.*

is interrupted by a rotating shutter which allows the two beams to impinge alternately upon a photocell. A commutator mounted on the rotating shutter provides phasing information for an algebraic rectifier.

The second method [137, 98] involves the use of two pickup devices. The radiation pulses appear simultaneously at the output of both pickups. The difference indicates the thickness difference between reference and test material. Measuring ranges of this type of thickness meter are given by GE as follows:

Material	50 kv		100 kv	
	<i>Mils</i>		<i>Mils</i>	
Lead.....	1 to	6	6 to	31
Copper.....	3 to	25	25 to	115
Mild steel.....	^a 5 to	40	^b 40 to	190
Brass.....	5 to	25	25 to	110
Glass (hot).....	60 to	500
Aluminum.....	80 to	500	200 to	1,000
Rubber.....	187 to	1,000

^aCold strip. ^bHot strip.

The third method [140, 219] uses two sources of X-rays and one pickup device. The two tubes are so connected that they operate 180 deg out of phase, so that pulses from each source arrive alternately at the common pickup. This method is used for the Westinghouse cold-rolled-strip-steel thickness gage (figs. 129, 130). The work X-ray tube, shown above the pass line, directs its beam downward through the material to be tested. The standard X-ray tube, shown below and to the left of the pass line, directs its beam horizontally through a standard sample. The transmitted radiation in both instances strikes the pickup unit. The indicator has a scale length of ± 3 in. with ± 10 divisions on each side of center, to give the deviation of the rolled sheet from the thickness of the standard sample. This is part of a calibration device, and it can be

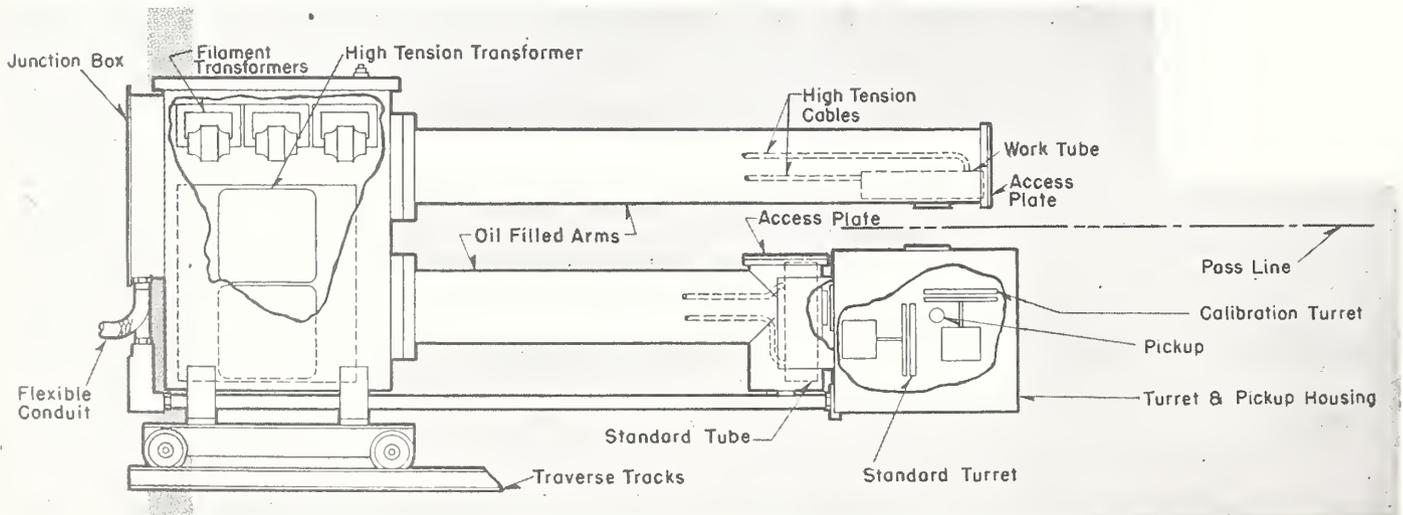


FIGURE 130. *Schematic view of Westinghouse X-ray thickness gage.*

adjusted in steps of 1 mil from 5 to 119 mils total thickness. One range of the instrument indicates ± 1 mil for all standard thicknesses from 5 to 49.9 mils. In this way the full deflection of 3 in. on the meter corresponds to ± 20 percent deviation in 5 mils, and one division 300 mils wide corresponds to ± 2 percent or 0.1-mil deviation, a magnification of 3,000. For a standard thickness of 49.9 mils, the full scale of ± 1 mil gives ± 2 percent or 0.1 mil, the magnification being 300. The second range of the instrument is ± 3 mils, for standard thicknesses from 50 to 119 mils, so that one division of the scale corresponds to 0.3-mil thickness difference, which is ± 0.6 percent of the low limit of 50 mils and ± 0.25 percent of the high limit of 119 mils for sheet thickness.

In the X-ray absorption gage of the Standard Electronics Research Corp. in New York City, developed to detect voids in artillery shells, [83, 84], the X-rays, after passing through the part under test, strike a pair of potassium iodide scintillation crystals one-eighth in. square, which emit visible light approximately proportional to X-ray intensity. The light from the crystals goes to the cathodes of secondary emission phototubes, whose output is amplified and applied to a d-c meter to indicate the difference of thickness. The device is very sensitive; a thickness difference of 0.5 mil in a sheet 250 mils thick, or a change of one part in 500, can be detected. The X-ray beams, obtained with 180 peak kv and 4 ma, have a diameter of three-sixteenths in. (190 mils).

Manufacturers of X-ray gages: General Electric Co., Schenectady, N. Y.; Industrial Gages Corp., Englewood, N. J.; Sheffield Corp., Dayton, Ohio; Standard Electric Research Corp., New York, N. Y.; and Westinghouse Co., Pittsburgh, Pa.

6.33. Special Varieties of X-ray Gages [127, 214]. In the manufacture of steel bottles for highly compressed gases, uniformity of wall thickness and freedom from flaws are very important. In testing the bottles the detector is inserted inside the bottle or tube by means of a cable or rod of appropriate length; the X-ray source is outside. The tube or bottle is rotated. At the same time, either the X-ray tube with the detector or the bottle alone is moved longitudinally so that the whole surface is inspected. The thickness is read on an indicating instrument. High speed is very important for this kind of testing. For large objects it may be desirable to have a recording of the uniformity of thickness, providing legal evidence of the test and saving the operator's time. A facsimile recorder [127] can be used with the intensity of the stylus marks proportional to the signal picked up by the detector. The bottle and the drum of the recorder are rotated in synchronism; likewise, the axial movement of the bottle

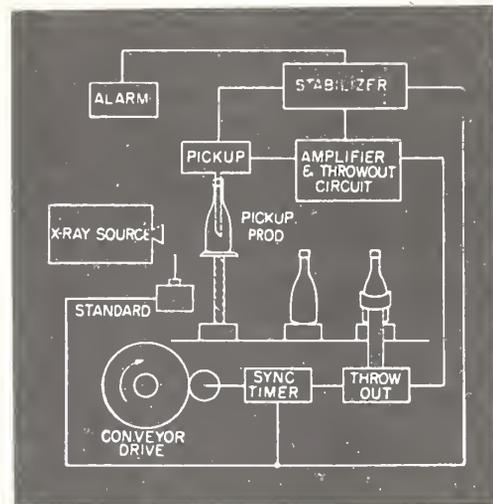


FIGURE 131. Measuring the thickness of glass bottles in a production line with a Westinghouse X-ray gage.

is synchronized with the axial movement of recorder pen or chart.

An X-ray gage to be inserted in glass containers [214] is shown in figure 131. The pickup consists of a photocell whose housing carries a small-diameter brass tube which can pass through the neck of the bottle. A fluorescent screen, mounted at the tube end at a 60° angle, is excited by the X-ray source and the pattern is sensed by the photocell. The effect of the bottle motion is that a point on the container describes a helix and the entire surface of the bottle wall is scanned. If the wall thickness is less than a predetermined value, the gaging circuits operate a mechanism to discard the defective container.

Manufacturer: Westinghouse Electric Corp., Pittsburgh 30, Pa.

6.4. Diffraction [32, 74, 95]. This method has been used to measure the thickness of electrodeposited crystalline films. Diffraction patterns of thin surface films are composed of diffraction lines from both the underlying base material and the surface layer. A comparison of the integrated intensities of the two sets of diffraction lines with a microphotometer can be used as a measure of the surface-film thickness. The theoretical investigation, made for both flat and cylindrical surfaces, was confirmed experimentally on nickel wires of 3-mm diameter, coated with barium carbonate and strontium carbonate. The usable range of the method is indicated as 0.01 to 500 μ . The experimental investigation covered the range from 1 to 60 μ [74].

In another system of measuring thin films on thick crystalline or polycrystalline backings, characteristic X-rays are reflected from the base material at one of the Bragg diffraction angles and the diffracted intensity is measured by a Geiger counter system [32, 95]. When the base material is covered by a thin coating, the

X-rays are reduced in intensity according to an exponential law. From the measured ratio of the coated and uncoated intensities, the geometry of the arrangement, and the known absorption coefficient for the coating, it is possible to compute the thickness of the coating. Measurements are made with a powder-diffraction spectrometer utilizing the focusing principle. The area tested is between 1 and 100 mm². Thickness was measured between 0.1 and 100 μ . The method is applicable to coatings of any material or combination of materials whose X-ray absorption coefficients are known.

The method has also been used to *identify* contaminating films less than 0.01 μ thick on electrical contacts without measuring their thickness.

6.5. Backscattering, Secondary Radiation [28, 29, 158, 205] (figs. 132, 133, 134). This technique measures the thickness of metallic coatings on a base metal. A device used by the canning industry to measure the thickness of a tin coating on hot dipped steel is used here as an example [158, 205]. A 20-kev X-ray beam is directed at an angle of 70 deg upon the surface of the sheet (figs. 132, 133), penetrating the tin coating and causing the underlying metal to emit secondary fluorescent radiation in all directions. The intensities are measured with Geiger counters during a 30-sec interval. The maximum thickness measurable is 3 lb per base box, which means 3 lb of tin on both sides of 112 sheets, each 12 by 20 in. With a count of 500 impulses per interval for uncoated sheets, the count was reduced to 200 for sheets with a 1-lb coating, 60 for sheets with a 2-lb coating, and 20 for sheets with a 3-lb coating.

The design shown in figure 134 uses two X-ray heads to measure the thickness of the

plating on both sides of the sheet at the same time. The test area is 4 in.²; the sheet is measured in 12 spots by moving it by hand under the detector assembly. The equipment is also used for checking the plating thickness in continuous production, using averaging circuits for the 30-sec measuring period. The count can be indicated (usually scaled down in multiples of 2) or recorded, either graphically or digitally. The range for tin coatings is from 0 to 200 μ in., too small for magnetic or electromagnetic methods. The accuracy is given as 2 percent of the measured thickness, which is about the same as is



FIGURE 133.

Measuring head consists of the X-ray tube (at top) and a lead-lined brass collimating tube designed to produce uniform radiation over a circular area of 4 in.² The Geiger tube at right measures the secondary emission after it has been reflected back through the tin coating at an oblique angle.

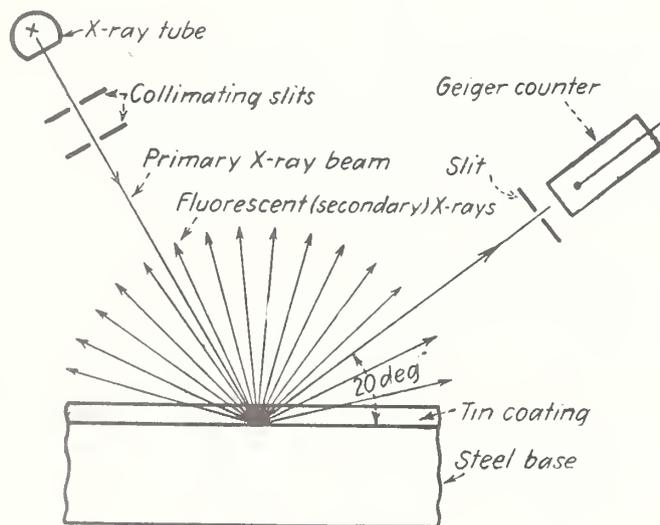


FIGURE 132.

Secondary fluorescent emission at the iron-tin boundary is measured from an acute angle; absorption by the tin coating reduces the intensity in proportion to the thickness of the tin coating.

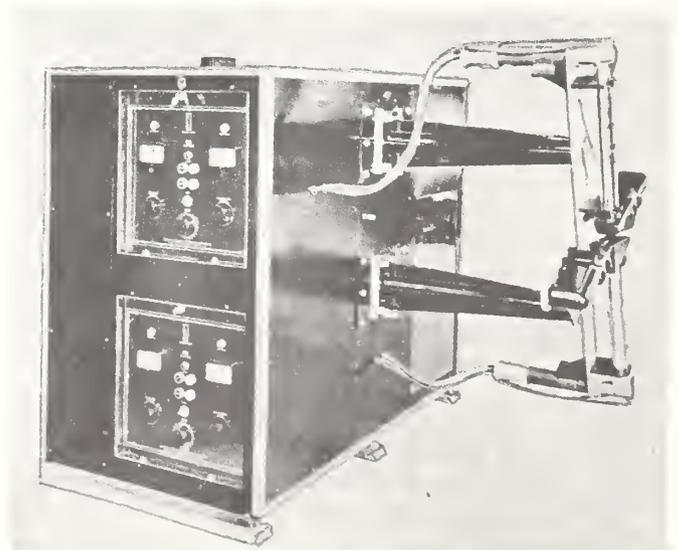


FIGURE 134. X-ray tin coating thickness meter.

Twin equipment, to measure the thickness of the coatings on both sides of the plate.

obtainable with the chemical method used for calibration [29].

The method can also be used to determine the thickness of metallic coatings on metals other than steel. It is, however, necessary that the coating material have an appreciably higher atomic number than the base metal. There are limits for both the maximum and the minimum thickness measurable: Very thick layers absorb all radiation before it can enter the counter, very thin coatings cause too little change compared with unplated material.

A similar method has been developed by H. F. Beegley, Jones & Laughlin Corp. [28].

Manufacturer: North American Philips Co., Mount Vernon, N. Y.

6.6. Spectrometry, For Depth of Cold-Working [87] (fig. 135). When the surface of a metal is cold-worked, as by sandblasting, the crystal structure of a thin layer is altered [87]. Figure 135 shows a special fixture, used to position a flat specimen at the Bragg angle and also to rotate the specimen about an axis normal to its flat surface, so that the entire surface is scanned. The output of the Geiger counter varies as the specimen is rotated. The variation of intensity recorded by the potentiometer is a function of the grain size of the specimen. Small grain gives small intensity variation, large grain gives large intensity variation. The method is most sensitive to grain sizes larger than 1μ .

After a first recording of the X-ray spectrogram under slow rotation of the specimen, the surface is etched to remove a known thickness of the cold-worked layer, after which a new spectrogram is taken. This is repeated until the variation of the intensity from the Geiger counter becomes negligible.

The advantage of this method is said to be in the higher speed of testing. Using five etching steps, only $1\frac{1}{2}$ hours were needed, compared with 10 hours when determining cold-work thickness by X-ray diffraction photographs.

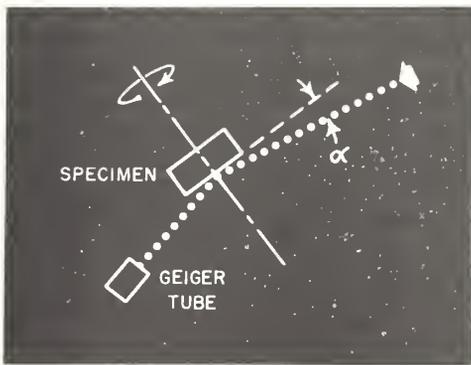


FIGURE 135. Measuring cold-work thickness with the X-ray spectrometer.

Nuclear radiations useful for measurement of thickness include alpha rays, beta rays, gamma rays, and neutrons [24, 48, 67, 93, 163, 184, 193]. The different radiations vary in ability to penetrate materials. Many devices are now available to measure the amount of radiation transmitted, absorbed, or reflected. These measurements can be interpreted in terms of the thickness of the material irradiated.

At this writing alpha rays are not known to have been employed in thickness gages. Their limited penetration power restricts their use to only the thinnest of materials. (Their maximum range in air is only about 4 in.; a sheet of paper can stop them.)

Nuclear radiations find extensive use in applications where contact with the material being measured is undesirable; they are especially useful in measuring the thickness of sheet material, either metallic or nonmetallic, in production. Electrostatic charges on the moving sheets do not affect thickness measurements made with gamma radiation or neutrons.

Radioactive materials which are sources of nuclear radiations are either naturally occurring radioactive isotopes such as radium, thorium, uranium, and actinium, or artificially produced radioactive isotopes. The many artificially produced radioactive isotopes, which have recently become available through the extensive operation of nuclear reactors, have given impetus to many industrial measurement techniques based on their use.

Selection of Radioactive Material [48, 86, 152, 182, 193]. The choice of a radioactive source for a particular application is based on its half life and the type and energy of the emitted radiation. Half life is defined as the time required for the activity of the source to fall from an initial value to half that value. It is desirable that a radioactive source have a sufficiently long half life so that frequent recalibration of the instrument will not be necessary. On the other hand, long half life means a lower particle emission rate and consequently slower measurements.

Correction for emission loss is made in some modern industrial instruments by periodic standardization with a sample of known thickness. Other instruments avoid this by using a split beam of radiation and passing one part through air or a calibrated sample, the other through the material under test and comparing outputs.

Of the naturally occurring radioactive elements useful as sources of radiation for thickness measurement, radium has the longest half life, 1,600 years. This means that only 1 percent of its atoms will decay in 20 years. Of the artificially produced isotopes only carbon-14 has

a comparable half life, actually greater, of about 6,000 years. For industrial purposes, cobalt-60 is most widely used as a source of gamma rays. The emission of cobalt-60 corresponds very closely to the effective energy of a 2,000-kv X-ray machine. This isotope has a half life of 5.2 years and a specific activity of approximately 20 curies/cm³, 10 times that of the usual radium sources. As a result, the dimensions of a cobalt-60 source can be very small, thus giving very clear radiographic pictures. The cost is also lower than that of radium.

The type of radiation and its energy determine the nature of the reaction with matter. Alpha particles, being ionized helium atoms, consist of two protons and two neutrons. They have a positive charge equal numerically to twice the charge of a beta particle (high speed electron) and a mass 7,400 times that of a beta particle. Because of their relatively great mass and charge, alphas have very low powers of penetration.

The depth to which either an alpha or beta particle will penetrate a substance depends not only on the mass, charge, and energy of the incident particle, but also on the density of the medium. For a given path length, a medium's stopping power is given by the mass per unit area presented to the incident beam. As the incident particles pass through a medium, they collide and interact with its atoms causing pairs of ions to form. At each such ionization the incident particle loses energy. If the density and thickness of the medium are great enough, the particle will lose all of its energy and come to a stop.

Because of their lack of charge, gamma rays and neutrons do not interact with matter in the same way as do alpha and beta particles. Gamma rays are high-energy photons akin to X-rays but of shorter wavelength. In matter, they lose energy by producing secondary beta particles. They do this in any of three ways: (1) The photoelectric effect, where the gamma photon completely transfers its energy to a single existing electron which is then ejected. (2) The Compton effect, which is the result of an elastic collision. Part of the photon's energy is lost to an existing electron, and the photon of reduced energy and frequency bounces off at an angle, much like a particle. (3) Pair formation where the energy of the gamma photon must be greater than about 1 million electron volts (1 Mev) in order to penetrate the cloud of electrons surrounding the nucleus. In the field of the nucleus it loses all of its energy in the production of a "pair," one positive and one negative electron.

Neutrons react only with the nuclei of atoms. They effect their energy transfer either by elastic collision or by absorption. The result of an

elastic collision is direct transferal of energy. The result of neutron absorption is either (a) the emission of high energy alpha particles, (b) fission, (c) the emission of beta or gamma rays, or (d) some combination of a, b, and c.

For a given application, greatest sensitivity is attained when the range of the radiation most nearly approximates the thickness of the material to be measured. For example, if a steel beam 4 in. thick were to be measured, alpha and beta radiation would be useless since $\frac{1}{4}$ in. of steel stops all such radiation. Gamma rays, on the other hand, will penetrate as much as 12 in. of steel. A high energy gamma ray of 1.2 Mev is reduced to only one-sixteenth of its original intensity by 4 in. of steel whereas a low-energy gamma ray, 0.3 Mev, is reduced to one-two-hundred-fiftieth by the same thickness. The latter would be preferable in this case.

Unlike the other types of radiative absorption, there is no simple relationship between neutron absorption and the density of the medium. Neutrons are absorbed in a selective fashion, depending not only on the density of the absorber, but also on its composition. What may otherwise be a drawback proves useful in determining relative proportions in a mixture of substances. In some cases, irradiation with neutrons causes the elements in the absorber to develop reaction products by which their relative proportions can be determined. Thus, if the absorber consists of a coating and a base, it may be possible to determine the proportion of coating to base and hence the coating thickness.

Instrument Types. Thickness-measuring instruments are of three types, transmission, reflection, or absorption-activation, according to specific needs. Transmission instruments are, on the whole, the simplest and most common type. The source and detector are on opposite sides of the material to be measured. The attenuation of the detected rays is a direct indication of the thickness of the measured material.

A reflection instrument has the source and detector on the same side and measures the radiation reflected back through the material. It is inherently a less sensitive instrument than the transmission gage due to difficulties in shielding the detector from the direct rays from the source, but is invaluable in the measurement of materials accessible from one side only, such as closed tanks and pipes or paper or rubber on the roll during calendering. A shield is used in these gages to prevent the detector from "seeing" the direct rays from the source. This is a simple matter with beta gages, but the gamma ray's greater penetration requires very heavy shielding.

Absorption-activation instruments make use of the behavior of neutrons. The sample is bombarded, then measured for induced radio-

activity as previously described. The nature and intensity of the resulting radiation is a good indication of the kinds and amounts of material involved.

Radiation Detectors [22, 24, 93, 153, 171, 182, 186]. The detector chosen for any measuring instrument employing radiation is as vital to the success of the instrument as the source and method used. Three types of radiation detectors are commonly used in industrial gages. They are the scintillation counter, the Geiger-Mueller counter, and the ionization chamber. Photographic emulsions are also employed to a much lesser extent.

The first type, the scintillation counter, usually consists of a phosphor and a photomultiplier combination in conjunction with a counting circuit. The particles to be detected strike the phosphor and expend their energy to produce photons. These are, in turn, detected by the photomultiplier and counted.

The choice of phosphor (organic, inorganic) and its form (shape, thickness, etc.) depend on the particle to be detected. The short-range alpha particle, for example, requires only a thin layer of material for energy absorption. Beta particles require thicker layers. Large blocks of phosphor of high stopping power may be used for gamma rays.

A photomultiplier tube consists of a photosensitive surface, the photocathode, a number of dynodes, and an anode. Photons, which are small bundles of light energy, strike the photocathode, causing electrons to be emitted. These electrons are directed toward the dynodes where they cause further emission. The process is repeated many times. Each time the electrons strike a new surface, multiplication takes place, i.e., each electron causes two or more electrons to be emitted. After striking a series of dynodes, the final stream of electrons is collected by the anode. The output current from the anode is, for a given photomultiplier, an indication of the photon input.

The photomultiplier varies according to prospective source and use. The variants in this case are the photocathode, which determines the efficiency of light collection from an extended source; the signal/noise ratio, which determines the particle energies to be detected; and the multiplication factor, which determines the amount of subsequent amplification necessary.

The scintillation counter is noted for its high sensitivity (ability to detect very low energy radiations) and counting efficiency (ratio of the number of ions counted to the total number produced). It is about 50 times more sensitive to gamma radiations than ion chamber instruments and is capable of speeds approximately 400 times as great as the Geiger-Mueller

counter. It also has the advantage of not suffering from the limited lifetimes due to gas dissociation of ion chamber instruments. The chief disadvantage of the scintillation counter is its need for a highly stable power supply for the photomultiplier tube. The scintillation counter is most commonly used in rapid scanning devices or for picking up low radiation fields such as those found in some reflection-type gages.

The other two main types of radiation detectors, known generally as ion chambers, are essentially two electrodes in the presence of a gas. Should the gas be ionized by radiations when the electrodes are uncharged, most of the ions will recombine to form neutral atoms and very few will strike the electrodes. If a potential difference is introduced, the electrons will be accelerated in the direction of the anode, the positive ions toward the cathode. As the potential difference is increased, the number of ions collected per unit time will increase until they are being collected at a maximum rate. This potential difference is called the saturation voltage.

The Geiger-Mueller (G-M) tube is a special form of ion chamber in which the applied voltage is higher than saturation voltage, and accelerates the electrons to such an extent that they cause further ionization of the gas molecules they strike. These ions in turn ionize other molecules, etc. The result is an avalanche effect and is called gas amplification. This multiplication of the initial ions causes a discharge along the length of the anode.

The G-M tube has three important properties. They are: (1) The output pulse at a given applied voltage is constant in amplitude and independent of primary ionization, i.e., an alpha and a beta particle of any energy would give a pulse of the same amplitude. (2) The output pulse is sufficiently large to be shown with little or no amplification. (3) The sensitivity is such that a count is obtained when a single ion is produced in the chamber.

The ionization chamber is a form of ion chamber operated at saturation voltage. It is essentially a central collecting electrode encased in a cylinder which acts as the second electrode. The kind of gas and amount of pressure used in the cylinder are determined by the particle to be detected. A hydrogen-filled chamber, for example, will be sensitive to fast neutrons, a boron trifluoride-filled chamber, to slow neutrons. Other gases are sensitive in varying degrees to different forms of radiation.

For saturation conditions it is necessary to have as high an electric field as possible. Thus the electrodes should be closely spaced to avoid the need for high voltages and the subsequent strain on the insulation. On the other hand,

sensitivity is increased by allowing for a large gas volume around the collecting electrode in which the particles may dissipate much of their energy. This requires that the electrodes be widely spaced. In some instances, high-pressure ionization chambers as well as some liquid and solid chambers have been found to be effective. Guard rings are often used to avoid fringing fields and to help in defining the sensitive volume.

If alpha and beta particles are to be measured, their lower penetrating power requires that a window be provided in the ionization chamber.

The properties of the ionization chamber are: (1) The size of the output current varies with a high degree of linearity as the energy of the incoming radiation. (2) Because the gas amplification is unity, the signal usually requires considerable amplification before it is useful. (3) The recovery time is negligible compared with that of a G-M tube, hence much higher activity can be successfully recorded. (4) The design of the ionization chamber is varied according to the particle type to be measured and the sensitivity desired.

Because of its adaptability and sensitivity, the ionization chamber is used in most modern radiation thickness gages.

Photographic emulsions are made developable by the passage of charged particles. The density of the image varies with the intensity of incident radiation and therefore the thickness of the object.⁴

Radiation Hazards [163]. Should one of the radiation methods of thickness measurement be adopted, care should be taken to protect the workers from the radiation. The shielding of any instrument should be checked with the manufacturer and, in cases where a certain amount of exposure is unavoidable, badges or personal counters should be worn by the workers involved and periodically checked.

The following isotopes have been used for thickness measurement; data from [48, 193]:

Bismuth, Bi-210.....	Half life, 5.0 days; beta radiation, 1.17 Mev; gamma radiation, none (to study the deposition of thin films of bismuth).
Carbon, C-14.....	Half life, 5,740 years; beta radiation, 0.154 Mev; gamma radiation, none (measuring thickness of plastic films).
Cerium, Ce-144.....	Half life, 290 days; beta radiation, 3.0 Mev; gamma radiation, 0.085 Mev (thin sheets of intermediate grade steel).
Cesium, Cs-137.....	Half life, 33 years; low-energy beta rays (for gaging light materials).

Cobalt, Co-60.....	Half life, 5.2 years; beta radiation, 0.31 Mev; gamma radiation, 1.17 and 1.33 Mev (measuring thickness of thick portions of metal; equivalent to 2,000-kv X-rays).
Gold, Au-198.....	Half life, 2.69 days; beta radiation, 0.97 Mev; gamma radiation 0.411 Mev (measuring thickness of films deposited by high temperature vaporization of gold).
Gold, Au-199.....	Properties similar to Au-198.
Iodine, I-131.....	Half life, 8.0 days; beta radiation, 0.60 Mev; plus gamma radiation (measuring rubber films of one micron thickness).
Iridium, Ir-192.....	Half life, 75 days; gamma radiation, 0.35 Mev (plate, pipe, and tank wall thickness).
Phosphorus, P-32.....	Half life, 14.3 days; beta radiation, 1.712 Mev; gamma radiation, none (measuring film thickness of printing ink).
Platinum, Pt-197.....	Half life, 18 days; beta radiation, 0.65 Mev; gamma radiation, none (measuring thickness of platinum plating on ceramic ware).
Radium, Ra-226.....	Half life, 1,600 years; gamma radiation, 0.8 Mev (pipe wall thickness).
Ruthenium, Ru-106.....	Half life, 1.0 years; beta radiation 0.041 Mev; gamma radiation, none.
Strontium, Sr-90.....	Half life, 25 years; beta radiation, 0.537 Mev; gamma radiation, none (for a number of thickness gages).
Thallium, Tl-204.....	Half life, 2.7 years; beta radiation, 0.8 Mev; gamma radiation, none (for thickness gages in general).
Yttrium, Y-90.....	Half life, 62 hours; beta radiation, 2.16 Mev; gamma radiation, none (decay product of strontium-90).
Yttrium, Y-91.....	Half life, 57 days; beta radiation, 1.537 Mev; gamma radiation, none (for thickness gages in general).

7.1. Beta-Ray Absorption [51, 56, 57, 92, 119, 132, 182, 189].

7.11. Tracerlab Gage [182] (figs. 136, 137). Radioactive strontium-90, with a half life of 25 years, is enclosed with proper safety devices in a hermetically sealed capsule in a cast-aluminum mount, at the end of a C-frame with a 19.25-in. throat and an opening of 2 in. between source and ionization chamber. The latter, with the preamplifier, is located above the material. The indicator, max 20 μ a, is so set that normal thickness appears at the center of the scale with plus-minus indication of thickness, weight per unit area, or percent deviation from unit weight. The possible ranges of weight and thickness with this meter run from 0.1 to 2,000 mg/cm², i.e., from 0.015 mil in aluminum to 100 mils in steel. Standard maximum ranges are: Paper,

⁴ For the most recent developments in the use of radiation, see *Nucleonics* 13, 76, 77, 82-88 (1955).

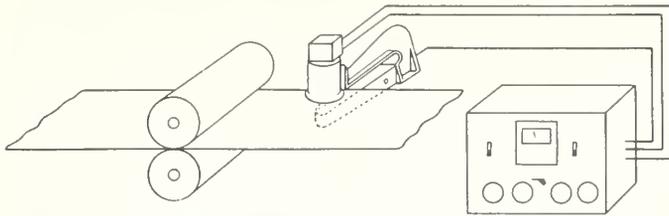


FIGURE 136. Typical application of absorption gage.

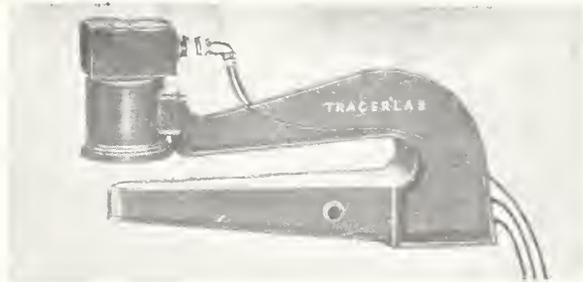


FIGURE 137. "Tracerlab" absorption gage.

600 mg/cm²; steel, 30 mils; aluminum, 90 mils; plastics, 200 mils; rubber, 250 mils. The accuracy is given as ± 2 percent; in special cases ± 0.5 percent is obtainable. For moving sheets measurements have been made at 3,000 fpm, which is about 30 mph. Generally it is desirable to get the average thickness of the sheet by adjusting the time-constant of the indicating circuits by electrical means.

Tracerlab has also developed a beta-ray thickness meter to measure the wall thickness (or eccentricity) of vertically mounted tubes of small or large diameter, beginning with 0.1-in. inner diameter. A source of strontium-90 is mounted inside at the tip of a long rod. Two detector units (ionization chambers) are mounted 180° apart near the source, which is held fixed inside the tubing while the tubing is moved past the source. Then the tubing is rotated 90° and returned to its original position. The wall thickness is recorded with two pens on a recorder. The response time of the gaging units can be made as short as 0.1 sec, and wall thicknesses up to 30 mils of steel or 80 mils of aluminum can be measured. Tubings of greater wall thickness can be measured with gamma radiation. The average tube speed is about 30 in./min. With higher testing speed the averaging of peaks and depressions would be more pronounced.

Manufacturer: Tracerlab, Inc., Boston, Mass.

7.12. "Accuray" Gage [92, 119]. The radiating source, strontium-90, and the operating principle are the same as in 7.11. The standardization is automatic, compensating for decay of source, change of characteristics of electronic components, accumulation of dirt on the housings, or changes in the density of the air path between source and receiver because of varia-

tion in temperature, pressure, or humidity. This gage is mainly used in metal, rubber, textile, and plastics industries. Four throat openings of the mount are available: 24, 36, 48, 60 in.; the position of the source is adjustable across the sheet.

A special design has been developed to measure the thickness of abrasive-coated sheets running at 350 fpm. Five beta-ray gages are used for this application, each one feeding a strip chart recorder: (1) weight of the backing material, (2) weight of the backing material plus adhesive, (3) weight of the backing material plus adhesive plus abrasive after oven treatment (curing), (4) total weight after partial cure, and (5) final weight including final adhesive.

Manufacturer: Industrial Nucleonics Corp., Columbus, Ohio.

7.13. GE Gage [56, 57] (fig. 138). In this gage a beam of beta rays (emitted by 2.5 millicuries of ruthenium-106 or 10 millicuries of strontium-90) is chopped 90 times per second by a motor-driven rotating disk before it passes through the material. An ionization chamber receives the energy of the beam and produces a 90-cps signal which is compared with a 90-cps reference signal from a small generator on the same shaft as the chopper disk. Any difference between the beta-ray signal and the reference signal indicates a deviation of the thickness from the standard value. To adjust the nominal thickness reading, the reference signal is adjustable by a voltage divider. The applications of this gage are the same as for others of the same principle. Under normal conditions a recalibration is recommended every 4 hours. The normal time constant of these gages is 1.5 sec, but by changing one resistor in the control cabinet, the minimum may be made as low as 0.3 sec. Ranges are 5 to 150 oz/yd² with a strontium source and 50 to 300 oz/yd² with a ruthenium source. The accuracy is given as ± 1 percent with strontium, ± 2 percent with ruthenium.

Manufacturer: General Electric Co., Schenectady, N. Y.

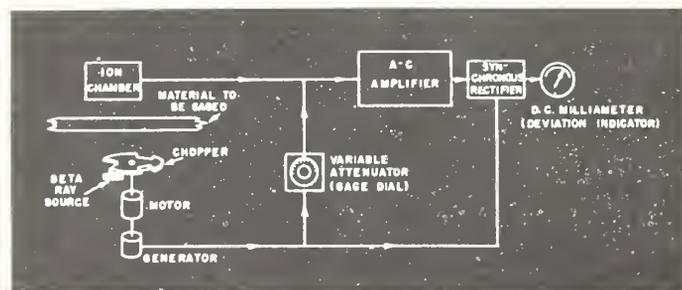


FIGURE 138. Block diagram of beta-ray thickness gage, GE.

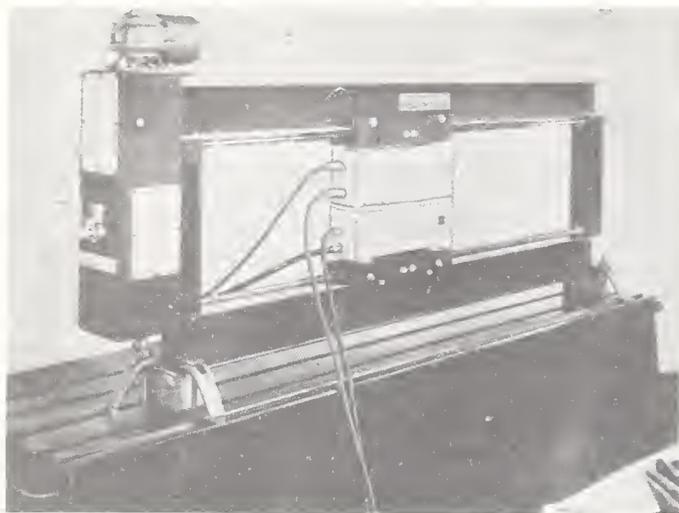


FIGURE 139. Scanning beta-ray gage, Pratt & Whitney.

7.14. Pratt & Whitney Gage (fig. 139). This company manufactures two designs of beta-ray gages, one in the usual arrangement of a deep-throated narrow C-frame (like fig. 137), and a special design of a scanning gage as shown in figure 139. In the latter, there are two boxes, one containing the radiating source, strontium-90, and the other the ionization chamber and preamplifier. These are oscillated automatically at the rate of 20 in./min across the width of the material, up to 54 in. One of the applications of this gage is the measurement of the entire thickness of tinplated sheets. The output can be used in the usual way to feed an indicator, a recorder, or alarm or control signals. The thickness (weight) range is approximately 2 to 200 oz/yd².

Manufacturer: Pratt & Whitney, Hartford, Conn.

7.15. "Betameter" [132]. In this beta-ray gage, errors caused by the decay of the radiation source are eliminated by using a second source of radiation and a second ionization detector. The second chamber is mounted in a box attached to one arm of the frame, and is known as the balance chamber. It is also used to adjust the instrument to any weight-range of the material under test. The calibration operation is the following: When the instrument is set to "operate," the signals from the two chambers are fed to the input. They are of opposite polarity, and by adjustment of the balance controls they can be made of equal magnitude. Then, with the material in the gap and the balance chamber correctly adjusted, there will again be zero signal on the input, and the meter and recorder will remain in the center of their scales. Changes in weight of the material in the gap will cause a signal and a deviation of the pointer from the scale center. As the two sources are similar, they both decay at the same rate, which gives equal signal changes in the

two chambers, preventing any error in standard weight due to source decay.

As a source, 20 millicuries of either strontium-90 or thallium-204 are used. The weight ranges of this instrument are:

SR-90	Tl-204
oz/yd ²	oz/yd ²
0 to 60	0 to 12
50 to 120	10 to 24
110 to 190	22 to 38

The time constant is 10 sec. The influence of a 10 deg F change in temperature is 0.025 oz/yd², and the influence of atmospheric pressure is 0.06 oz/yd² per inch Hg.

Manufacturer: Isotope Products, Ltd., Oakville, Ont.

7.16. "EKCO" Gage [155, 197]. There are two different designs of this instrument. One has a single channel, comparing the output of the detecting head with an adjustable voltage derived from a stable d-c source. In the other design there are two radioactive sources and two detecting heads connected in opposition in a bridge circuit. One set is applied to a sample of known constant absorption, the other to the material under test. The indicator is a meter with zero in the center. For this instrument the ionization current is measured with a vibrating capacitor electrometer [155, 197] across a high resistance of 10¹² ohms. The oscillation frequency of the capacitor is 550 cps. An a-c voltage of the same frequency, representing the unbalance, is generated, amplified, and rectified for indication on a d-c instrument with a scale length of 6 in.

Radiation sources are either strontium-90 or thallium-204. The ranges of measurement are from 5.8 to 160 oz/yd² for strontium, and from 0.29 to 43 oz/yd² for thallium. The accuracy is given as ± 1 percent when using the instrument for time constants of sufficient length, adjustable from 0.25 to 60 sec.

Manufacturer: E. K. Cole, Ltd., London. Representative for United States: American Tradair Corp., New York, N. Y.

7.17. Goodyear Corp. Film Gage [105]. This gage, developed by the Goodyear Research Laboratories, uses carbon-14 as a source of beta radiation, which presents no shielding problem on account of its softness. It has a half life of about 5,700 years. Measurement is made during production, on plastic films with a weight of only 20 mg/cm² (5.8 oz/yd²). For material with a specific gravity of 1.0 (like Pliofilm and vinyl films) this corresponds to a thickness of 0.2 mm (8 mils). Such a film thickness is gaged to an accuracy of ± 1 percent.

7.2. Beta-Ray Backscattering [59] (figs. 140, 141a, b, 142, 143, 144). To measure the thickness of sheets with the foregoing absorption

methods, the sample has to be accessible from both sides and the measurement of coating thickness is not practicable. To measure from one side only, a method using beta-radiation backscattering has been developed by Tracerlab.

In this case (fig. 140), the radiation source is located in the same instrument head as the radiation detector, but it is outside the window and shielded in such a way that radiation emitted from it cannot directly reach the detector. When a layer of the material under test is placed in or passed through the measuring position, the radiation detector (here an ionization chamber) is actuated by the beta rays diffusely reflected from the material, and the resulting chamber current indicates the thickness. The window is thin, to permit the entrance of beta particles. The ability of a material to backscatter beta rays is a function of the atomic number. Increasing thickness of the material produces lessening increments of detector response until an asymptotic value of chamber current is reached. Figure 141a is a typical

curve obtained when the maximum beta-ray energy can no longer penetrate to the far surface of the material, and still return by scattering to the chamber. The thickness at which this saturation occurs is frequently referred to as "infinite thickness" and depends on the maximum beta-ray energy emitted by the source. The saturation value of detector response is a function of the scattering properties of the material under test.

If now a layer of another material of different atomic number is present on the scattering surface of the base material, the detector response decreases or increases accordingly, as the change is to lower or higher atomic number. Figure 141b shows an increase, indicating a change to a higher atomic number. The chamber current continues to increase with additional thickness of the coating material until a new saturation value is reached, depending on the atomic number of the coating. To make useful measurements of the thickness of a coating, the thickness of the base material has to be "infinite" so that its variation in weight per unit area does not affect the readings, while the thickness of the coating has to be such that it lies in the initial approximately linear region of the characteristic curve. This requires the proper selection of isotopes, and of course it is necessary that the atomic number of coating and base be sufficiently far apart.

In figure 140 the distance d of the detector from the surface of the material under test is very important. For distance zero, no reflected beta particles can enter the chamber. As the distance increases, more and more radiation enters until a maximum is reached (at a distance of 400 mils for the design, fig. 142). For larger distances the radiation falls again. The curve is a function only of design; it is independent of the material under test and of the

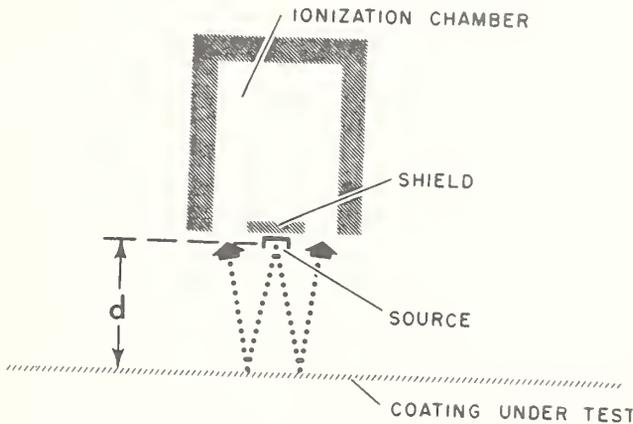


FIGURE 140. Source and chamber mounting. Thickness measurement through backscattering of beta rays.

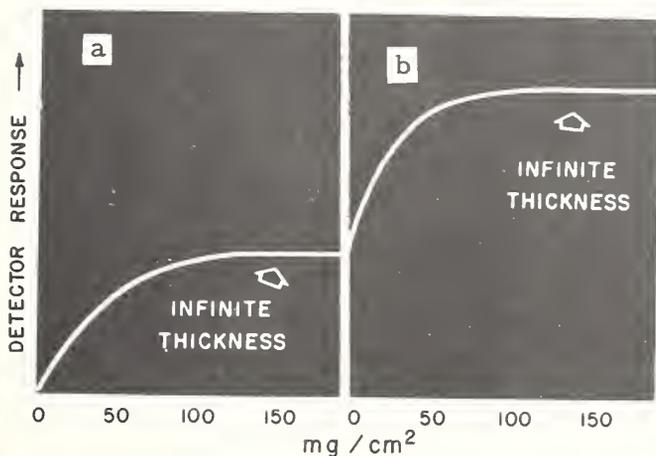


FIGURE 141. Backscattering absorption curves for two types of material, each absorber of higher atomic number than the backing material.

Thickness measurement through backscattering of beta rays.

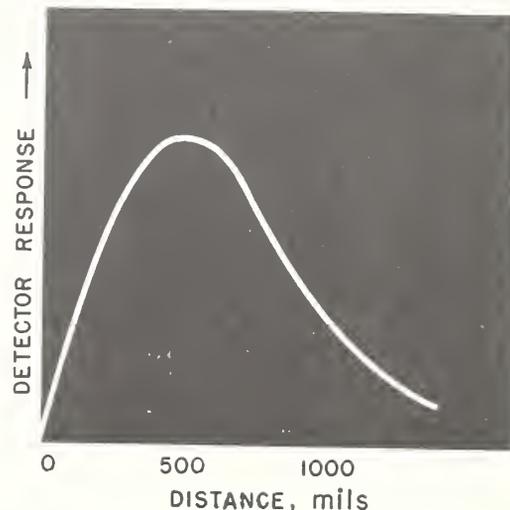


FIGURE 142.

Thickness measurement through backscattering of beta rays.

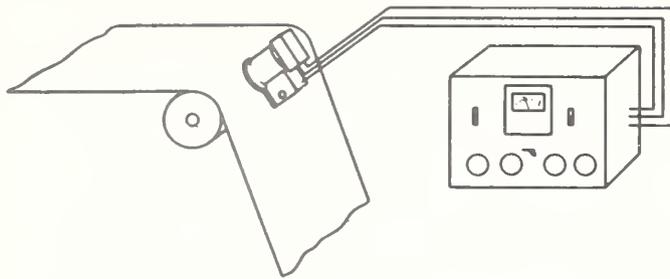


FIGURE 143. Typical application of backscatter gage.
Thickness measurement through backscattering of beta rays.

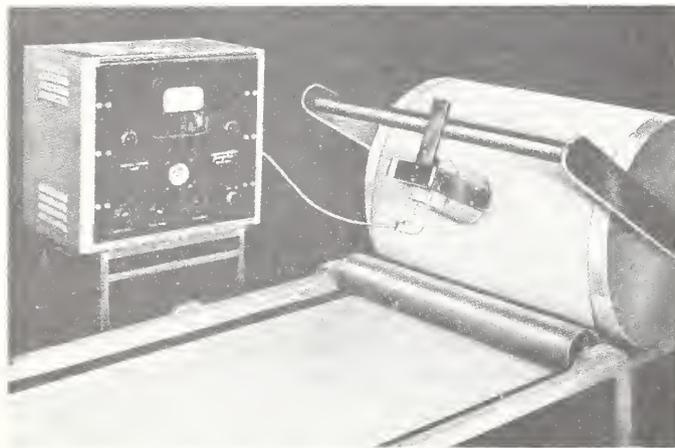


FIGURE 144. Backscatter beta gage measuring the thickness of rubber film on demonstration calender roll.

isotopes used. A typical application of the backscatter gage is given in figure 143. Figure 144 shows an installation to measure the thickness of rubber film on a calender roll.

The reading error for these gages has been found practically to be ± 0.3 mg/cm², divided by the difference of atomic number units, or ± 1 percent of full-scale meter reading. For a difference of 30 between the atomic numbers (tin or steel), measurements can thus be made to ± 0.01 mg/cm².

7.3. Neutron Bombardment, Measurement of Beta-Ray Emission [212]. This method has been developed by Armour Research Foundation to replace destructive methods of measuring silver coatings about 1 mil thick on the internal surface of 50-mil brass (90 Cu, 10 Zn) waveguides. The entire waveguide is bombarded for about 140 sec with neutrons emitted by a 200-millicurie Ra-Be fast-neutron source in a cylindrical steel water tank. After this the object is immediately brought to the counting station with a special handling tool. Beta rays are emitted by the base metal as well as by the silver coating; however, the nuclear cross sections, half lives, and beta-ray energies of the individual elements involved are such that the resulting emission is largely due to the silver coating. Further, in the range of interest (0.1

to 1 mil) the net counting rate is proportional to the amount of silver on the surface under examination. The total time for one thickness measurement over an area of 1 cm² is about 5 minutes, and the result is accurate to better than ± 0.01 mil.

7.4. Gamma-Ray Absorption and Backscattering [12, 13] (figs. 145, 146, 147, 148). The main application of gamma rays, which are far more penetrating than beta rays or X-rays, in thickness measurement, is in the measurement of thickness of heavy steel objects. Where X-ray equipment using 1,000 kv or more would be needed to penetrate 8 in. of steel, 200 mg of radium furnishes sufficient gamma-radiation. Thus, the gamma-ray instrument is not only smaller (more mobile), but is also less expensive.

To measure the thickness of very heavy steel plates or coatings the same photographic methods for measuring the absorption can be used as with X-rays. The radium pill corresponds to the focus of the X-ray tube. The amount of radium needed depends on the thickness of the object.

The *Penetron* (fig. 145) is used for measuring the thickness of the walls of pipes, containers, or vessels, from one side only and is based on the backscattering of gamma rays. The source of gamma radiation is 1 mg of radium in the form of a commercially available salt, surrounded with a shield containing a window, which directs the beam of gamma rays as desired. The shaded layers 1-2-3 represent adjacent layers of a homogeneous wall. Gamma rays emerge from the source S, impinge on the wall, and penetrate into it. Rays emerging on the other side serve no useful purpose, as it is the backscattered portion of the radiation which is utilized for measuring wall thickness from one side. The intensity of this backscattered radiation is a function of the wall thickness. To prevent radiation from falling directly on the detector D, the block SH is used as a shield. Figure 146 shows the calibration of the instrument for a flat iron plate. Up to 0.2 in. thick, the deflection of the gamma meter is approximately linear with the thickness.

Another device using reflected gamma rays for the indication of the thickness of metal sheets is described in (pat. 6). Radioactive cobalt is positioned in a body of lead a predetermined distance from the surface of the material under test, so that the rays are directed towards the face of the sheet. A detector, which is shielded from the direct radiation from the cobalt source, is positioned in the same block of lead. It receives only reflected radiation from the material.

Special heads are used in the measurement of tubes from both the outside and the inside. For measuring straight tubes from the outside, the

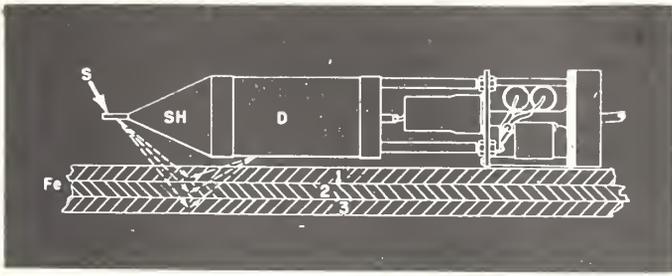


FIGURE 145. Diagrammatic illustration of the "Penetron."

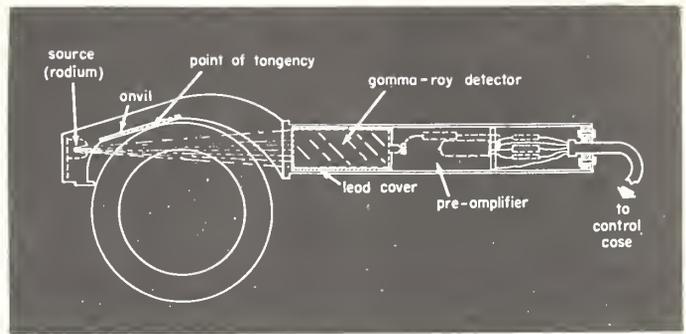


FIGURE 147. Principle of measurement, "Penetron" tangential head.

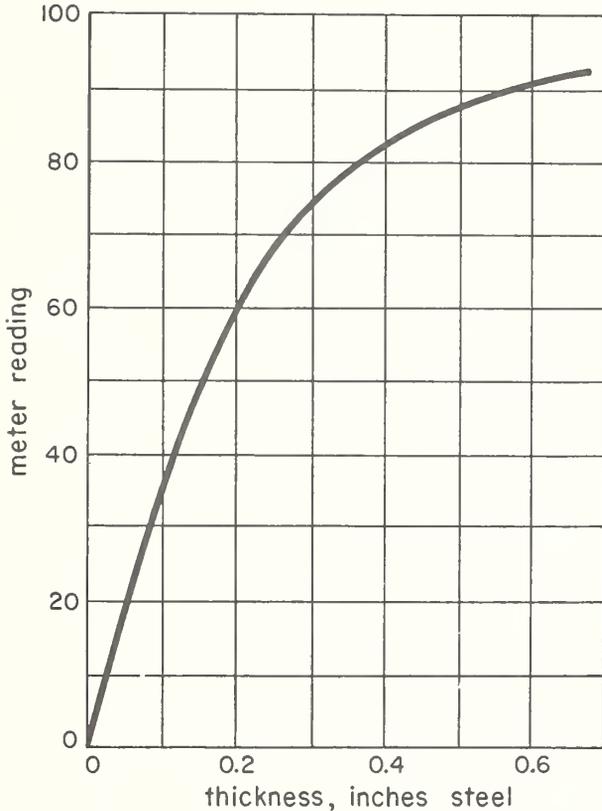


FIGURE 146. Calibration curve of the "Penetron" for a flat iron plate or a tube measured from the outside.

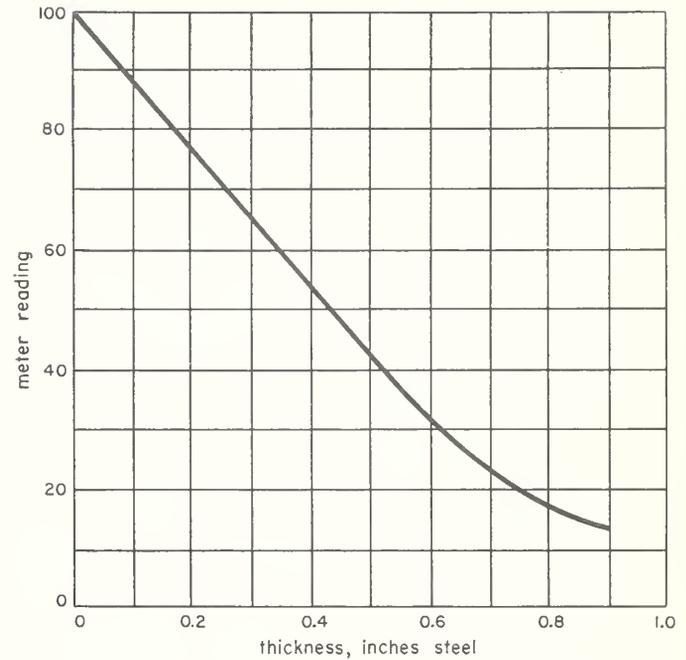


FIGURE 148. Calibration curve for the tangential head of the "Penetron."

standard head, a steel tube 2.5 in. in diameter and 13 in. long is placed in a holder with permanent magnets, which serve both to support the head and to hold it onto the surface of the metal object. To measure the inside of tubes, a pneumatic holder is used.

The tangential head (fig. 147) is a special auxiliary device which was developed to measure boiler tubes. With this instrument, tubes up to 12.75 in. in diameter with a wall thickness up to 2.5 in. can be measured. The calibration curve of this instrument (fig. 148) is different from figure 146. The meter gives maximum reading with zero thickness of the tube under test. The accuracy of these meters is given as ± 3 percent.

Manufacturers: (a) United Engineers, Inc., Tulsa, Okla., with the trade name "Penetron"; (b) Instruments, Inc., Tulsa, Okla., with the trade name "Rad-O-Thik."

7.5. Reduction of Thickness By Wear, Autoradiography [121, 162] (pat. 9). Some difficult problems in measuring the removal of material by wear have been solved by the use of radioactive tracers. Typical examples are: Piston rings, bearings, gears, rubber tires, cutting tools, polishes, automotive finishes, floor waxes.

To measure the wear of piston rings or bearings, the metal is first exposed to neutrons in a reactor and the radioactivity is measured before the part is placed in a testing engine. The quantity of metal worn off is determined by measuring the radioactivity in the lubricating oil.

Actual transfer of the radioactive metal can be detected by autoradiographing the part that has been in contact with the activated component. Tracerlab, Inc., and the Massachusetts Institute of Technology have worked on this method.

8.00. Thickness Meters For The Blind

To make it possible to employ blind people in production, a number of thickness meters, especially of the mechanical type, have been adapted for nonvisual use. Devices such as dial micrometers and other pointer instruments and watches have been modified to be read by touch. The standard Starrett micrometer, for example, has been modified for fingernail reading. All graduations on the sleeve have been deepened; the barrel graduations have been deepened and varigated raised indications attached. The operator can quickly determine in which 25-mil division the measurement falls and, according to instruction, can determine the final measurement within $\frac{1}{4}$ mil.

Auditory devices for thickness indication vary either the intensity or the frequency (pitch) of sound. The hearing ability of the blind operator being more highly developed than in others, these devices often provide the same speed of operation as the conventional devices.

The catalog "Aids for the Blind," published by the American Foundation for the Blind, New York 11, N. Y., lists many of the adaptations developed by manufacturers for thickness measurement. The Foundation will assist in duplicating earlier designs of such meters or in developing new ones.

9.00. ASTM Acceptance Tests

Thickness measurements for acceptance tests on many different objects have been standardized by the American Society for Testing Materials; the methods are listed by number below. In all these specifications, which can be found in the ASTM Standard or its periodical revision, thickness is only one of a number of properties of the objects under test. The thickness meter in most cases is a mechanical micrometer, but the preparation of the sample is different.

A. FERROUS MATERIALS

- 90. Weight of zinc coatings.
- 166. Electrodeposited coatings of Ni and Cr.
- 219. Local thickness of electrodeposited coatings.
- 239. Uniformity of Zn coating.
- 267. Lead alloy coating.
- 309. Weight and composition of coatings, spot test.
- 361. Zn coating roofing sheets.
- 363. Zn coating ground wire strand.

B. NONFERROUS METALS

- 33. Tinned electric copper wire.
- 101. Lead-coated copper sheets.
- 110. Dielectric method for anodically coated aluminum.
- 128. Sleeves and tubing for radio tube cathodes.
- 136. Sealing of anodically coated aluminum.

- 137. Weight of coating on anodically coated aluminum.
- 141. Electrodeposited coating of Ni and Cr on Cu.
- 142. Electrodeposited coating of Ni and Cr on Zn.
- 189. Lead-coated electric copper wire.
- 200. Lead coat on steel by electrodeposition.
- 205. Weighing method for fine wires.
- 219. Testing fine round and flat wire.
- 244. Thickness of anodic coatings measured with "Filmeter."
- 246. Hard-drawn electric copper wire.
 - C. CERAMICS, CONCRETE, ETC.
- 167. Thermal insulating materials.
- 220. Flat asbestos cement sheets.
- 221. Corrugated asbestos cement sheets.
- 222. Roofing shingles.
- 223. Siding shingles.
 - D. MISCELLANEOUS MATERIALS
- 27. Rubber-insulated wire and cable.
- 39. Woven fabrics.
- 69. Electric friction tape.
- 76. Textile testing.
- 119. Rubber insulating tape.
- 120. Rubber insulating gloves.
- 146. Felted and woven fabrics for roofing.
- 178. Rubber matting, 3,000 v.
- 202. Paper for electric insulation.
- 229. Sheets and plates for electric insulation.
- 295. Varnished cloth and tape for electric insulation.
- 351. Mica testing.
- 353. Rubber on wire and cable.
- 354. Tubular sleeving and braids.
- 374. Solid electric insulation.
- 376. Holland cloth.
- 378. Flat rubber belting.
- 380. Rubber hose.
- 395. Vulcanized rubber.
- 414. Cotton fibers.
- 418. Pile floor covering.
- 419. Fineness of wool.
- 461. Felt.
- 468. Lime-glass insulators.
- 527. Bulking thickness of paper.
- 545. Expansion-joint fillers.
- 574. Ozone-resistant wire insulation.
- 578. Glass yarn.
- 580. Woven glass tape.
- 634. Laminated sheets, uniformity.
- 645. Paper and paper products.
- 652. Mica stampings.
- 668. Rigid tubes for electric insulation.
- 733. Compressed asbestos sheet packing.
- 734. Insulated wires and cables.
- 751. Rubber-coated fabrics.
- 754. Heat-resisting rubber-insulated wires and cables.
- 755. Synthetic rubber-insulated wires and cables.
- 823. Paint films on test panels.
- 898. Weight per unit area of adhesive solids.
- 899. Weight per unit area of liquid adhesives.
- 902. Varnished glass fabrics and tape.
- 1000. Pressure-sensitive adhesive tape for electric insulation.
- 1005. Dry film thickness of paint, etc.
- 1048. Rubber insulating blankets, 16,000 v.
- 1049. Rubber insulator hoods, 20,000 v.
- 1050. Rubber-insulated line hose, 20,000 v.
- 1051. Rubber insulating sleeves, 10,000 v.
- 1055. Latex foam rubber.
- 1056. Sponge and cellular rubber products.
- 1186. Dry-film thickness of nonmagnetic coatings.
- 1212. Wet-film thickness of nonmagnetic coatings.
- 1233. Twine made from bast and leaf fibers.

Literature References

The following items cover a wide variety of methods for thickness measurement. However, completeness cannot be expected, since articles on the subject are scattered over a tremendous number of technical magazines.

Where the method described is not adequately indicated by the title of the article, this information has been added. As far as possible the company affiliation of the author has been given. References are indicated thus [] in the text.

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Patents

1. Austrian Patent 133,092 (1935), C.H.F. Mueller and A. G. Hamburg. X-ray shadow method.
2. British Patent 378,983 (1932), H. Moore and G. Williamson. Measuring the thickness of iron plates. Parallel magnetic flux method.
3. British Patent 620,062, Niles-Bement-Pond Co. (now Pratt & Whitney, Inc.). Pneumatic gages to measure the thickness of moving metal strips.
4. British Patent 601,523, Sigma Instrument Co. and J. Loxham. Pneumatic gages for internal dimension measurements.
5. Swiss Patent 241,935 (1942). Pneumatic measuring instruments.
6. U. S. Patent 2,675,482, Donald C. Brunton.
7. U. S. Patent 2,469,476, J. C. Sellars. Magnetic testing apparatus. "Elcometer" using magnetic attraction.
8. U. S. Patent 1,869,336, A. V. DeForest. Electrical-thermal-visual method.
9. U. S. Patent 2,315,845 (April 6, 1943), Atlantic Refining Co. Wear-test method and composition (Tracer method).

List of Manufacturers

An attempt was made to effect a complete survey of thickness meters built in the United States. Unfortunately, completeness is next to impossible, as a number of our letters requesting information were not answered, although many manufacturers were extremely cooperative. No attempt has been made to list the many sources of simple thickness meters like slide calipers or screw micrometers.

In many cases only a prototype thickness meter has been built. In clear cases of this sort no "manufacturer" is mentioned. Some devices that are not now manufactured are included because many times an old method is taken up again years later, either for a special problem or with components that had not been originally available.

Aireon Mfg. Co., Burbank, Calif.	Optical micrometer.	Browne & Sharpe Mfg. Co., Providence, R. I.	Comparator gages; dial micrometers; electronic calipers; screw micrometers.
American Instrument Co., Silver Spring, Md.	Capacitive thickness meters — <i>Filmeter</i> , <i>Compressometer</i> , <i>Magne-Gage</i> , <i>Magne-Probe</i> .	Brush Development Co., Cleveland, Ohio.	Textile uniformity analyzer.
American Tradair Corp., New York, N. Y.	EKCO nucleonic gages.	Cleveland Trust Co., Cleveland, Ohio.	Half-millionth microcomparator.
Ames Co., B. C., Waltham, Mass.	Caliper gages; comparator gages; dial gages, micrometers; dead-weight gages; screw micrometers.	Commercial Research Laboratories, Detroit, Mich.	Cylinder - wall thickness gage.
Amthor Testing Instr. Co., Brooklyn, N. Y.	Dead-weight gages; dial micrometers.	Decker Aviation Corp., Philadelphia 25, Pa.	Comparator micrometer; ionization transducer.
Anderson Machine Shop, Inc., Needham Heights 94, Mass.	<i>Pacific</i> evenness testers.	Dice, J. W. Co., Englewood, N. J.	<i>Carson</i> electronic thickness gage; ultrasonic thickness gages.
Ateliers de Normandie, Paris 8e, France.	<i>Etamic</i> pneumatic gages for rolling mills.	DoAll Co., Des Plaines, Mich.	Comparator gages, electromagnetic; dial micrometers; <i>Mauser</i> vernier calipers, slide calipers; <i>Tumico</i> screw micrometers.
Automatic Temperature Control Co., Philadelphia, Pa.	Thickness meters with differential transformers.	Eastman Kodak Co., Rochester, N. Y.	Optical comparators.
Bausch & Lomb Optical Co., Rochester, N. Y.	Optical thickness meters.	Electric Eye & Equipment Co., Danville, Ill.	<i>Hurletron</i> gage.
James G. Biddle Co., 1316 Arch St., Philadelphia 7, Pa.	Tinsley thickness gage (for metallic coatings).	Electronic Control Corp., Detroit, Mich.	<i>Bathytrol</i> paint thickness gage.
Boeckler Instrument Co., Tulsa, Okla.	Screw micrometers.	Federal Products Corp., Providence, R. I.	Calipers; comparator gages; dial gages; <i>Dimensionnaire</i> pneumatic gages; <i>Electricator</i> mechanical-electric gages; electronic gages; recording gage; ring thickness gages; roller gages; screw micrometers.
Boonton Radio Corp., Boonton, N. J.	Metal film gauge.	Fielden Instruments Division, Philadelphia, Pa.	Capacitive thickness gage; evenness recorder for textiles.
Branson Instrument Co., Stamford, Conn.	<i>Audigage</i> (ultrasonic); <i>Vidigage</i> (ultrasonic); <i>Coatingage</i> (electromagnetic); <i>Sonigage</i> (ultrasonic).	Foxboro Co., Foxboro, Mass.	<i>Verigraph</i> thickness recorder.
		Gardner, Henry A., Inc. Bethesda, Md.	<i>Elcometer</i> ; "Interchemical" roller gages; penetrating needle gages.
		General Electric Co., Schenectady, N. Y.	Beta-ray thickness meters; eddy-current thickness meters; electromagnetic thickness meters; step gage; X-ray thickness meters.
		Graydon Smith Prod. Corp., West Concord, Mass.	<i>Metrisite</i> thickness gages.
		Gustin-Bacon Manufacturing Co., Kansas City, Mo.	<i>Measurematic</i> gage.
		Haskins Turner Co., Jackson, Mich.	Scale thickness meter for boiler tubes.

- O. Hommel Co., 209-213 Fourth Ave., Pittsburgh, Pa.
- Industrial Electronics Inc., Detroit, Mich.
- Industrial Gauges Corp., Engelwood, N. J.
- Industrial Nucleonics Corp., Columbus, Ohio.
- Instrument Development Laboratories, Needham Heights, Mass.
- Instruments, Inc., Tulsa, Okla.
- Isotope Products, Ltd., Oakville, Ont.
- Johansson, C. E., Gage Co., Detroit, Mich.
- Koehler Instrument Co., Jamaica 33, N. Y.
- Kokour Co., Chicago, Ill.
- Krouse Testing Machine Co., 573 East Eleventh Ave., Columbus 3, Ohio.
- Lea Manufacturing Co., Waterbury 20, Conn.
- Lufkin Rule Co., Saginaw, Mich.
- Magnaflux Corp., Chicago 31, Ill.
- McGlynn Hays Industries, Inc., Belleville 9, N. J.
- Merz Engineering Co., Indianapolis, Ind.
- Metron Instrument Co., Denver, Colo.
- Metzger, F. F., & Son, Philadelphia, Pa.
- Micrometer Pneumatique, SOLEX, Courbevoie, Seine (France).
- Moore Products Co., Philadelphia, Pa.
- North American Philips Co., Mt. Vernon, N. Y.
- Photoconn Research Products, Pasadena, Calif.
- Platers Research Corp., 59 East Fourth St., New York 3, N. Y.
- Poole Instruments, Inc., Dallas, Texas.
- Pratt & Whitney, Inc., Hartford, Conn.
- Precision Thermometer & Instrument Co., Philadelphia, Pa.
- Radium Elektrizitäts-gesellschaft m.b.H in Wippenföh, Germany.
- OHCO miniature layer thickness meter (probably similar to the Elcometer).
- Electromagnetic thickness meters, *Dyna-Myke*.
- X-ray gages.
- Abrasive coating gages; *Accuray* (beta ray) thickness gages.
- Optimike* for flat surfaces.
- Rad-O-Thik* gamma-ray gage.
- Betameter* beta-ray thickness gage.
- Dial micrometers; *Mikro-kator*, *Minikator* gages; screw micrometers.
- Pfund* paint film thickness gage.
- Dropping testers; electronic thickness testers.
- Krouse magnifying micrometer (permanent magnet type for plated coatings).
- Lectromag* (Lipson) thickness gage.
- Calipers; dial micrometers; screw micrometers.
- Ultrasonic thickness gages (*Sonizon*); electromagnetic thickness gages (*Magnatest*).
- Optimike* for internal measuring.
- Electromagnetic comparator gages.
- Electromagnetic comparator gages.
- Pileometer* thickness gage.
- Continuous pneumatic micrometers.
- Pneumatic comparator gages.
- X-ray coating thickness gage.
- Metroscope* ultrasonic thickness gage.
- Pocket Handigage (permanent magnet type for plated coatings).
- Beta-ray gages; eddy-current foil gages, *Electrolimit* gages; *Magnetic* gages; magnetic *Schuster* gage; millionth comparator gages.
- Calender micrometers, *Princo*.
- Record Electrical Co. Ltd., England.
- Rimat Machine Tool Co., Glendale, Calif.
- Salford Electrical Co., Ltd., England.
- Schaevitz Engineering Co., Camden, N. J.
- Scherr, George, New York, N. Y.
- Sheffield Corp., Dayton, Ohio.
- Shell Development Co., Emeryville, Calif.
- Siemens & Halske
- Siemens New York, Inc., 350 Fifth Ave., New York 1, N. Y. (distrib.)
- Sigma Instrument Co., Letchworth, Hertfordshire, England.
- Sperry Products, Inc., Danbury, Conn.
- Standard Electronic Research Corp., New York, N. Y.
- Standard Gage Co., Poughkeepsie, N. Y.
- Starrett, L. S. Co., Athol, Mass.
- Stevens Arnold, Inc., Boston, Mass.
- Streeter Amet Co., Chicago, Ill.
- Swedish Gage Co. of America. See Johannsson Gage Co.
- Tinsley Industrial Instr. Ltd., London NW 10, England
- Ti-Pi Co., Kansas City, Mo.
- Tracerlab, Inc., Boston, Mass.
- Tubular Micrometer Co., St. James, Minn.
- Union Tool Co., Orange, Mass.
- Unit Process Assemblies, 75 East 4th St., New York, N. Y.
- United Engineers, Tulsa, Okla.
- Van Keuren Co., Watertown 72, Mass.
- Westinghouse Co., Pittsburgh, Pa.
- Wilmotte Raymond Co., Washington, D. C.
- Resistance method for intricate castings.
- Dial gages.
- Eddy-current gage.
- Thickness meters with differential transformer.
- Projectometer*.
- Electrigage*; *Measuray* X-ray gages; *Precisionair* pneumatic gages; reed gage; *Visual* gage.
- Probolog* tube thickness meter.
- Pneumatic comparators; foil thickness meters.
- Reflectogage* ultrasonic thickness meters.
- Diamatrol* photoelectric thickness gages; electron micrometer; X-ray thickness gage.
- Comparator gages; dial micrometers; *Micronar* indicator.
- Calipers; dial micrometers; comparators, indicators; portable gages; sheet gages.
- Electronic micrometer.
- Dial gages; *Guyer* gages; micrometers.
- Pencil thickness gage.
- Ti-Pi floating-caliper gage.
- Beta-ray thickness gages; isotopes.
- Tumico* screw micrometers.
- Calipers; dial gages.
- Dermitron*.
- Penetron* gamma-ray thickness gages.
- Light-wave screw micrometers.
- X-ray thickness gages.
- Visi-Limit* thickness gages.

WASHINGTON, January 23, 1957.

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Metrology of Gage Blocks

Proceedings of a Symposium on Gage Blocks
Held at NBS on August 11 and 12, 1955



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Foreword

A major function of the National Bureau of Standards as set forth in the Act of Congress, March 3, 1901 and amended by Congress in Public Law 619, 1950 is the development and maintenance of the national standards of measurement. As custodian of our national standard of length the Bureau is obligated to calibrate secondary standards of length for other Government agencies, State governments, and industry. By far the most widely used precise standards of length in mechanical industry are precision gage blocks which by virtue of their accuracy, range of sizes, and relatively low cost have made precise standards of length available to even the smallest units of industry. Probably more than any other single development, the availability of such precise standards has made possible the mass production of interchangeable industrial components of the vast array of mechanical devices which have so greatly enhanced our standard of living in this century.

There exists a need to meet even more adequately some of the more critical requirements in the manufacture of industrial components. The Symposium on Gage Blocks was held to permit the exchange of ideas between the National Bureau of Standards and manufacturers and users of gage blocks in the hope that it might be possible to develop better techniques for their manufacture and use.

A. V. ASTIN, *Director*,
National Bureau of Standards.

Preface

The Symposium on Gage Blocks was held at the National Bureau of Standards on August 11 and 12, 1955 under the auspices of the Engineering Metrology Section of the Optics and Metrology Division.

Two sessions, at which fifteen papers were presented, were held. About ninety people attended the sessions.

Three papers dealt with the metallurgical and physical properties of gage block materials, with particular reference to dimensional stability. Another paper considered the characteristics of gage-block measuring surfaces, such as average roughness, depth of individual scratches, and condition of edges.

The present state of the art of applying light waves as standards of length is such that optical interference methods have attained supremacy in the accurate determination of length of contact length standards such as gage blocks. Accordingly, six papers dealt with the application of interferometry to gage blocks, conditions affecting the accuracy of interferometric measurements, and the precision and accuracy attained in recent comparative length measurements by different laboratories. Two other papers were on recent improved interferometer designs.

The four concluding papers, as well as two formal discussions and an informal discussion period, were concerned with the development of commercial and government standards and specifications for gage blocks. Consideration was given to procedures for the surveillance of gage blocks after they have been in use.

IRVIN H. FULLMER, *Chief*,
Engineering Metrology Section,
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1. Precise Interferometric Measurement of Gage Blocks

By E. Engelhard ¹

The purpose of this report is to give a survey of the work of the Physikalisch-Technische Bundesanstalt (PTB) and the former Physikalisch-Technische Reichsanstalt (PTR) concerning length measurements based on light waves, and some suggestions on how to avoid errors which, according to our experiences, occasionally occur with this technique.

I shall start with a brief historical introduction. Length measurement by light waves was started in Germany by Kösters ² nearly 40 years ago, but it should be remembered that Michelson, in his famous determination of the length of the meter with wavelengths of the red cadmium line, ingeniously anticipated the most important elements of practical length measurement by means of light waves.

Now, more than 60 years later, the most precise length measurements are made by means of light waves, although the meter and the yard, strictly speaking, are defined by a metal bar. This can be done by virtue of a resolution of the Seventh General Conference for Weights and Measures in 1927 stating the relation between the wavelength of the red cadmium line and the meter prototype.

The special significance of the light-wave technique for length measurement is founded on the well-known principal features of a light-wave scale, its indestructible and unchangeable character and its natural, very fine and accurate graduation. The most decisive attribute is that everyone, particularly the engineer, can make use of it as a practical standard of length. Thus the great progress in manufacturing modern industrial gage blocks would have been impossible without the technique of length measurement by light waves.

In order to explain the essential features of the technique of length measurement by means of light waves, reference is made to figure 1 which illustrates a very simple, well-known apparatus which was designed by Kösters more than 30 years ago and manufactured by Carl Zeiss. This instrument is generally used in Germany for measurement of gage blocks up to about 100 mm in length. This apparatus includes a monochromator having a collimator consisting of the slit C and the lens O, which makes light from C parallel. The dispersion prism Pr separates the light into beams of different wavelengths which are focused by the lens O₂, on the slit B, after reflection from several mirrors, Pl, S₁, P, and S₂. This apparatus provides a light-wave scale for the measurement of gage blocks in the following manner: The parallel monochromatic beams coming from Pr pass through the beam-dividing glass plate Pl to the mirrors S₂ and P,

¹ Physikalisch-Technische Bundesanstalt, Braunschweig, Germany.

² W. Kösters, Prüfung von Johansson-Endmassen mit Lichtinterferenz. *Feinmechanik (Präzision)* 1, 2-5, 1920; 39-41, 1922.

which represent the measuring surfaces of a gage block. Light is also partially reflected from P1 to the mirror S₁. An observer looking through the slit B sees the image of the gage block PS₂ superimposed on the image of mirror S₁. The mirror S₁ is imaged at R between the two parallel measuring surfaces of the gage block PS₂. If PS₂ is somewhat inclined with respect to S₁ by the screws below the plate P, two wedges are formed, one built up by S₂ and R and the other

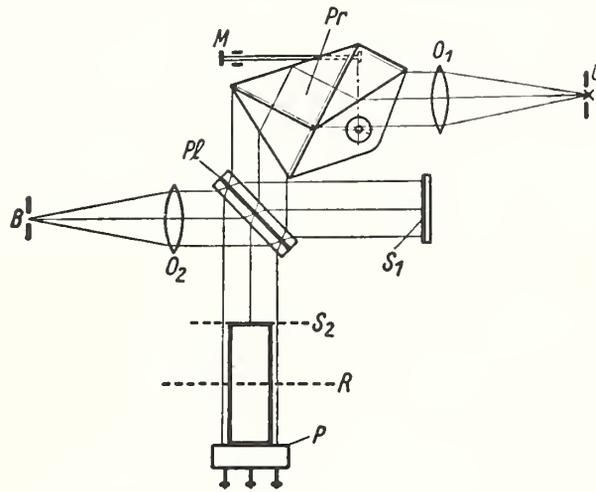


FIGURE 1.

by P and R. In this manner the length of the gage block PS₂ is divided into the thicknesses of two wedges.

If monochromatic light is incident on such a wedge, the well-known interference fringes of equal thickness, so-called because every fringe represents points of the same thickness, are observed. At the apex of such an interference wedge is a fringe of the order 0, the next fringe of the order 1 indicates a thickness of $\lambda/2$, the fringe of the order 2 marks the thickness $2\lambda/2$ and so on, λ representing the wave-

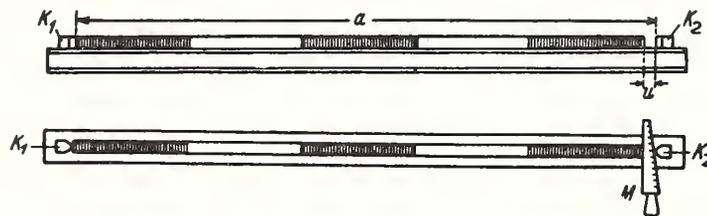


FIGURE 2.

length of the light wave. In general, the fringe of the order n corresponds to a thickness of $n\lambda/2$: The interference wedge resembles very closely the common measuring wedge used in ordinary length measurement for the determination of distances as shown in figure 2. This figure demonstrates the measurement of a surveyor's pole. The distance u between the fixed contact K_2 and the end of the pole is indicated as shown in figure 2 by a wedge graduated with lines on one face. Such a wedge is a very simple instrument for obtaining amplification in length measurement. If the amplification which depends on the angle of the wedge, is, for instance, 1: 10, a graduation in millimeters allows one to read a tenth directly and to estimate a

hundredth millimeter. In the same manner the interference wedge enables us to measure distances, but its graduation is much finer, the discrimination being half a wavelength, i. e., approximately 0.25μ (0.00001 in.). Its amplification is much higher, in the same degree as the graduation is finer. Its graduation lines, unfortunately, are not numbered, which causes some complications.

The field seen by the observer viewing through the slit of this gage-block interferometer is as shown in figure 3. One observes two interference wedges side by side, established as pointed out by the images of the measuring surfaces of the gage block and the reference mirror. If the interference fringes were numbered like the graduation lines on the common measuring wedge, then the numbers as indicated by each wedge would need only to be added. The fraction between the two sets of fringes should, of course, be included. As the fringes are not numbered, it becomes necessary to determine the order of the fringes. To overcome this difficulty in practice two operations

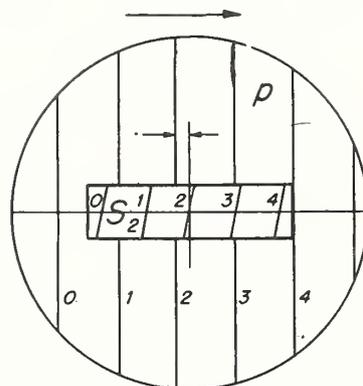


FIGURE 3.

are usually performed. First, a preliminary determination of an approximate value of the length in question is made by comparing the gage block with a known length standard, for instance, by means of mechanical equipment. Therefrom one obtains a probable value of the order within the accuracy of the mechanical comparison of gages, i. e., within a few units of the order of interference. Second, a determination is made of the fractional orders observed with different wavelengths and therefrom the exact value of the order is found by the method of coincidences. Because the latter procedure is somewhat complicated, present practice at the PTB is, in general, to increase the accuracy of the preliminary determination of length of a gage block to such a degree, that the order of interference is known with certainty by the approximate value only.

The method used to reach the necessary accuracy in determining approximate values of the gage blocks is based on interference in white light. In light-wave measurement white light plays a special part by indicating in a striking manner the zero order by means of the so-called achromatic fringe. Thus, the interference fringes in white light are very well adapted to the measurement of the small differences in length which occur in the comparison of gage blocks of equal nominal length.

An essential detail of the apparatus we use for comparing gage blocks by means of interference in white light is the double prism designed by Kösters (fig. 4).

Figure 4 shows the cross section of this double prism as an equilateral triangle ABC . The double prism is cut into two completely symmetrical prisms of 30° , ABD and ACD , which are cemented together with oil along the semimirrored surface AD . A beam incident normally on the surface AB from the left is divided into two beams of equal intensity at the partially coated surface AD . Both partial beams leave the prism after total internal reflection on the surfaces AB and AC in a direction perpendicular to the base surface BC . Suppose a mirror $F_1 F_2$ is adjusted nearly perpendicular to the beams. Then both beams are reflected by the mirror $F_1 F_2$ and sent back to the prism. After another total reflection on AB and AC , respectively, one beam is transmitted through and the other reflected by the semi-mirrored surface AD , both reaching the observer's eye on the right.

In principle, the prism represents a system of well-adjusted plane mirrors AD , AB , and AC imaging the point J_1 on the point J_2 or

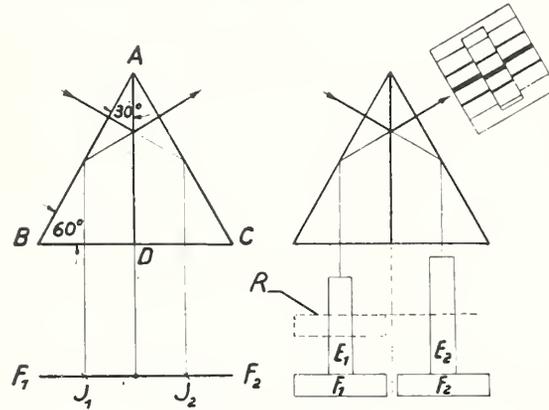


FIGURE 4.

vice versa. Therefore, if two nearly equal gage blocks, wrung to the surfaces F_1 and F_2 , are slightly inclined one to the other as shown on the right, the prism forms two interference wedges by superimposing the images of corresponding surfaces of the gage blocks and the two projecting lower surfaces $F_1 F_2$. If the difference in length of the gage blocks is small enough, interference fringes are visible in white light, the achromatic fringe indicating the zero order or zero thickness of each wedge. Thus, the displacement of the achromatic fringes on both interference wedges corresponds to the length difference of the gage blocks. If one of the two gage blocks, for instance E_1 , is replaced by a mirror, as shown in figure 4, the other gage block may be measured in monochromatic light in the usual manner. The prism forms an image of the mirror between the parallel surfaces of the gage block E_2 and thus, as pointed out before, divides the length of E_2 into the thicknesses of two wedges if the mirror is slightly inclined to the surfaces of the gage block. It makes no difference whether the inclination between the images of the reference mirror and the gage block faces is obtained by inclining the mirror or the gage block or by inclining one-half of the double prism with respect to the other half.

Figure 5 represents an apparatus used at the PTB for the comparison of gage blocks by means of the double prism. The two gage

blocks to be compared are wrung on a single plate, the interference wedge being permanently adjusted by inclining the halves of the double prism. A collimator on the right makes beams of white light parallel. The fringes are observed in the usual manner through a slit on the left. The shift of the fringes, i. e., the difference in length of the gage blocks, is very precisely determined by means of an achromatic glass compensator indicating directly the difference in thousandths of a micron ($0.04 \mu\text{in.}$). The accuracy in comparing gage blocks with this apparatus is at least $\pm 0.01 \mu$ ($0.4 \mu\text{in.}$). This accuracy is high enough for exact determination of the order of interference. Therefore, in measuring absolutely the length of the gage block in question by means of monochromatic light it is sufficient to determine the fraction in one color only, for example, in cadmium red, which is done by means of the glass compensator after removing the known gage block. In this way, very accurate absolute values

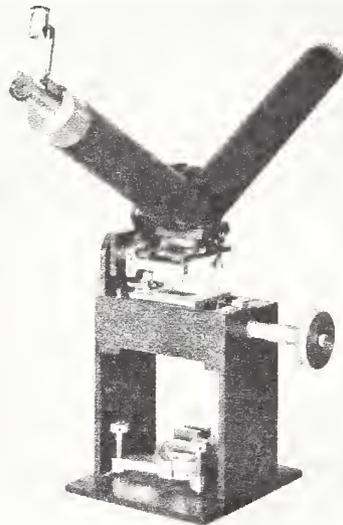


FIGURE 5.

for gage blocks up to 100 mm (4 in.) in length are obtained in a short time with an accuracy of the order $\pm 0.01 \mu$ ($0.4 \mu\text{in.}$).

When longer gage blocks are measured by this method difficulty arises because of the effect of air. Until now, it has not been mentioned in this paper that the wavelength of light depends on the conditions of the atmosphere. Our primary length standard is the wavelength of the red cadmium line which is defined in dry air of 15°C and 760 mm Hg. Therefore, all length measurements by means of light waves usually are reduced to standard conditions by calculated corrections based upon the value of the refraction of air. This requires an accurate measurement of the temperature, barometric pressure, and moisture content of the ambient air. In general, this cannot be done with high accuracy and, therefore, the uncertainty of the corrections due to the refraction of air is somewhat high for gage blocks much over 100 mm (4 in.) in length.

For this reason Kösters designed an apparatus for the measurement of gage blocks up to 1 m in length, with which it is possible to eliminate the influence of air completely. This apparatus is used in the PTB

for the most precise measurements of industrial gage blocks, the highest possible accuracy being about $\pm 0.01 \mu/m$. The simplest method of eliminating the influence of air is to make the measurements in vacuum. But it is not feasible to place the blocks in a vacuum, because they would become longer as the atmospheric pressure was removed and corrections depending on the elasticity of the gage blocks would be necessary. Therefore, the effect of the air is eliminated in the method introduced by Kösters by determining it experimentally using a vacuum chamber for this purpose.

Figure 6 represents the scheme of the PTB apparatus for light-wave measurement of gage blocks up to 1-m length. An essential feature of this apparatus is once more the use of a double prism, forming an image of the reference mirror R between the surfaces M of the gage block E, and dividing the length of the gage block into two wedges. Viewed through the slit B two interference wedges, formed by the faces of the gage block and the reference mirror, are seen if monochromatic light is used for illumination. Another essential feature of this

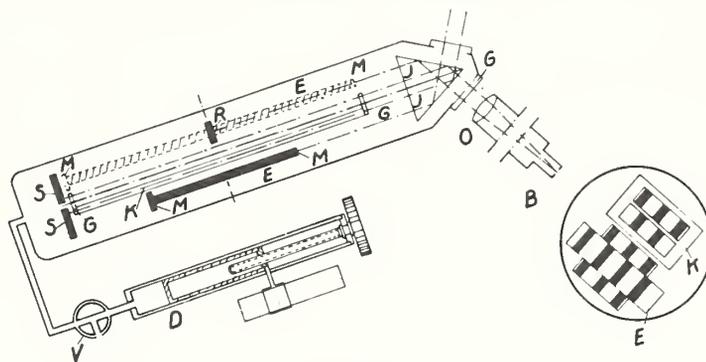


FIGURE 6.

B, Viewing aperture; D, piston and cylinder; E, gage block; G, glass plate; J, double prism; K, vacuum chamber; M, measuring surface; O, objective lens; R, reference flat; S, mirrors; V, valve.

apparatus is the use of a vacuum chamber for the elimination of the refraction of air. The vacuum chamber in its simplest form is an iron tube of rectangular cross section, 1 m in length, closed at both ends with plane-parallel glass plates so that it can be evacuated with a small high-vacuum pump. If this chamber is removed, the double prism images both mirrors S, one on the other, and if one mirror is inclined with respect to the other an interference wedge of nearly zero thickness arises. Introduction of the chamber into one beam, but with the end plates extending into the other beam does not modify the wedge in any manner unless it is evacuated. If it is evacuated, light in the chamber travels faster and the wavelengths become longer. Assume the wavelength in air is λ and in vacuum λ_0 , the index of refraction of the air n is then defined by $n = \lambda_0/\lambda$. Then the number of wavelengths for light passing to and fro in the chamber of 1-m length is $1: \lambda_0/2$ or $2/\lambda_0$ if the chamber is evacuated, and $2/\lambda = 2/\lambda_0 n$ if not, the difference between the unevacuated and the evacuated chamber being $2(n-1)/\lambda_0$.

When the chamber is evacuated an observer looking through the slit B has no idea that the wavelength in the vacuum chamber is changed, so he has the impression that the thickness of the wedge formed by the mirrors S has changed across the field covered by the

evacuated part of the chamber, the change in the order of interference being equal to $2(n-1)/\lambda_0$. Consequently, an observer has a view as illustrated in the inset of figure 6. In addition to the two wedges corresponding to the gage block length in air on the left, at E, there are two wedges referring to the chamber, one wedge representing the unchanged thickness in air, being zero or nearly zero as given by the position of the mirrors S, and another wedge representing the thickness of the same wedge apparently changed by evacuating the chamber. If the fringes in both wedges were numbered, then the difference of the orders equal to $2(n-1)/\lambda_0$ could be immediately read from the wedges. Remember that the order of interference for a gage block of 1-m length in air is $z=2/\lambda$ or $z=2n/\lambda_0$ and that the order for the same gage block in vacuum is $z_0=2/\lambda_0$. Therefore, if the order $2(n-1)/\lambda_0$ as given by the chamber is deducted from the order of interference $2n/\lambda_0$, corresponding to the gage block in air, the order of interference corresponding to the gage in vacuum z_0 is obtained by the equation

$$\frac{2}{\lambda_0} n - \frac{2}{\lambda_0} (n-1) = \frac{2}{\lambda_0} = z_0.$$

Thus, the order of interference for a gage block of 1-m length in vacuum is obtained, without putting the gage block in vacuum, by deducting the order of interference indicated by the vacuum chamber from the order indicated by the gage block in air.

If the length of the gage block is not 1 m, but, let us say, 500 mm, the order indicated by the chamber of 1-m length must be reduced by dividing it by two.

In practice it is not feasible to measure gage blocks over 700 mm in length by this procedure. Measurement of blocks from 700 mm to 1,000 mm (1 m) is made in two steps, the first step being the measurement of a 500 mm gage block and the second step being the measurement of the difference in the lengths of the 500 mm and the longer gage block.

To simplify the method for the elimination of the air refraction the whole interference arrangement is fitted in a box which may be closed. The enclosure is connected to a piston D, which allows the volume of the sealed box to be varied. The piston can be moved to and fro in a cylinder by means of a spindle. Thus, the density of the air in the enclosure and hence the wavelength of light, which depends on the density of air, can be altered by compression or expansion, and the excess fraction or the fractional fringe displacement observed on the gage block can be adjusted to zero.

A scale is attached to the piston. In order to obtain the order of fringes referring to the length of the gage block in vacuum, a number that is read on the scale is deducted from the number of fringes corresponding to the length of the gage block in air, this last number being an integer because the fraction is adjusted to zero. So the decimal complement of the fraction read on the scale represents directly the fraction for the gage block in vacuum.

The graduation of the scale on the piston could in principle be obtained in the following manner: the chamber is filled with air and then slowly evacuated; hereby the number of the passing fringes in cadmium red is counted. This gives some number, let us say 843.92. After sealing the enclosure, the air contained therein is compressed by the piston until a whole number of fringes in the chamber is

observed, the fraction in the field of the chamber being zero. This must be the 844th fringe. Then the position of the index on the scale is marked 844 and the piston is moved until the 845th or the 843d fringe is observed, and so on. The same method can be applied to cadmium green, blue, and other colors. In practice the scale is calculated, but it should be kept in mind that no known values of the refraction are necessary.

The scale does not cover the full range of wavelengths from vacuum to ambient conditions but only the range of wavelengths corresponding to the barometric pressure and temperature likely to be encountered in the measurement of gage blocks. With the valve, V, open there obviously is no relation between the scale reading and the difference in the number of wavelengths per meter in vacuum and for ambient conditions. The method of alining the scale is as follows:

The gage block to be measured is placed in the enclosure. The enclosure is then filled with dried air at ambient atmospheric pressure. After temperature equilibrium of the equipment has been reached the ambient barometric pressure B and temperature t are approximately determined. If, for example, the barometric pressure is 749.7 mm and the temperature 20.1°C , an assumed number of cadmium red fringes may be computed from the formula

$$z = z_{(760\text{ mm } 20^{\circ}\text{ C})} \frac{1 + \alpha 20}{1 + \alpha t} \frac{B}{760},$$

where $\alpha = 0.00367$ and $z_{(760\text{ mm } 20^{\circ}\text{ C})} \approx 843.9/\text{m}$.

Substituting the assumed values of B and t in this formula, $z = 832.2$.

The valve is opened for a short time immediately after determining z so that the chamber and the cylinder are open to the outside air. The piston is moved so that the scale for cadmium red reads 832.2. The valve is then turned so that the piston and enclosure are connected and both closed to the outside air.

The dispersion prism is adjusted for cadmium red and the fringe system associated with the vacuum chamber is adjusted horizontal by rotation of the double prisms by means of a hydraulic fine adjustment. The piston is moved in one direction or the other until a whole order is indicated for the vacuum chamber. By moving the scale in the appropriate direction the index is alined with number 832.00 or 833.00.

Changing to cadmium green the piston is moved so that a whole order is indicated in the vacuum chamber pattern. In this position the index should coincide with a whole number on the green scale such as 1062.00. If this is conspicuously not the case, the scale is moved one order right or left in cadmium red and the fringe pattern for cadmium green again checked for coincidence. The index should also align with whole numbers, for example, 1129.00, 830.00, and 952.00, respectively, on the cadmium blue, krypton red, and krypton yellow-green scales when the fringe patterns are brought into coincidence. Then the index, for any position of the piston, indicates the correct number of fringes corresponding to an air column 1 m long.

If the enclosure is subsequently opened, the position of the scale is incorrect and must be readjusted.

Measurement of a long gage block, 500 mm for example, is made in the following manner:

After the gage block with a steel flat wrung to one end has been inserted in the enclosure and the equipment has come to temperature equilibrium, the scale is adjusted as previously described. The temperature of the block is then measured. The dispersion prism is set for krypton red and the fringe pattern of the gage block is appropriately oriented with a hydraulic fine adjustment. The piston is advanced until the fringe pattern formed by the reference mirror and the gage block coincide. The reading of the index on the krypton red scale is noted. A similar reading is made using krypton yellow-green. The temperature of the gage block is read a second time. The values of the index on the scale are multiplied by 0.50000 for a 500-mm block. The decimal complements of the vacuum fractions are converted to units of length. With the aid of a table of nominal excess fractions for krypton red and yellow-green the nearest coincidence is found. The value obtained with the krypton yellow-green

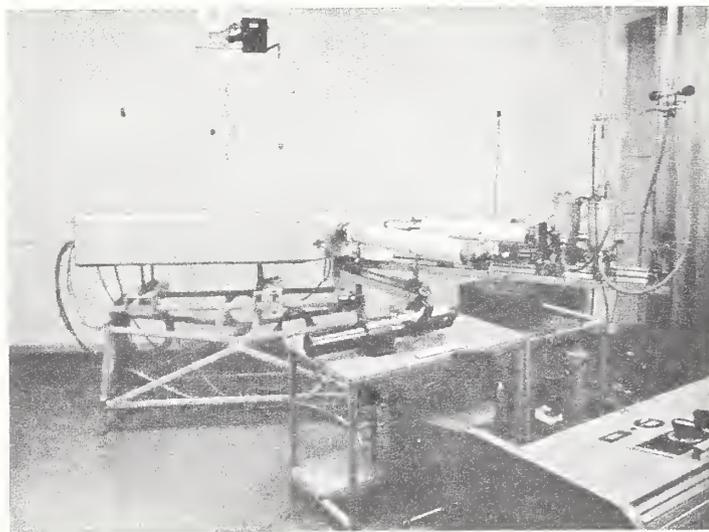


FIGURE 7.

is used as the length of the gage block because of the superior quality of this line.

It should be noted that the measurement of barometric pressure and air temperature for the alinement of the scale in no way signifies that the index of air must be accurately known as these data serve only to give an approximate index of refraction.

Figure 7 is a view of the complete equipment of the PTB for the measurement of gage blocks up to 1 m by means of wavelengths in vacuum. The thick-walled, airtight, aluminium enclosure covered with cork for thermal insulation is seen on the left. On the observer's table, in the middle ground, the piston is mounted, the scale on the piston being read by means of an eyepiece. A telescope of long focal length with an autocollimating eyepiece is needed for the observation of the fringes. On the right, in the background, the lamp houses for the lamps and a collimator with a powerful dispersion prism system are positioned. A small high-vacuum pump to evacuate the vacuum chamber is located at the side of the observer's table.

Figure 8 shows the opened chamber. The double prism is located at the left; the vacuum chamber is located along the middle axis of the trough; the reference mirrors for the chamber are on the right; a 500-mm block with an auxiliary steel plate wrung to it is on the side of the chamber; and the reference mirror for the measurement of the gage block is on the other side. All necessary adjustments are made from the outside by hydraulic controls.

Figure 9 is a view of the scale on the piston with the index indicating the fringes referring to the refraction of air. There are five graduated scales corresponding to five colors.

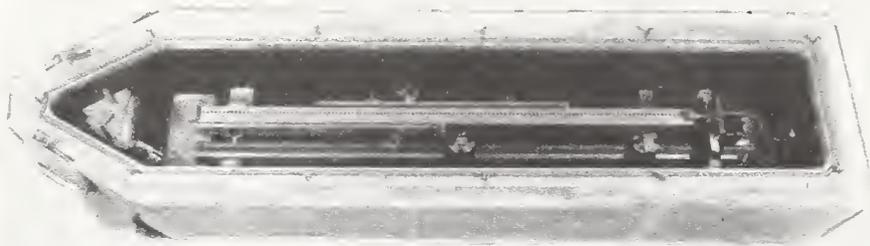


FIGURE 8.

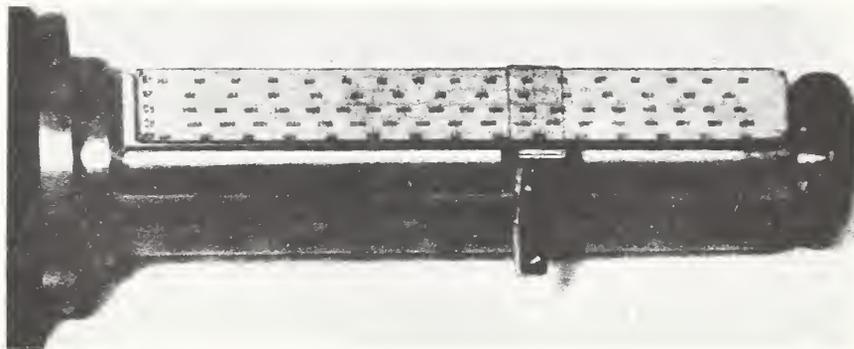


FIGURE 9.

This apparatus was designed by Kösters more than 20 years ago. It is unique at present but a new instrument, taking into account the experiences of the past 20 years, is now under construction at Carl Zeiss in Western Germany. It will be available in about 2 years. This apparatus will presumably play an important part in the future, because it makes possible the measurement of gage blocks to a very high accuracy by means of wavelengths in vacuum. This will be essential in the future, as the primary standard of length will probably be a wavelength of light in vacuum.

Another instrument designed by Kösters, quite similar in principle, but especially designed for the measurement of gage blocks up to 100 mm was built by Zeiss. Several of these instruments were completed at the end of the war but were dismantled by the Russians. This apparatus is now under construction in Germany and it is expected

that it will become the most suitable equipment for the measurement of industrial gage blocks up to 100 mm, especially for the use of gage-block manufacturers.

The maximum accuracy required for the apparatus described in the foregoing is of the order $\pm 0.01 \mu$ ($0.4 \mu\text{in.}$). Naturally, this implies that all uncertainties must be reduced to a minimum. The degree of uncertainty in the measurements made by laboratories of a number of countries is illustrated by an international comparison of gage blocks organized by the BIPM (International Bureau for Weights and Measures) some time ago. The differences among the results of several official institutions and the BIPM were found to be about $\pm 0.05 \mu$ ($2 \mu\text{in.}$). Inquiring into the reasons for these differences which considerably exceed the accuracy possible using the wavelength measurement technique, one finds repeatedly the same sources of error.

The most common error is due to the uncertainty in the measurement of temperature. The thermal coefficient of steel commonly used



FIGURE 10.

for gage blocks, is about $10 \mu/\text{m}/\text{deg C}$. This means the uncertainty in the measurement of temperature should be less than $\pm 0.01 \text{ deg}$ if the required accuracy in length is $\pm 0.01 \mu$ for a gage block 100 mm long. To place a mercury thermometer near the gage block is quite inadequate in this case. According to our experiences, the simplest and best method to obtain higher accuracy in measuring temperature by means of Hg-thermometers is to place a large metal block near the apparatus, the thermometer being placed in a bore of the metal block and the difference of temperature between the metal block and the gage block being determined by thermocouples. Naturally, the thermometers must be calibrated from time to time. Temperature measurements accurate to $\pm 0.02 \text{ deg C}$ can be obtained in this manner. This corresponds to $\pm 0.02 \mu$ for 100 mm or 0.2μ for 1-m length. If higher precision is required, a platinum-resistance thermometer is used at the PTB, the difference in temperature between the platinum-resistance thermometer and gage block being determined by one or more thermocouples. Equipment for electrical measurement of temperature by means of a platinum-resistance thermometer is shown in figure 10. An electrical potentiometer, used to measure

the electromotive forces of the platinum-resistance thermometer and the thermocouples, is shown in the center of the table. A galvanometer of high sensitivity is mounted inside the table. From time to time, i. e., several times a year and at least before every important length measurement, the platinum-resistance thermometer is checked in the usual manner by determining the triple-point of water. The uncertainty in determining this temperature usually is not greater than ± 0.0005 deg C. For a single measurement near 20 deg C the uncertainty is about ± 0.002 deg C and thus the mean value for two or more single values is accurate to nearly ± 0.001 deg C, which corresponds to an error in length of ± 0.01 μ /m.

Next to temperature the refraction of air is the most important source of error in length measurements based on light-waves. A method has previously been described to avoid this error by elimination of the refraction of air. If that is not feasible, an accurate barometer which has been checked under all conditions and calibrated periodically should be used. An error in barometric pressure of ± 0.2 mm Hg corresponds to an uncertainty in length of nearly ± 0.05 μ /m.

Other common sources of error originate in the properties of the interference wedge generally used for length measurement by light waves. Such an error arises from the inevitable inclination of the interference wedges and is equivalent to the well-known inclination error in length measurement. Inclination errors arise in length measurement, for example, if two gage blocks or two scales being compared are inclined relative to each other. In these cases the result of the length measurement is $L \cos \varphi$ instead of L , L being the real length, φ being the angle of inclination. If φ is very small, $L \cos \varphi$ is nearly equal to $L[1 - (\varphi^2/2)]$, the error in length due to inclination being $-L(\varphi^2/2)$. This is the well-known second order inclination error which also arises in measuring length by interference wedges. The condition for elimination of the inclination error consequently is $\varphi = 0$, meaning in practice that the observer's slit must be so adjusted that one looks exactly perpendicularly at the interference wedge. This is commonly done by means of an autocollimating eyepiece, which is quite essential for such apparatus. If the observer's slit deviates by the distance r from the normal direction, φ becomes equal to r/f , f being the focal length of the objective lens (see fig. 6). The inclination error, therefore, is $(-Lr^2)/(2f^2)$. Assume r to be 1 mm and f about 400 mm, then the error for a 100-mm gage block would be $-100/2 \times 160,000$ or nearly equal to -0.3 μ (1.2 μ in.). This indicates that the interference wedge or, in practice, the gage block surface must be exactly adjusted perpendicular to the light beams.

Strictly speaking this adjustment cannot be exactly accomplished for all beams reaching the observer's eye. Due to the finite size of the observer's slit there are also visible beams reflected in directions making an angle φ with the perpendicular beam. Thus most beams, except the central beam perpendicularly incident, give rise to an inclination error $-L(\varphi^2/2)$ and by superposition of all beams of different inclinations coming through the observer's slit a lack of sharpness of the fringes results, increasing to such a degree that the fringes may disappear entirely if large slits are used. In addition a general shift of the fringes or an error in length occurs. It is very

easy to calculate that this error for a circular slit is given by the expression:

$$e = -L \frac{D^2}{16f^2},$$

L being the length of gage block being measured, D the diameter of the circular slit, and f the focal length of the objective lens. For a rectangular slit, the error is given by

$$e = -L \frac{b^2 + h^2}{24f^2},$$

b and h being the breadth and the height, respectively, of the rectangular slit. To compensate for this error, a plus correction of the same amount must be made. This correction is often neglected, but assuming the breadth and the height of a rectangular slit to be 2 and 0.5 mm, respectively, and the focal length 400 mm, the correction would be

$$-e = +L \frac{0.5^2 + 2^2}{24 \times 400^2} \sim +L \times 10^{-6},$$

hence the correction in measuring a gage block of about 100 mm is nearly $+0.1 \mu$ (4μ in.). Therefore, a slit as small as possible should be used not only to avoid errors due to fringe shift but also to avoid loss of fringe sharpness. A circular slit 0.5 mm in diameter should be sufficient as the correction for this slit does not exceed about $+0.01 \mu$ for a 100-mm gage block when a focal length of 400 mm is used.

Another error may arise when measuring gage blocks by light waves in consequence of the definition of the length of a gage block. According to a recommendation of the ISA (International Standards Association) the length of a gage block is defined by the distance of one surface of the gage block from the surface of an auxiliary plate wrung to the other surface of the gage block, the plate having the same surface finish as the gage block. Corrections must be made if these conditions are not fulfilled. Occasionally gage blocks made of steel are wrung to plates of glass or quartz in order to judge, by viewing through the plates, whether the gage blocks are wrung completely. With glass or quartz plates errors arise due to the difference of the phase change of light. If light waves are reflected from a surface, they suffer a change in phase as if the plane of reflection were not identical with the mechanical surface. There is a difference in the optical properties of glass or quartz and steel, the first two being insulators and the last an electrical conductor. From optics it is well known that the phase change on insulators is π and on metals $\pi - \epsilon$, π corresponding to $\lambda/2$ in phase or $\lambda/4$ if interpreted in terms of the length of a gage block. Therefore, $\pi - \epsilon$, being the phase change on a metal such as steel, corresponds to a length of $\lambda(1 - \epsilon/\pi)/4$, ϵ being a material constant, depending in general on the color. Hence it is easy to see that gage blocks measured by light waves seem to be too short by $\lambda\epsilon/4\pi$ when wrung on an insulating plate such as glass or quartz. At the PTB we have determined this value in different ways and have found it to be very nearly -0.018μ for all colors.

If we measure a gage block wrung to glass or quartz we use a correction of $+0.018 \mu$ ($0.7 \mu\text{in.}$).

A further correction related to the surface quality, i. e., to the roughness, is applied at the PTB to all measurements of gage blocks by light waves. Assume the value of roughness for the surface of the gage block to be ρ_1 , and for the surface of the auxiliary plane wrung to the gage block ρ_2 , the error in length would be $-(\rho_1 - \rho_2)$ and a correction by $+(\rho_1 - \rho_2)$ would be necessary. Naturally the value of roughness ρ cannot be any of the usual units of roughness, for example, root mean square. We have determined the values in question in different ways, for example, in the following manner (fig. 11). Assume three gage blocks of different roughness are

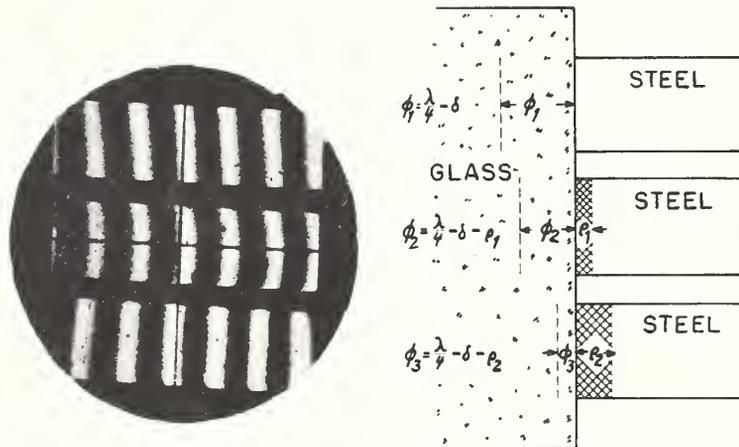


FIGURE 11.

wrung on a glass plate and an interference wedge is formed by the glass surface to which the gage blocks are wrung and a virtual image of a suitably located real mirror. This can be done by means of a Kösters interferometer, the only difference between this arrangement and an ordinary gage block measurement being that the gage block surface wrung to the plate is viewed through the plate. On the left of figure 11 the field of view to be observed in this case is shown. The upper field on the left represents a gage block surface of the highest possible finish; the shift of the fringes on the gage block surface relative to the fringes on the plate corresponds to the phase change on a steel surface of only $\psi_1 = \lambda/4 - \delta$. The middle field and the lower field refer to gage block surfaces of roughness ρ_1 , and ρ_2 , respectively. The fringes on the steel surfaces are shifted to the left by ρ_1 and ρ_2 with respect to the position in the upper field. Hence, the values for ρ_1 and ρ_2 can be easily determined.

If many gage blocks are to be measured, it is not practical to determine the roughness in this way. Therefore, we have developed a method for the determination of the roughness correction by measurement of the light scattered by the gage block surfaces. The design of the apparatus used for this purpose is shown by figure 12. The apparatus consists mainly of an Ulbrichts' sphere which is commonly used for measurement of light flux. From an objective lens a slightly convergent flux of white light is transmitted into the sphere through a hole. Light is reflected from the surface of a gage block placed in

another small circular hole of the sphere. Specularly reflected light leaves the sphere by a third hole. Scattered light R_d is integrated by the Ulbricht's sphere and measured by means of a photoelectric cell. If the hole at the bottom is closed by a flap, the total amount of

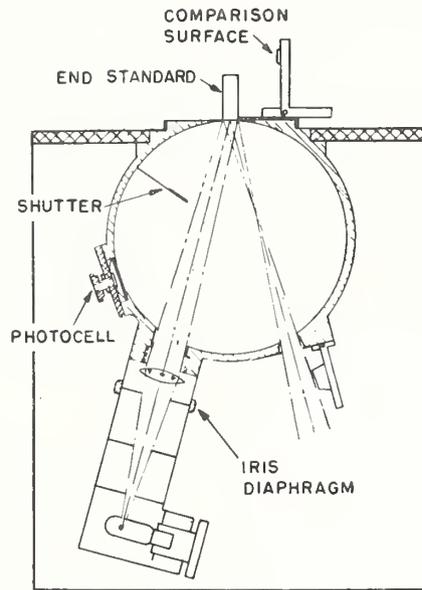


FIGURE 12.

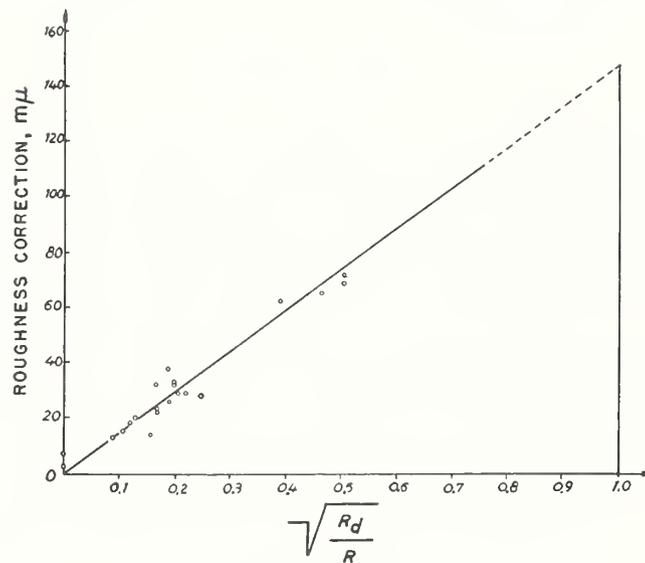


FIGURE 13.

reflected light R , both specularly reflected and scattered light, is indicated by the photoelectric cell. In this manner we measured a number of gage block surfaces. The roughness corrections for the same surfaces were determined by interference measurements. In comparing the results we found a simple relationship between the reflection measurements and the values of roughness (fig. 13). The abscissa represents the values $\sqrt{R_d/R}$, R_d being the scattered and

R the total reflected light as measured by the Ulbrichts' sphere. The ordinate represents the values of roughness in millimicrons as determined by interference. The curve indicates that there seems to be a linear relationship with the deviations from linearity being less than about $10m\mu$. Thus, it seems possible to determine the roughness corrections by measurement of the ratio of light scattered to that specularly reflected from the gage-block surfaces. The surfaces of every gage block measured by means of light waves are checked in this manner, the roughness correction being read off directly from the scale of a galvanometer indicating the photoelectric current. Thus, we get corrections due to roughness in a very short time. The total range of the observed roughness correction is, in practice, nearly 0.1μ ($4\mu\text{in.}$). Even for the same manufacturer a range of 0.05μ is found in some cases. This indicates that corrections for roughness are very essential, if an accuracy in length measurement approaching $\pm 0.01 \mu$ is required.

Having discussed errors associated with external influences such as temperature and the conditions of air and with the optical conditions such as inclination, aperture correction, phase change, and roughness, we still need to consider the part played by the lamps which are essential to the formation of light-wave scales as represented by interference wedges. The question is, in what degree or in what manner are light-wave scales influenced by the characteristics of the lamps? At the PTB a great deal of the metrological work is concerned with this task, but time will not permit a discussion of the details.

Nearly 80 years after Michelson started his famous investigations of light-wave scales, spectroscopists and metrologists have reached an understanding of certain deficiencies of light-wave scales.

There are two essential characteristics to be considered. First, there is an immense number of wavelengths obtained from lamps because they are produced by an aggregate of atoms, each being in motion relative to the observer and each interacting with the others.

Second, the materials—gases or vapors—which are electrically excited in the lamps, are, in general, not homogeneous with regard to nuclear structure. Most of the materials used for light emission in lamps are mixtures of isotopes, the light-waves being modified by their different nuclear structures.

Therefore the following conditions for establishing light-wave scales of highest accuracy are desired:

First, low temperature and high mass of the light emitting atoms to meet the requirement that the atoms be as nearly at rest as possible.

Second, low pressure of the gas or vapor in the lamps and absence of external electric or magnetic fields, to meet the requirement that the emitting atoms be uninfluenced by secondary disturbances.

Third, the use of gases or vapors consisting of a single isotope of even mass only, in order to reduce errors of subdivision.

In the United States a lamp filled with mercury isotope 198 and excited by high-frequency voltage has been developed which fulfills these conditions very well. At the PTB we have designed a Geissler hot cathode type of lamp filled with krypton isotope 84 or 86 (fig. 14). The lamp is cooled with liquid air to the triple point of nitrogen, i. e., 63°K. For this purpose we use an airtight metal container. Figure 15 shows the container with the cover to which the lamp is fastened. A Dewar vessel is placed in the container. After having closed the

container with the cover and having filled the Dewar vessel with liquid air (fig. 16) the container is connected to a vacuum pump to lower the pressure of the liquid air in the Dewar vessel (fig. 17). The triple point of nitrogen is reached in this manner in a short time. At

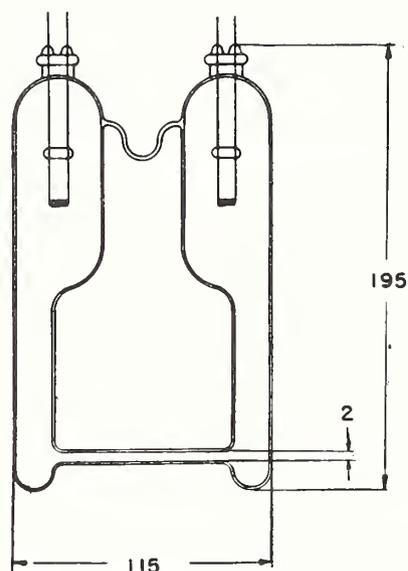


FIGURE 14.



FIGURE 15.

this point the vapor pressure of krypton, frozen at about 120° K, is only a few hundredths of a millimeter. Thus, very low krypton pressure is reached. The lamp can be filled with any pressure desired, the krypton pressure being determined by the temperature of liquid air only. According to our experiences it is possible to obtain light wave scales of incontestable exactness of subdivision with this lamp.

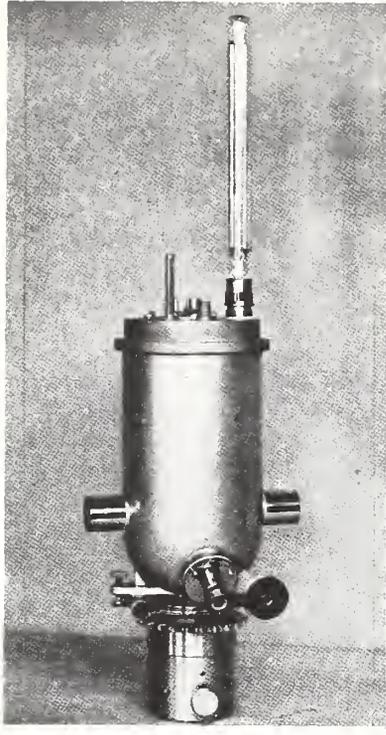


FIGURE 16.

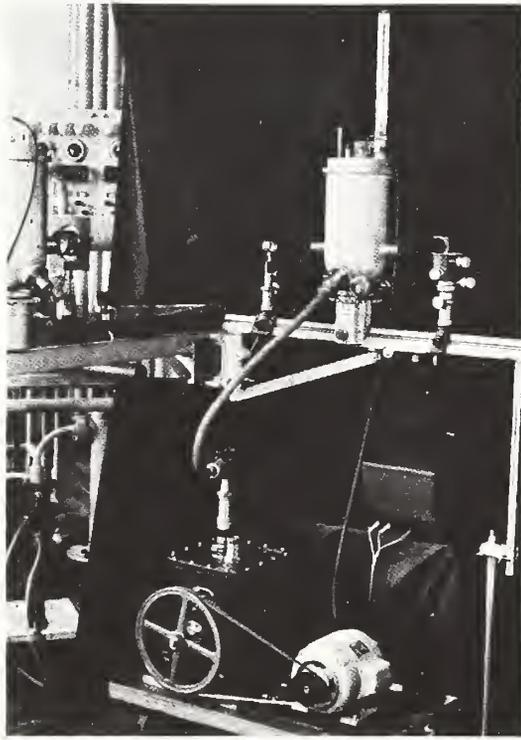


FIGURE 17.

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The longest scale to be realized is nearly 800 mm including more than 2,000,000 graduation lines. The uncertainty of length at any point of the scale is less than $\pm 0.01 \mu/m$.

The necessity of using lamps filled with isotopes of even mass requires the production of pure isotopes in adequate quantities. In the case of mercury-198 this is done by bombarding gold-197 with neutrons. Krypton isotopes can be separated by thermodiffusion as was done by Clusius and Dickel. A great deal of our work at the PTB during the last year was spent in building a thermodiffusion column for producing Kr-84. Thermodiffusion occurs between a hot and a cold plate positioned face to face, having a mixture of heavy

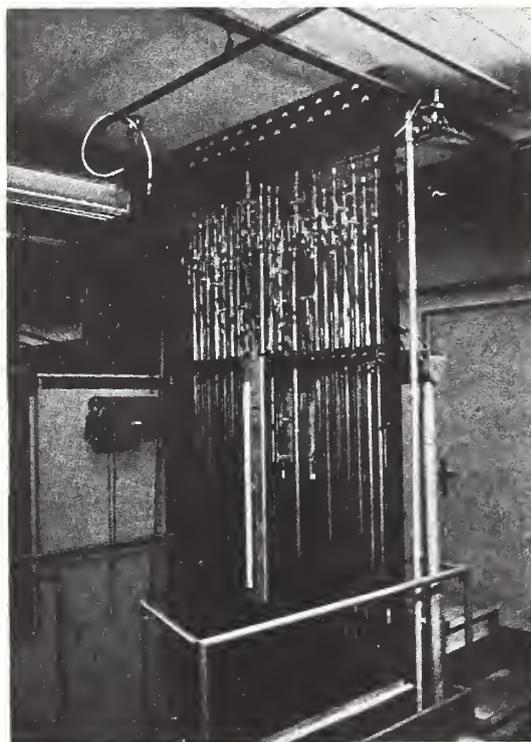


FIGURE 18.

and light particles between them. Heavy particles then diffuse to the cold plate and light particles to the hot. Clusius modified this arrangement by putting an electrically heated wire on the axis of a vertical tube of about 10-mm internal diameter and several meters length. This tube is filled with gas, krypton for example, and cooled externally with water. Thermodiffusion then takes place between the hot surface of the electrically heated wire and the water-cooled wall of the tube. Near the wire there is an upward flow of the heated gas, whereas near the wall the gas flows downward. Thus, due to the combined effects of diffusion flow and convection flow the lighter atoms are carried inward and upward and the heavier ones, conversely, outward and downward. Naturally the tube must be long to get complete separation of isotopes. The upper part of the equipment we have assembled for the separation of Kr isotopes is shown in figure 18. This column has an effective length of 72 m and consists of 23

single tubes made from copper, each 6 m long, which are connected together by circulating systems consisting of tubes of small diameter, one link of each system being heated so that a convection flow arises in the system conveying material from the upper end of one tube to the lower end of the next tube. This equipment can produce about 1 liter of Kr-84 with a concentration of more than 99 percent in 6 months. This is sufficient to fill several thousand lamps.

By utilizing pure isotopes in the production of lamps, the light-wave technique of length measurement now seems to have reached the highest possible perfection. Undoubtedly, light-wave scales as realized by means of isotope lamps are much more precise than the old-fashioned meter prototype which is the legal standard of length. It seems to be the intention of most of the industrial countries to relegate the meter prototype to the museum and to replace it by a light-wave scale which is limited in accuracy only by the laws of nature. Whether or not we succeed in doing that in the near future, light waves emitted by atoms will be more and more the actual length standards of industry. It is very instructive to follow the development of length units during the history of mankind. In prehistoric times the limbs of the human body were the common length units. After the creation of state authorities these units were superseded by arbitrary units symbolizing the sovereign power. These predominated in principle many thousand years. It is noteworthy that the rise of new ideas regarding a return to a natural length unit in the eighteenth century coincides with the abolition of dynastic power and with the creation of new public authorities. It is very attractive from the mental point of view that the meter is a child of the French Revolution, in its original state being connected to the globe, allegorizing on the one hand the universal political ideas of that period and demonstrating on the other hand the high level of geodesy in the eighteenth century. Revolution was followed by restoration of dynastical power, and curiously enough the meter was degraded at the same time to an arbitrary unit, lingering in this antiquated form until now. So the units of every epoch seem to be characteristic, to a certain degree, of the spirit of the age. Now we seem to stand at the beginning of a new era, the age of democracy and the age of the atom. Doesn't it seem to be a matter of course that we shall abolish the autocratic meter prototype by designating a new length unit, one that can be used anywhere in a real democratic manner and that we take our measures from the atom so significant to our century?

2. Calibration of Gauge Blocks in Canada

By K. M. Baird ¹

I should like to describe briefly the methods and equipment that are used at the National Research Laboratories of Canada for the calibration of end standards for industry. Although the basic principles of our methods are the same as used elsewhere, the way in which they are applied is, of course, governed by our particular needs in Canada and by the functions of our laboratory. Therefore, I shall first say something of these in order to indicate why our procedures are not quite the same as those used elsewhere.

There are two main reasons why our procedures differ somewhat from those of the national laboratories of larger industrial countries: In the first place industry in Canada is generally on a smaller scale and less highly developed than in countries like the United States or Great Britain. Consequently, the number of gauges that are calibrated is not large, about 1,000 a year at present, and we do not have to be equipped for testing in large quantities.

In the second place, the National Research Laboratories have not been called upon to do routine testing to the extent that other national laboratories have. Although its Applied Physics Division has among its responsibilities the maintenance of Canada's national units of measurements, the main emphasis is on their fundamental aspect, and the provision of reference standards on which routine tests can be based. The main work of the Division is concerned with research investigations likely to be helpful to the development of Canada's resources and industry.

The work of the Interferometry Section, which has charge of the unit of length insofar as it involves wavelengths, consists mostly of research concerning techniques of measurement, new sources of radiations useful for interferometry and investigation and measurement of wavelengths proposed as the new standard of length. The section also calibrates end gauges for industry but in general these are to be used as reference standards; it is not often required to make tests as to conformity to any particular specifications.

Because of these two reasons our procedures are suited to relatively small numbers of tests under the supervision of people who are also engaged in research, and our equipment is designed from the point of view of versatility rather than ordinary quantity testing.

End gauges are all measured by absolute calibration against wavelengths of light. Comparisons with steel reference standards are made only in exceptional cases.

Gauges up to 100 mm, or 4 in., in length are measured in the interferometer shown in figure 1. This was designed and built in the laboratory. It is of the Fizeau type and is somewhat similar in optical

¹ National Research Council, Ottawa, Canada.

construction to the interferometer in use at the British National Physical Laboratory. Light from a suitable source is collimated by a lens at the left end of the tube, T; it then passes through a dispersing prism in the rectangular box, M, and is reflected down onto the quartz reference flat and the gauge, G, which is wrung down onto another quartz flat. Light is returned through the dispersing system and collimator lens to the eyepiece, E, through which the interference fringes are viewed.

A general view of the interferometer in use is shown in figure 2. It is mounted in a room in which the temperature is controlled at 20°C to within ± 0.1 deg C. A mercury-198 source can be seen at

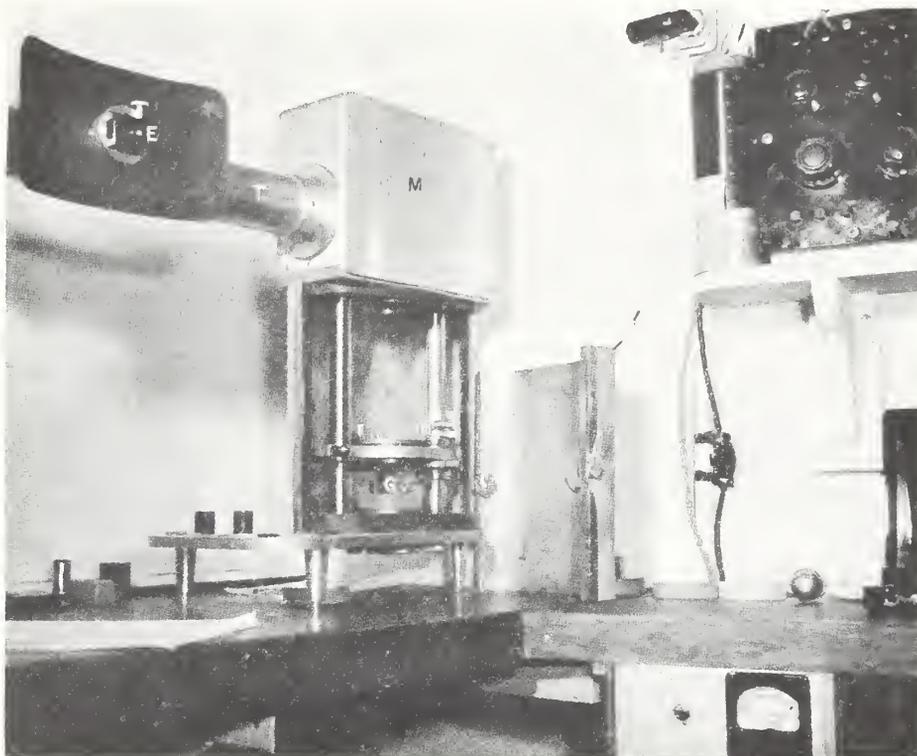


FIGURE 1. *Fizeau-type interferometer.*

the left; this has now been replaced by an arrangement whereby several alternative sources, including Hg^{198} and cadmium can be conveniently interchanged. A potentiometer bridge seen at the right is for the measurement of the temperature of the gauge by means of thermocouples, one junction of which is clipped to the gauge, the other placed in an ice bath. Temperature measurements are considered accurate to ± 0.025 deg C.

The procedure in the measurement of a set of gauges is as follows:

Gauges are wrung onto several quartz flats, two to a flat, and set on the bench to come to temperature equilibrium. One flat at a time, with its gauges, is placed in the interferometer whose cover is closed; further time is allowed for final temperature equilibrium. During this time the observer calculates from his observations the length of the gauge measured just previously and wrings up a new pair of gauges to replace those just measured.

Observations are made of the fractional part of the order of interference for four colors of the cadmium spectrum with gauges less than 1-in., or 25-mm, length and of the Hg^{198} spectrum for longer gauges up to 4-in., or 100-mm, length. The length of a gauge at the center of its face is determined from these values by the use of prepared tables.

Corrections to the observed length are made as follows:

The gauge length at 20.0°C is calculated from the observed deviation from this temperature; it is assumed that the coefficient of dilatation of the gauge is the same as for a typical steel from which the gauge is made. Because the temperature is controlled to within

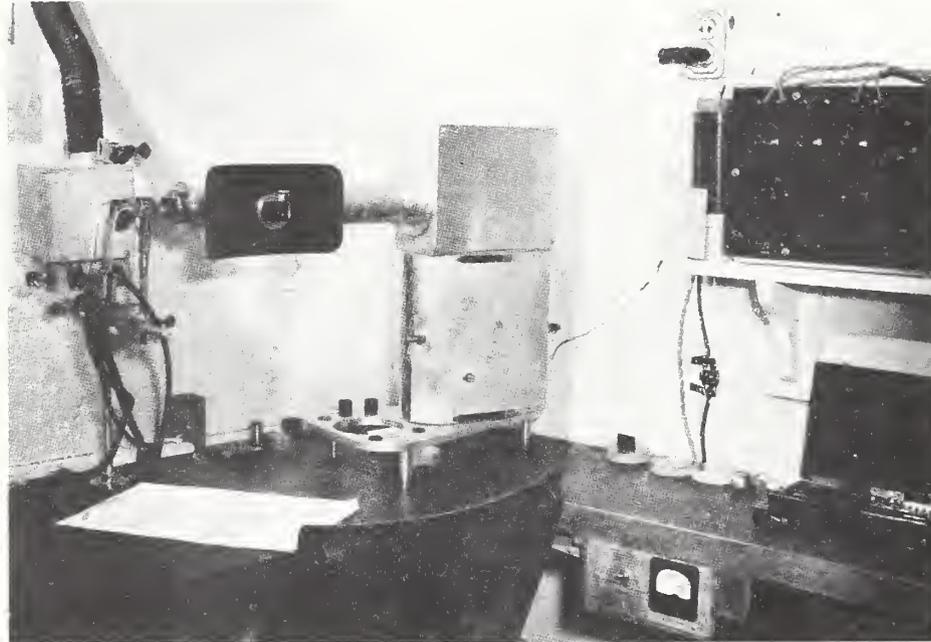


FIGURE 2. General view of interferometer, light source, and potentiometer bridge.

0.1°C of 20.0°C no significant error occurs. The dilatation of other materials is measured.

The corrections due to the refractive index of air are made by the assumption that it is the same as for normal air at the temperature, pressure and relative humidity at which the observations are made. The pressure is read on a sensitive barometer, the temperature is assumed to be the same as the gauge and the relative humidity 35 percent. Tests have been made occasionally by means of a sensitive refractometer and have shown that no significant error results from this cause.

The phase change on the surface of the block is determined by comparing the observed optical length of a wrung combination of about five blocks, taken at random from a set, with the sum of their optical lengths. From the difference is obtained a correction which is applied to the observed optical lengths of all the gauges in the set to give their physical lengths.

With this procedure a single observer is able to make an absolute calibration of about 20 gauges per day.

Standards longer than 4 in., or 100 mm, and up to 40 in., or 1 m, in length are measured on the larger interferometer shown in figure 3. This was also designed and built at the National Research Laboratories. It is of the Michelson type and normally has a cover for temperature stability which is not shown in the illustration. This interferometer was designed with emphasis on applicability to other research work and not for routine calibration in quantity.

Light from suitable sources which are mounted behind the monochromator, M, is passed through the monochromator and reflected into the collimator, C, to the beam splitter, S, from which one beam

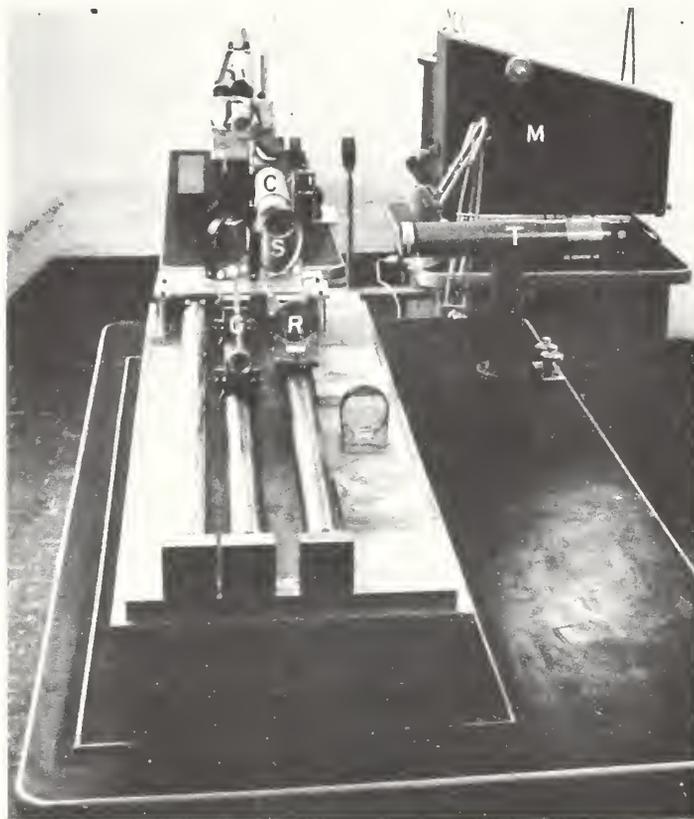


FIGURE 3. Michelson-type interferometer for long lengths.

goes to the reference mirror, R, the other onto the gauge, G, which has a flat wrung to its rear gauging surface. Interference fringes formed on recombination of the beams at S are observed through the telescope, T. Leveling is done hydraulically by means of the control knobs under the telescope.

Gauges up to 20 in., or $\frac{1}{2}$ m, in length are calibrated in one step by comparison with seven wavelengths emitted by a Kr isotope lamp operated at the temperature of the triple point of nitrogen. Longer gauges are calibrated by measuring the difference between their lengths and those of shorter gauges. Gauges of 12 in. and less can also be checked against the wavelengths of Hg¹⁹⁸. Both these single isotope sources are made at the National Research Laboratories where investigations are being made as to their suitability for length standards.

Corrections to the observed gauge lengths are made in a similar way to that described for the gauges of 4-in. length and under.

Incidentally, the apparatus that can be seen in the background of figure 3 is a new instrument under development for the calibration of line standards or scales directly in terms of wavelengths of light.

In conclusion I shall say a few words about our accuracy of measurement. In our routine measurements on good gauges we guarantee an accuracy of plus or minus two millionths of an inch for gauges of 1 in. or less (or the equivalent in metric gauges) and plus or minus two parts per million for longer gauges. We feel that this limit is mostly imposed by the nature of the gauges. We do not contract to measure to a lower accuracy (in any case it is generally not any easier) but sometimes quote a lower accuracy on the lengths reported where the gauge quality makes the higher accuracy of no significance. It is possible to measure any given length to much better accuracy than the above, but we do not feel that one is justified in giving to any higher accuracy a general statement as to the length of a steel block which may be used in any number of ways, e. g., in a wrung combination, between points of a comparator, for optical comparison, etc.

If asked, we would calibrate a set to higher accuracy in special cases but then would wish to know how the gauge was to be used and would state the length that was significant for this purpose. A statement, in a general way, as to the length of a steel block to plus or minus tenths of millionths of an inch has not much more significance with present steel gauges than has the length of, say, a piece of rubber given to an accuracy of thousandths of an inch.

3. Use of Light Waves for Controlling the Accuracy of Block Gauges

By F. H. Rolt ¹

Gentlemen:

I feel it a distinct honor to have been invited to offer you a lecture today on the "Use of Light Waves for Controlling the Accuracy of Block Gauges." Unfortunately, I cannot deliver this lecture to you in person, but Mr. E. J. Schneider ² has kindly offered to present it in my place.

As you all know, the manufacture of block gauges calls for the very highest degree of mechanical precision on the following features:

(1) The working faces must be flat to obtain the necessary degree of adhesion when the gauges are wrung together.

(2) This flatness must include a very high degree of surface finish of the faces.

(3) The faces must be parallel to each other to ensure equality of size at all positions between the faces.

(4) The actual size of a gauge, represented by the distance between its faces, must agree with its nominal size to within a very small tolerance.

To minimize burring in use, a further requirement is that there must be no sharp edges round the faces; beveling is not sufficient; the edges must be very carefully rounded off.

Now, the flatness and reflectivity of the faces of block gauges lend themselves admirably to the application of the principles of optical interference as a means of controlling the accuracy of these gauges to all the above requirements. As you know, the use of optical interference provides, in a sense, a series of finely divided scales of natural origin, the divisions of which are spaced perfectly regularly and at intervals that are known very precisely both in inches and millimeters. The divisions of these scales are approximately 0.00001 inch apart, and as they can be subdivided without difficulty into tenths it is possible, with them, to achieve an accuracy of one millionth of an inch in the examination of block gauges.

Various types of instruments based on the use of optical interference have been designed from time to time over the last 30 to 40 years for inspecting block gauges, and it is proposed in the short time I have available to offer you some general account of a few typical instruments of these types without entering too deeply into the detail of their construction.

Monochromatic Light

Interferometers in general make use of what is known as monochromatic light, that is light of a specific color or wavelength. Monochromatic light can be produced from a number of different sources

¹ Consultant in Metrology and Standardization, Hampton, England.

² Engis Equipment Co., Chicago, Ill.

in the form of electrical discharge lamps, the most familiar of which contain either cadmium, mercury-isotope 198 or krypton. These particular lamps have the property of producing a number of very pure monochromatic radiations, the wavelengths of which have been determined very accurately in various laboratories throughout the world.

Optical Interference

The phenomenon of optical interference has, of course, been known for a very long time. It is the term applied to the combined effect of two rays of monochromatic light of the same wavelength which, emanating from a single source, are reunited after being separated and made to travel over paths of different lengths.

As you all know, a very simple case of optical interference can be observed with an optical flat supported over a plane reflecting surface

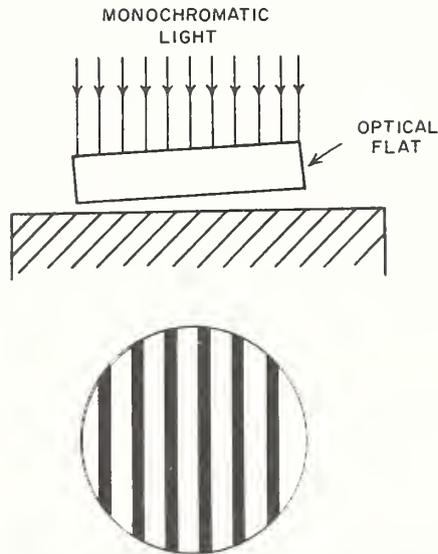


FIGURE 1. *Interference fringes formed between a flat reflecting surface and a slightly inclined optical flat.*

so as to enclose a slightly tapered air gap, as in figure 1. When illuminated normally by a parallel beam of monochromatic light, such a system is found to be traversed by a series of bright and dark fringes that are straight, equispaced, and parallel to the line of intersection of the two plane surfaces, as shown in the lower diagram.

This very simple optical system is the basis of all the interferometers used for testing the accuracy of block gauges as regards the flatness, parallelism, and surface finish of their faces, and for measuring actual size. For the purpose of all these tests the faces of the gauges are used as the lower reflecting surface of the system, and a series of straight bands or fringes, as they are called, is formed between the gauge face and that of a suitably disposed optical flat in a parallel beam of monochromatic light.

Tests for Flatness and Parallelism of Block Gauge Faces

A schematic diagram of an interferometer set up at the N.P.L. at Teddington in 1921 for inspecting the flatness and parallelism of the faces of block gauges is shown in figure 2. In this instrument the

source of illumination is a mercury vapor lamp and a green filter is used in order to separate its strong green monochromatic radiation. The light from this lamp, after being concentrated on a pinhole, is rendered parallel by means of a collimating lens and illuminates the gauge that is wrung down on to a baseplate. Fringes are formed on the upper surface of the gauge and on the baseplate by means of an

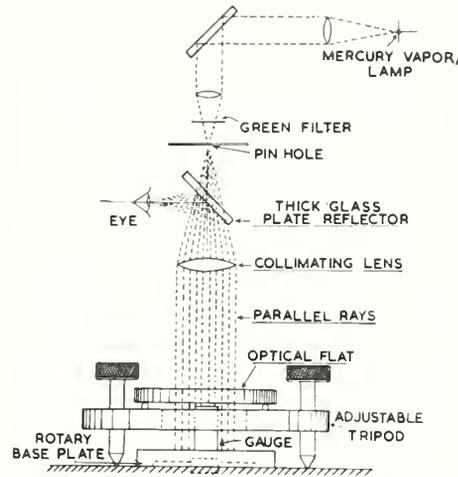


FIGURE 2. Diagrammatic optical arrangement of N. P. L. interferometer for checking flatness and parallelism.

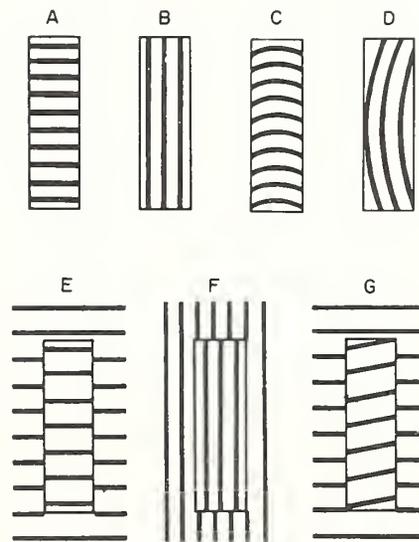


FIGURE 3. Types of interference fringes formed on surfaces of gauges and baseplate.

optical flat that is supported just above the upper surface of the gauge. If the surface of the gauge is perfectly flat the fringes will be quite straight and equispaced, as shown at A or B in figure 3. If the gauge face is not flat, even by only a microinch, it is revealed by a corresponding bowing of the fringes, as at C or D. The actual error in flatness of a gauge surface can readily be estimated from the amount of curvature of the fringes in terms of their spacing which represents a scale of approximately 0.00001 inch.

The parallelism between the two gauge surfaces is examined by noting the degree of parallelism between the two sets of fringes, one formed on the upper surface of the gauge and the other on the surrounding surface of the baseplate to which the gauge is wrung. Diagrams E and F in figure 3 represent cases of gauges with truly parallel surfaces. Diagram G, on the other hand, is what would be seen in the case of a gauge that has a transverse lack of parallelism

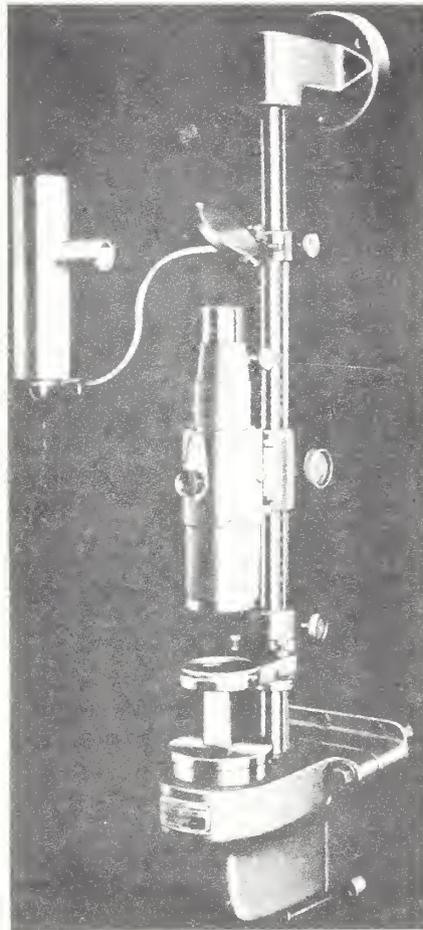


FIGURE 4. *N. P. L. type interferometer for inspecting flatness and parallelism of gauge faces.*

between its faces amounting to about a third of a fringe, that is approximately 3 microinches.

The N.P.L. type of interferometer for carrying out such tests on the flatness and parallelism of gauge faces has been made for several years jointly by Coventry Gauge & Tool Co. Ltd. and Hilger & Watts Ltd. A photograph of one of these instruments is shown in figure 4.

Tests for Surface Finish

When one carefully examines the fringes formed on the surface of an ordinary block gauge in an interferometer of the type just referred to, it will be seen that their edges are not perfectly straight. They

present a very finely serrated appearance and this serration can be used as a criterion of the surface finish of the gauge under examination.

The type of instrument used for examining the surface finish of block gauges is known as an interference microscope. Generally speaking these instruments employ much the same optical system as in the flatness interferometer, but in order to see the serrated effect more clearly the fringes are viewed through a microscope.

The first interference microscope was designed by the Russian scientist Linnik. In the latest development of the Linnik instrument, Carl Zeiss provides three degrees of microscope magnification of 80, 200, and 480 times. In this instrument the optical flat is not placed directly above the surface to be tested but is replaced by a reference mirror situated to one side of the optical axis of the instrument, as in the well-known Michelson type of interferometer. The light source

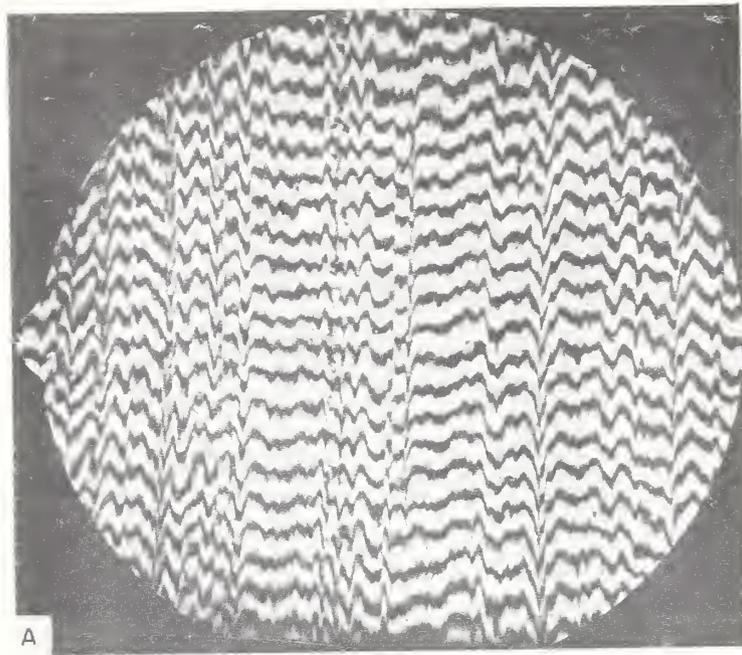


FIGURE 5. *Hilger and Watts microinterferometer for measuring surface finish.*

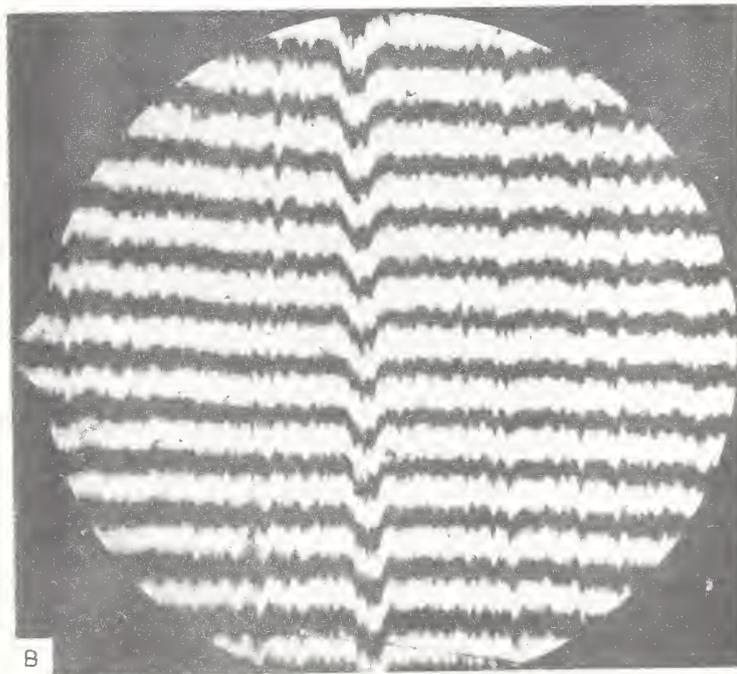
is a thallium lamp, from which the strong green radiations are practically monochromatic. Figure 5 shows a similar type of microinterferometer which has recently been produced by Messrs. Hilger & Watts Ltd. of London. A special feature of this instrument is that the reference mirror has 3 sections with reflecting powers of 4, 50, and 90 percent. Any one of these sections can be brought into play to match the particular reflectivity of the surface to be tested, and the most distinct fringes thereby obtained.

Small objects to be examined are placed on the table as shown. This is provided with micrometer and tilting arrangements in two directions at right angles. The whole of the upper part of the instrument can be turned through 180° so as to project over the back of the base for viewing large objects resting on any convenient rigid support.

Two examples of photographs taken with this particular make of instrument in mercury green light at a magnification of 360 times are shown in figure 6. The one at A is of a ground steel surface, the



A



B

FIGURE 6. *Microinterferograms of ground steel surface A and lapped steel surface B.*

roughness of which, judging from the irregularities of the fringes, amounts to 2 or 3 fringe spaces, that is 20 to 30 microns. The lapped steel surface at B is much smoother; the irregularities of its surface do not in general exceed a quarter of a fringe space, that is 2-3 microns. Towards the center, however, there is a vertical scratch about 5 microns deep.

In addition to this microinterferometer, Messrs. Hilger & Watts have constructed a simple attachment for fitting to the objective of their engineer's microscope. This instrument is shown in figure 7. The microscope works at a magnification of 120 times and a small camera is provided to enable the fringes to be photographed if desired.

I think it would be of interest to mention to you a comparison which was made at the N. P. L. a few years ago between "Talysurf" records of the surface of certain block gauges and photographs of fringes formed in a microinterferometer on the same gauges. To facilitate the comparison, the photographs were arranged to show the same horizontal and vertical magnifications as the Talysurf records. These two entirely different methods of delineating the irregularities over precisely the same areas of the surfaces examined

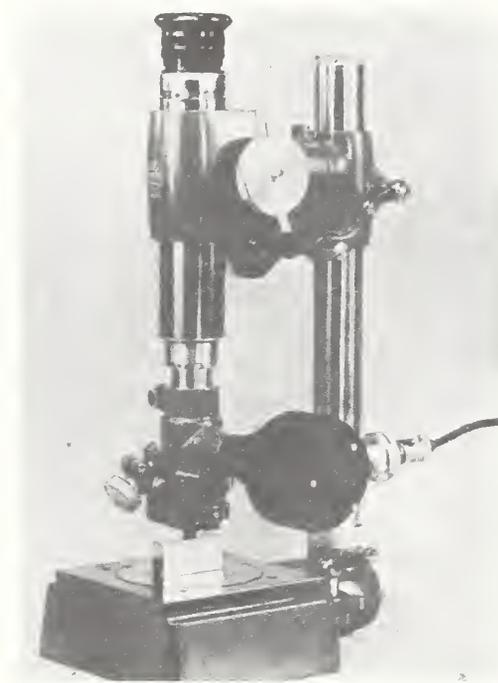


FIGURE 7. Hilger and Watts surface finish microscope set on gauge block.

showed truly remarkable agreement. The relative positions of the individual scratches were exactly the same and their depths agreed to appreciably less than a single micron.

Microinterferometers can also be used with advantage for examining the smoothness of the rounding off of the edges of the faces of block gauges. Unless proper care is used in the process of rounding off these edges the peripheries of the faces are likely to be left in a rather ragged condition. This in practice leads to the setting up of minute burrs and the detaching of tiny fragments of metal from the edges,

both of which can cause scratching of the faces when the gauges are wrung together.

Microinterferograms of the surfaces and edges of two block gauges taken in a Hilger microinterferometer are shown in figure 8. The one marked A shows the effect of carelessly chamfering the edge of the gauge in grinding. Photograph B in contrast is taken from a gauge, the edges of which have been carefully rounded off by fine stoning.

Measurement of Size of Block Gauges

I need hardly stress the importance of accuracy of size in block gauges. This applies particularly to reference sets of these gauges used in laboratories and standards rooms of factories as a basis for the calibration of other sets that are used for ordinary inspection and workshop use. Given good gauges, that is, gauges having faces that are accurately flat and parallel and of high quality surface finish, it is possible with modern interferometers to determine their size to an accuracy of one millionth of an inch, or even less in the case of the shorter gauges.

Block gauges are ordinarily made up to sizes of 4 in., or 100 mm, but interferometry can be applied to the measurement of much longer gauges, even up to $\frac{1}{2}$ or 1 m in length.

In using such interferometers particular care has to be taken with regard to temperature as this, of course, has an important influence on the length of the gauge. The density of the atmosphere also influences the lengths of monochromatic radiations, so that in all interferometry work of the highest order, observations have to be recorded of the pressure, temperature and humidity of the air and the necessary corrections made for these factors in order to determine the true value of the wavelengths at the time of measurement.

Quite early, work on the application of interferometry to the measurement of block gauges was carried out at the Bureau International, Paris, and the National Bureau of Standards, Washington. It was subsequently pursued by the N. P. L. and Messrs. Adam Hilger Ltd. in England, and by Dr. Kösters and Messrs. Carl Zeiss in Germany. As a result of all this work several types of gauge measuring interferometers have been designed and made available commercially. I now propose to refer to some of the more recent designs of these instruments.

N. P. L. Gauge Interferometer

Figure 9 is a diagram of the optical arrangement of the gauge interferometer which was set up at the N. P. L. some 25 years ago. Basically this optical system is very similar to that of the N. P. L. flatness interferometer. The most important difference is the introduction of the dispersion prism, CD, which enables the various monochromatic radiations emanating from the source, S1, to be directed on to the gauge, G, through the optical flat, F.

As in the case of the flatness testing instrument, the gauge to be measured is wrung on to the lapped surface of a baseplate, B, and it is interesting to point out that the measured length of a gauge under such conditions is equivalent to what is known as its "practical length", that is the material length of the gauge plus one wringing film.

Figure 10 shows a diagram of the interference fringe systems formed on the upper surface of the gauge and the surrounding surface of the baseplate. With a good gauge these two sets of fringes are both

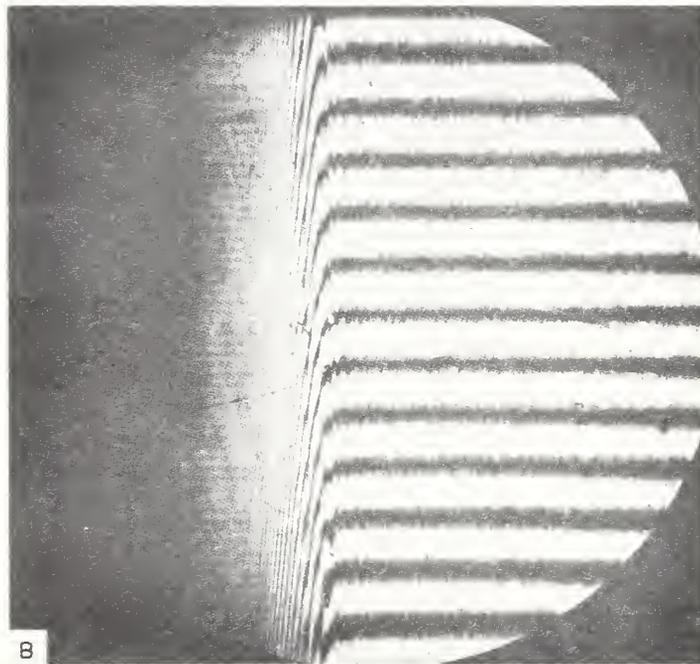
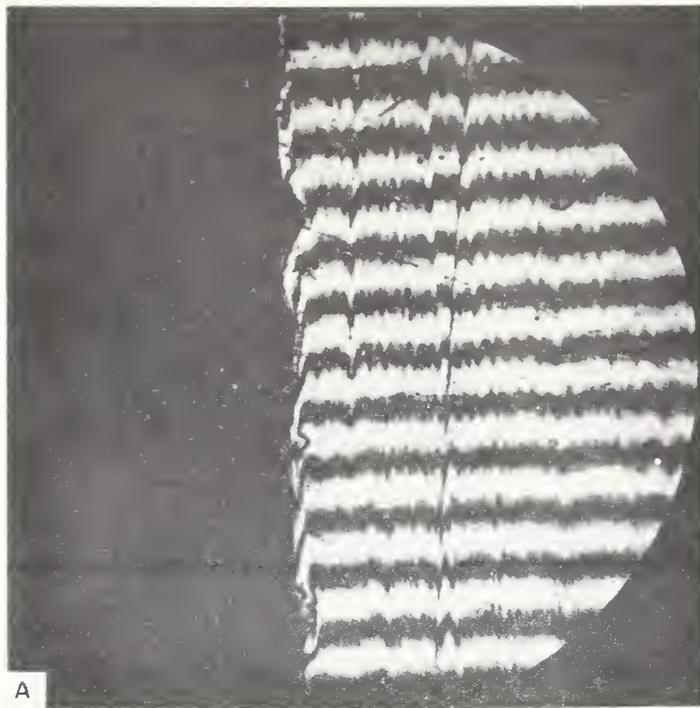


FIGURE 8. *Microinterferograms of surfaces and edges of gauge blocks.*

straight, equispaced and parallel to each other, but they are not necessarily in line with each other. In the diagram, the fringes on the gauge are offset with reference to those on the baseplate by a fraction a/b of a fringe space.

I do not think it is necessary for me to spend time describing to you all the details of the method of measurement, but essentially the measurement resolves itself into a measurement of the fractional

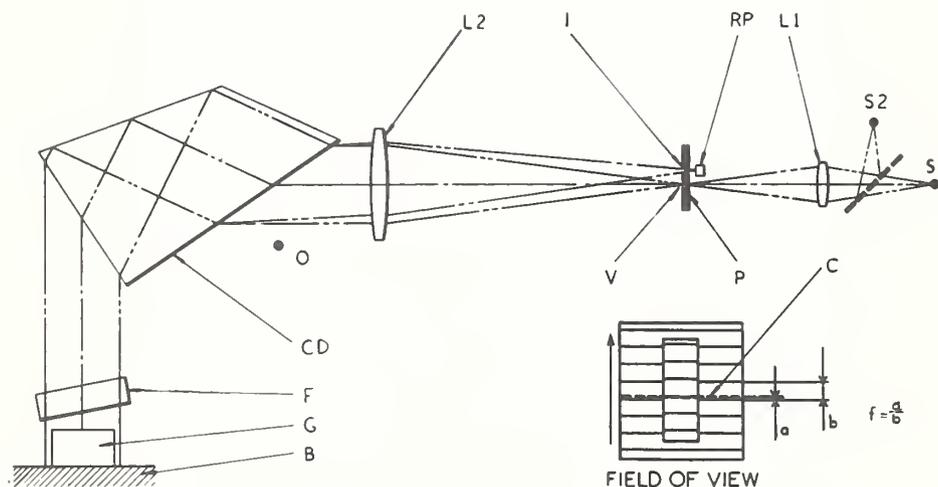


FIGURE 9. Optical system of N. P. L. gauge interferometer.

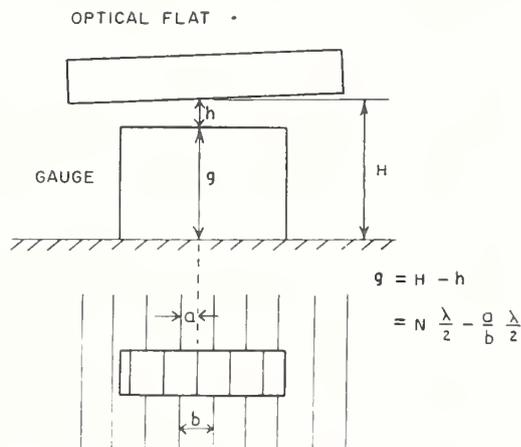


FIGURE 10. Diagram of system of interference fringes formed on surface of gauge block and surrounding baseplate.

displacement a/b for a series of monochromatic radiations on each particular gauge that has to be measured. From the measurement of these fractional displacements it is possible, by what is known as a method of coincidence, to determine the error of the gauge from its nominal length. Computed tables or a slide rule are commonly used to assist in this computation work.

The task of measuring a whole set of slip gauges by interferometry becomes very protracted if the gauges have to be dealt with one by one. It was for that reason that the N. P. L., when designing their gauge interferometer, arranged that the gauges could be wrung down in batches of 18 round the periphery of a rotatable baseplate.

This baseplate is so arranged that each of the gauges can be brought in turn under the optical flat and observations carried out upon it. A batch of 18 gauges can be dealt with and the results fully computed within about an hour.

The instrument is provided with a spare baseplate which can be loaded with a second batch of 18 gauges, and kept within the case of the instrument, so as to obtain thermal stability during the measurements of the first batch.

This N. P. L. type of gauge interferometer is now being made by Messrs. Hilger & Watts Ltd. of London, and a photograph of this commercially made instrument is shown in figure 11. Various constructional improvements have been embodied in this instrument which is fitted with two sources of monochromatic light, a cadmium and a mercury-198 discharge tube. This enables a choice to be made of the purest and the strongest radiations from these two sources.

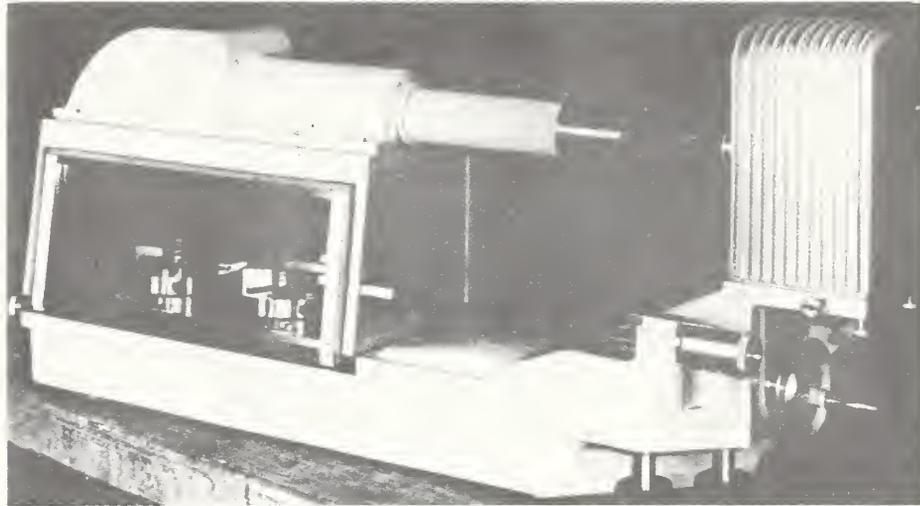


FIGURE 11. N. P. L. type gauge interferometer built by Hilger and Watts, Ltd.

Incidentally it may be mentioned that this interferometer can be used not only for measuring the lengths of gauges but also for viewing the flatness and parallelism of their faces. It should be noted that the instrument calls for no special supports for the gauges or for adjustments of their position for orientation. It can be used for measuring gauges of square or circular section, as well as those of the usual rectangular form.

Using the steel baseplates provided with the instrument, no difficulty need arise due to differences in phase change when measuring carbide or chromium-plated gauges. A short auxiliary gauge of steel or other material is first measured alone; this is followed by measurement of the gauge with the auxiliary gauge wrung on to its upper face, the differences between these two measurements giving the length of the gauge irrespective of the material of which it is made.

Hilger Double-Ended Gauge Interferometer

The optical principle of another type of Hilger interferometer, which was introduced about 1930, is shown diagrammatically in figure 12. It will be noted that in contrast with the N. P. L. instru-

ment, this interferometer is based on optical reflections made simultaneously at both ends of the gauges to be compared and not at one end only. The system of measurement is not so simple as in the N. P. L. instrument. It ordinarily involves the use of white light as well as monochromatic light. It can be used for comparing the

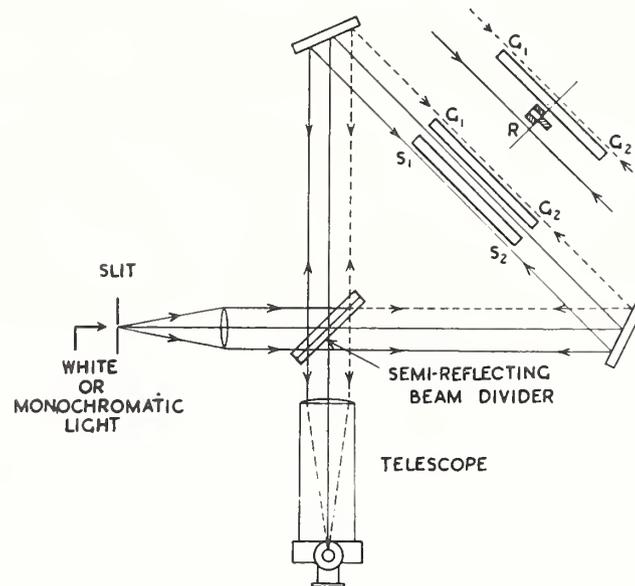


FIGURE 12. Diagram of optical arrangement of Hilger double-ended gauge interferometer.

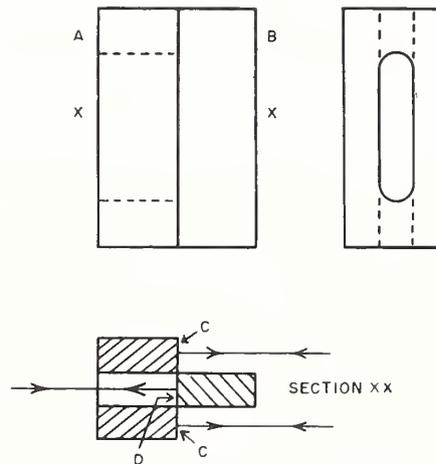


FIGURE 13. Reference gauge, of zero length, used in Hilger double-ended gauge interferometer.

lengths of two gauges S_1 , S_2 and G_1 , G_2 placed side by side between the two mirrors as shown. The same system is used when one wishes to determine the absolute length of a single gauge G_1 , G_2 . In that case one uses a reference gauge, R , as indicated in the figure, which in a sense has no material length. The construction of this reference gauge is shown diagrammatically in figure 13. It will be noted that the two reflecting surfaces of this gauge, C and D , face in opposite

directions as in an ordinary length gauge, but the distance between these two faces is only that of a wringing film.

Kösters' Interferometer

Let me now turn to some of the gauge interferometers which have been designed in Germany during the last 25 years or so.

The earlier types of these instruments were designed by Dr. Kösters and were constructed by Messrs. Carl Zeiss. The diagrammatic optical arrangement of Dr. Kösters' first gauge interferometer is shown in figure 14. As in the case of the N. P. L. instrument, the gauge to be measured is wrung vertically on to a steel or quartz baseplate. The optical system is based on that of the well-known Michelson interferometer. Interference takes place between the beams reflected from the reference mirror on the right and the surfaces of the gauge and of the baseplate, respectively. The instrument is so adjusted that the image of the reference mirror in the semitransparent, diagonally-placed beam divider is situated at about the midlength

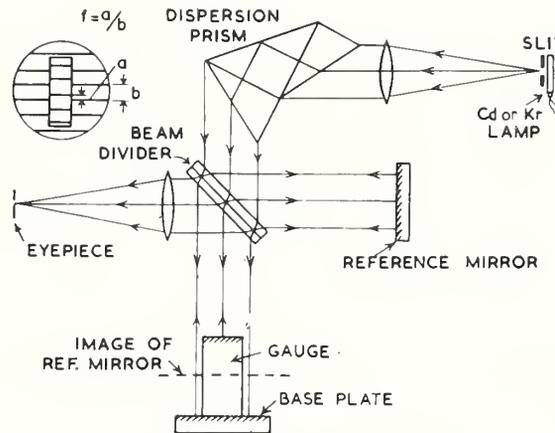


FIGURE 14. Diagram of optical arrangement of Zeiss-Kösters gauge interferometer, vertical type.

of the gauge. The path differences between the two interfering beams is thus approximately half the length of the gauge. With this instrument therefore it is possible to measure gauges of twice the length as compared with the N. P. L. type of gauge interferometer.

The basis of measurement in this Kösters' interferometer is somewhat similar to that used in the N. P. L. instrument, inasmuch as observations are made on the fractional displacement of the fringes formed on the upper surface of the gauge and on the baseplate, as shown in the inset diagram of figure 14.

The source of illumination in the Kösters' interferometer is either a cadmium or krypton lamp.

It may be interesting to mention that, by modifications to the Zeiss design of Kösters' interferometer, the N. P. L. has found it possible to measure gauges as long as 20 in. in this type of instrument. When dealing with such long gauges, however, it is preferable to arrange them horizontally rather than vertically. This was done in another design of interferometer by Dr. Kösters, with which he was able to measure gauges up to as much as 1 m in length.

It is interesting to note that Dr. Kösters used this horizontal type of interferometer in his determination of the relationship between wavelengths of light and the meter length. He first measured a gauge 500 mm long, and then the difference between this gauge and one having a length of 1 m. This provided him with sufficient information to determine the length of the meter gauge, and this in turn was compared with the length of a meter line standard, the value of which was known in terms of the prototype meter.

Shortly before the war, Dr. Kösters designed yet another type of gauge interferometer for measuring block gauges up to 200 mm in length. This interferometer was so arranged that it would accommodate six gauges at a time. Three of these instruments are said to have been constructed by Messrs. Carl Zeiss during the war, but

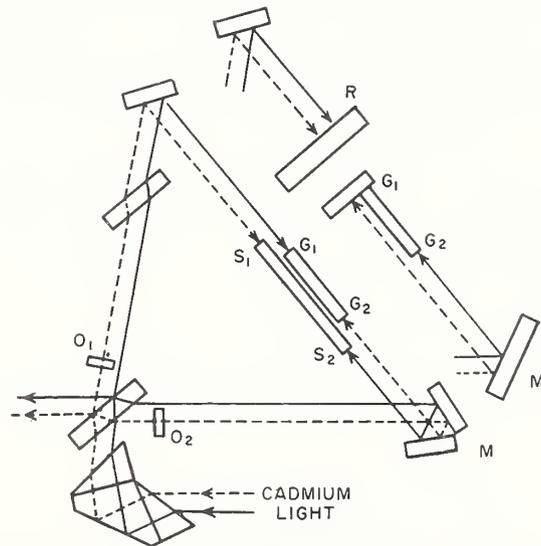


FIGURE 15. Diagram of optical arrangement of Zeiss gauge interferometer, horizontal type.

the only additional information that can be found about them is that they were dismantled in 1945.

Zeiss Gauge Interferometer

The latest form of block gauge interferometer made by Messrs. Carl Zeiss of Oberkochen in Western Germany was introduced about 3 years ago. Like the Hilger double-ended instrument it was designed for measuring block gauges by comparison one with another, or absolutely. From the diagrammatic optical arrangement of the Zeiss gauge interferometer shown in figure 15, it will be noted that the optical scheme of this instrument is rather similar to that of the Hilger double-ended interferometer. It possesses, however, one original feature in the form of inclinable optical plates O_1 and O_2 which are interposed in the paths of the interfering beams. By rotation of these plates it is possible to bring the two sets of interference fringes into exact coincidence and thus obtain a measure of their fractional displacement, instead of having to estimate it as in the case of the N. P. L. and Hilger instruments. In this Zeiss interferometer it is possible to measure a gauge by comparing its length with that of

another gauge of known length, or alternatively a gauge can be measured absolutely by wringing a small baseplate on to one of its ends, as shown towards the top right-hand corner of figure 15.

If three gauges of nominally the same length or of different lengths are available, it is possible to determine their individual absolute lengths by a scheme devised by Messrs. Zeiss. The differences between two of these gauges wrung together and the third gauge are measured in all possible arrangements, and from the three differences thus obtained it is a simple matter to determine the individual lengths of the gauges.

It was mentioned earlier that when carrying out any interferometric measurements of gauges it is necessary to take readings of the pressure, temperature, and humidity of the air during the measurements. Messrs. Zeiss have suggested an alternative scheme for taking account of the variation in the density of the air, by providing with their instrument two quartz end gauges, one 20 mm and the other 70 mm in length, the difference between these two gauges being known under standard conditions. By comparing this true difference with that measured during any set of measurements on any other gauge, it is possible to arrive directly at the correction that should be applied to the standard wavelengths to suit the particular set of measurements concerned.

Accuracy and Adaptability of Gauge Interferometers

I have already mentioned that with suitable care it is possible to use gauge interferometers for measuring lengths of gauges to an accuracy of the order of one millionth of an inch. When measuring to such an accuracy, variations in the type and quality of finish of the surfaces of gauges can produce corresponding variations in phase change in light reflected from their surfaces. I mentioned earlier how this variation in phase change can be avoided by the use of an auxiliary gauge.

As you know, in ordinary sets of block gauges quite a fair proportion of the gauges are relatively thin, and although the faces of these thin gauges may be parallel to each other, the gauges themselves in the free state are often bent to quite an appreciable amount. To measure such thin gauges by reflection from both their faces then becomes very difficult owing to the distortion of the interference fringes that they produce.

4. Achromatic Interferometer for Gage Block Comparison

By T. R. Young¹

The Optics and Metrology Division of the National Bureau of Standards has recently become concerned with the problem of extending the precision of measurement of length to 0.1 microinch per inch. The Engineering Metrology Section of the Division has been assigned the task of making measurements to this precision on end-blocks of the primary-echelon category. The factors that must be considered in an absolute measurement to such precision indicate that, at the present technological level, this measurement will be a laboratory endeavor requiring considerable time, control, and instrumentation. Fulfillment of these requirements may be justified in the measurement of primary standards; however, for the measurement of the much greater number of lower-echelon gage blocks existing in this industrial age, each gage block cannot be the subject of a laboratory investigation. Therefore, inherent to the problem of extending the precision of absolute measurement of gage-block standards is the problem of measuring lower-echelon gage blocks practically and with a precision making use of the new ultraprecision of the gage-block standard.

The precise measurement of lower-echelon gage blocks will undoubtedly be achieved by comparison with primary gage blocks. For this reason, a group at the Bureau consisting of Irvine C. Gardner, James B. Saunders, Edgar Robinson, and the author has, for the past year, been concerned with the design and development of an achromatic interference comparator. Although the principle of the achromatic compensator was originally conceived by Wilhelm Kösters and Paul Lampe² and later applied to a gage-block comparator, it is believed to be relatively unknown in this country and commercially unavailable. This paper will be concerned with the embodiment of the Kösters achromatic wedge system in a mechanical mounting temporary in nature but sufficiently precise to enable the possibilities of the wedge system to be thoroughly explored. A final form of the instrument remains to be designed, and it will be necessary on this instrument to incorporate adequate temperature control and to provide for the rapid inspection of gages. The specific prism system used in this instrument was designed by J. B. Saunders from glasses that he selected as specially suitable when combined to match the dispersion of air. E. L. Robinson made the optical components, which had to conform very precisely to the design if the system was to function in an adequate manner. Mr. Saunders devised special interferometric tests to control the optical components.

¹ National Bureau of Standards, Washington.

² W. Kösters and P. Lampe, German Patent No. 577377 (1929); *Werkstattstechnik u. Werksleiter* 23, 527 (1938).

Figure 1 shows the essential parts of the present instrument. Achromatic lens B (fig. 1) collimates white light, e. g., tungsten illumination, emitted from point source A. This light is transmitted by a double prism, C, in two parallel beams, as shown by the two typical rays of each collimated bundle. The double prism consists of two 30-, 60-, 90-degree prisms having rectangular faces. These are joined together as shown, using a very thin layer of oil or optical cement after one of the interfacing surfaces has been provided with an evaporated film of a density to provide equal intensities to the reflected and transmitted beams. The compensation necessary for white-light interference is achieved by making the prisms identical in size, using glass having the same index of refraction, taking care in

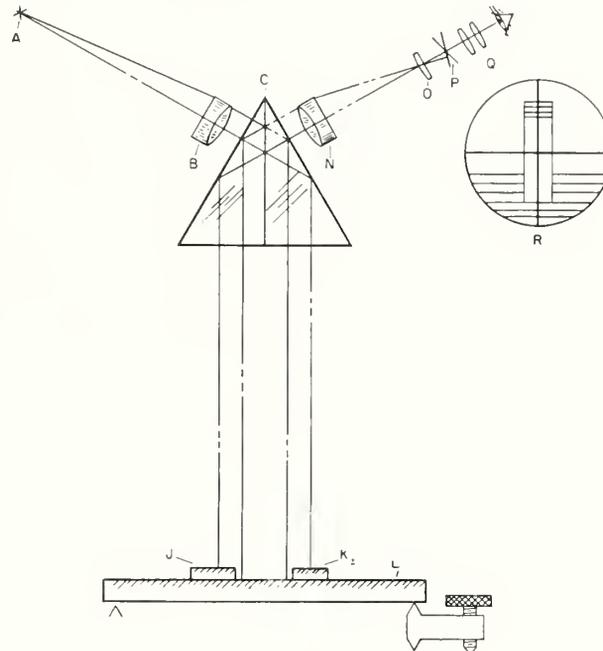


FIGURE 1.

the orientation of the prisms during the joining process and keeping the layer of cement or oil extremely thin.

After the two beams are transmitted by the double prism they are reflected normally from the measuring surfaces of the two gage blocks, J and K, and also from the surface of an auxiliary plate, L, to which the gage blocks are wrung. After reflection, the two beams return over their previous paths and are recombined at the dividing surface of the double prism, C. After recombination, part of each beam is transmitted through the collecting system of lenses N and O and a part is returned to source A. The collecting system of lenses images the surfaces of the gage blocks, as well as the surface of the auxiliary plate, L, in the plane of the cross hair, P. If the gage blocks are positioned properly in each beam, the images of the gage-block surfaces will be superimposed. A slight tilt of auxiliary plate L about an axis perpendicular to the plane of the drawing will cause two interference patterns to be formed, one between the images of auxiliary plate L as formed by the two beams, and the other between the two images of the gage-block surfaces. Viewed with an eyepiece, Q,

the field may appear as shown in R. Each of the two fringe patterns will be a typical white-light pattern consisting of a small number of multicolored fringes. If the gage blocks are equal in length, the interference pattern formed by the images of their measuring surfaces will be identical to the pattern formed by the images of the common surface, L, and the fringes appearing within the outline of the gage blocks will be aligned, color for color, with the fringes appearing from the auxiliary plate. If the two gage blocks are unequal in length, the two fringe patterns will be displaced from each other. If the inequality in length of the gage blocks were limited to such degree that both fringe patterns appeared simultaneously in the field, an observer still could not determine the difference in length directly from the

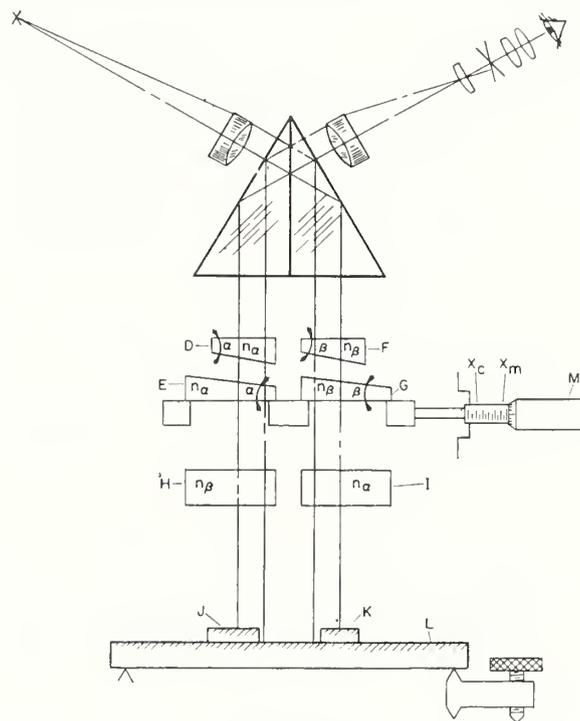


FIGURE 2.

displacement of the fringe patterns, even though he might recognize identical orders of interference in each pattern. Unlike the monochromatic fringe, no unit of length can be attached to an order of white-light interference, this being the resultant of interferences formed by a multitude of different wavelengths. Therefore, when using this interferometer, the observer does not follow the usual procedure of measuring the displacement of one fringe pattern with respect to the other. He measures instead, the movement required of an optical wedge system to compensate the optical-path difference arising when comparing gage blocks of unequal length. The form of this wedge system is shown in figure 2. For illustrative purposes, each wedge has been rotated 90 degrees to the right from its operating position. This also applies to the direction of thrust of the micrometer screw.

Wedges D and F, circular in form, match, respectively, rectangular wedges E and G in both wedge angle and index of refraction. When

properly alined, these wedges form effective plane parallel plates in each arm of the interferometer. Once alined, wedges D and F are fixed in position. Wedges E and G are positioned on a movable stage driven by the micrometer head, M. In this manner, a plane parallel plate of variable thickness is introduced in each beam. The tangents of the wedge angles determine the change of thickness per unit distance of advance of the micrometer screw. The wedge angle and the index of refraction, α and η_α , differ from β and η_β , so that a differential change in the optical paths of the two arms is initiated by advance of the micrometer screw. The wedge angles are chosen in the design to provide a convenient relationship between the differential change of optical path and the advance of the screw. The wedge angles of this interferometer were chosen to provide a differential change in optical path per 0.001-inch advance of the wedges equal to that which would occur if one of the gage blocks were changed in length by 0.2 microinch. Therefore, the calibration constant K for the screw was designed to be 2.000×10^{-4} ; i. e., a magnification of 5,000.

A differential wedge system with glasses in each arm differing in index of refraction is used because calibration constant K must be kept nearly invariant for the range of visible wavelengths emitted by the white-light source. In general, complete invariance cannot be attained in attempting to compensate a change of thickness of air by a change of thickness of glass. The use of a wedge system in only one arm of the interferometer would require for invariance a glass matching the dispersion of air. A glass even remotely approaching this dispersion does not exist, so an attempt is made to obtain approximate invariance by using two different glasses in a differential wedge system. Using this approach to the problem, the degree to which the designer can approach invariance depends upon his selection of the glass combination. When calibration constant K varies with wavelength a progressive deterioration of the fringe occurs as one attempts to compensate for increasing differences in air path; the closer the calibration constant is held to invariance, the lower is the rate of deterioration of the fringes. The effect of this deterioration can be observed as a shift of the black, zero-order fringe. This shift must be restricted to an undetectable amount because the black fringe is alined with the cross hair in the measurement procedure. Thus, a small rate of deterioration permits compensation for a greater range of air path difference and so permits comparison of gage blocks having greater differences in length. For this reason the range of the comparator depends to a large extent on the designer's choice of glasses. He has at his disposal a large number of possible glass combinations. If the design is to be obtained with a minimum of time and labor, he must, early in the design procedure, select from this large number a combination offering a good range. A method of easily determining a good combination of glasses has been devised, but as it involves a rather detailed description, it is not given in this paper.

To complete the requirements of this interferometer, plane parallel plates, H and I (fig. 2), are used. Each has an index of refraction identical to that of the wedges in the opposing beams. Each has a thickness equal to that of the wedge combination in the opposing beam when that wedge combination is of such thickness as to compensate for a zero path difference in air. Their purpose is to compensate for the glass of the wedges in the opposing beam. This compensation is necessary to retain the white light fringe condition.

A description of the alinement of the optical elements follows: The alinement of this instrument is particularly important in that the balance of the wedge system specified by the design must be achieved. Furthermore, the mechanical movements necessary for the various optical elements are implied in this description.

1. Starting with the instrument as shown in figure 1, the observer replaces the white light source with a monochromatic source so that aperture A emits light of wavelength λ . He uses for J and K two gage blocks differing in parallelism by several fringes as measured along the surfaces of the gage blocks. These gage blocks need not have the same nominal length. One tilts auxiliary plate L about an axis perpendicular to the plane of the drawing until a single broad fringe forms the auxiliary plate pattern. Several fringes will appear in the gage-block pattern.

2. One places wedges D and E in position, as shown in figure 2, and rotates wedge D about its optical axis until a single broad fringe again appears in the auxiliary plate pattern. This insures that wedges D and E function as a parallel plate.

3. One determines the effective angle of wedge E in relation to the direction of thrust of the micrometer screw by counting the number of gage-block fringes passing the cross hair for a given advance of the micrometer screw. The effective angle of wedge E is given by

$$\tan \alpha = \frac{\lambda}{2(n-1)} \frac{\Delta N}{\Delta x},$$

where λ is the wavelength of monochromatic light in standard air, n is the relative index of refraction of the wedge for wavelength λ , and ΔN is the number of fringes passing the cross hair for an advance Δx of the micrometer screw. If the effective wedge is not that specified by the design, wedge E is rotated a small amount and procedures 2 and 3 are repeated.

4. Compensating plate I is placed in position. Monochromatic light is replaced with white light. The auxiliary-plate pattern should still consist of a single broad fringe. If a colored interference order is observed, or if no interference order is observed, the micrometer screw is advanced until the black interference order forms the auxiliary plate pattern. The reading on the micrometer scale, x_c , is the compensated position for zero air path difference. It is convenient here to have a micrometer scale adjustment so that the reading x_c can be positioned at a convenient place on the scale.

5. Wedges F and G and compensating plate H are placed in position. With the micrometer set at x_c , G is translated by means independent of the micrometer screw until fringes in the auxiliary-plate pattern are observed. Wedge F is rotated until a single fringe forms the wringing-plate pattern. Then the translation of wedge G is continued until a black fringe is observed. This procedure insures that wedges F and G function as a parallel plate and are also compensated at the position x_c of the micrometer scale. Notice that in procedures 1 through 5 no change in the tilt of auxiliary plate L is to be allowed. If a drift does occur, it can be corrected at appropriate times in the procedure.

6. The micrometer screw is advanced to the midpoint of its scale, x_m . If x_m differs from x_c , the wedge system is then in a position to compensate for a difference in air path, the amount depending upon

the calibration constant of the screw. This air-path difference is accomplished by tilting auxiliary plate L about an axis perpendicular to the plane of the drawing in figure 2. The plate is tilted until the black fringe is alined with the cross hair. A number of fringes are now observed in the auxiliary-plate pattern. The fringe width will depend upon the magnitude of $x_m - x_c$ and the calibration constant of the screw. This can be explained by noting that when the auxiliary plate is tilted the position of zero air-path difference immediately moves to the intersection of the plane of symmetry of the prism and the plane of the auxiliary plate. When the micrometer is advanced from x_c to x_m , plate L must be tilted so that the difference in height between positions conjugate to the cross hair must be equal to $(x_m - x_c)K$. Then the black fringe will be alined with the cross hair. Thus, the angle of tilt is determined and the magnitude of this tilt determines the fringe width. If it is found that the fringe width is inconvenient for use, the position of x_c on the scale should be changed. This may be accomplished by the use of the micrometer-scale adjustment mentioned in procedure 4.

7. The calibration constant, K , of the micrometer screw must still be determined. White light is replaced with monochromatic light of wavelength λ . Auxiliary plate L is tilted so that one broad fringe forms the auxiliary-plate pattern. One can then determine the calibration constant of the screw by counting the number of fringes in the gage-block pattern that pass the cross hair for a given advance of the micrometer screw. One can determine the calibration constant by the equation

$$K = \frac{\lambda}{2} \frac{\Delta N}{\Delta x},$$

where λ is the wavelength of the monochromatic source for standard air conditions, and ΔN is the number of gage-block fringes passing the cross hair for an advance Δx of the micrometer screw. If it is found that K is different than that specified by the design, the calibration constant may be changed by rotation of wedge G. If this is done, wedge F is rotated until a single broad fringe again forms the wringing-plate pattern.

8. If desired, one can check the lack of invariance of K with wavelength by determining K for various wavelengths. The alinement is then completed by replacing the monochromatic source with a white-light source.

An alinement procedure, such as that mentioned above, is only necessary when the instrument is first assembled or when a reassembly is required. It is to be stressed that it is not a part of the measurement procedure.

The instrument on display at this symposium has a micrometer head with a range of 1 inch. Scale divisions can be read directly to 0.001 inch. The calibration constant varies with wavelength as follows:

$$\begin{aligned} K_{656.3} &= 0.0001998 \\ K_{589.2} &= 0.0002000 \\ K_{486.1} &= 0.0001999 \\ K_{434.0} &= 0.0001998. \end{aligned}$$

The subscripts refer to the wavelength in millimicrons. The calibration constant assumed for the instrument is 2.000×10^{-4} as the maximum sensitivity of the eye is at $555 \text{ m}\mu$. Using the full range of the instrument (1 in.), a maximum systematic error of 0.2 microinch is involved. As the effective calibration constant is undoubtedly between $K_{486.1}$ and $K_{589.2}$, gage blocks differing in length by as much as 0.0002 inch can be compared with error less than 0.2 microinch. To match this potential precision the micrometer screw must measure the advance of the wedge system more accurately than 0.001 in. per inch. In addition, if a precision exceeding 0.2 microinch is to be obtained in comparing steel gage blocks, the temperature of the gage blocks should be held equal to within 0.01 deg C. per inch of gage-block length. Furthermore, if the measuring surfaces of the gage blocks differ in surface finish a correction for phase change must be applied to achieve this precision. In addition to providing a simplified measuring technique and a large range, this achromatic compensating system provides a precision suitable for exacting laboratory measurements.

The convenient place on the scale for the reading x_c for this instrument was found to be 0.2500 inch. Placing x_m at 0.5000 or 0.0000 on the scale the wedge system is at a position to compensate a difference in height of 50 microinches. Tilting the auxiliary plate an amount to align the black, zero-order fringe with the cross hair results in approximately six fringes in the field. This provides a fringe width convenient for alinement. With x_m at 0.0000 the whole range of the instrument (0.0002 in.) can be used. Placing x_m at 0.5000 on the scale, the instrument has a range of ± 0.0001 inch.

To show the simplicity of the measuring technique when using this instrument, the measuring procedure is given. Assume that the lengths of two gage blocks with equal surface finishes are L_1 and L_2 and that unknown to the operator $L_1 = L_2 + 95.4$ microinches.

1. The gage blocks are wrung to the auxiliary plate with the spacing between them such as to cause their images to superimpose in the center of the field of the instrument, as shown in figure 1.

2. After a time interval for temperature equilibrium, the micrometer screw is advanced to a reading of 0.5000.

3. The auxiliary plate is tilted about an axis perpendicular to the drawing in figure 2 until the black fringe of the auxiliary plate pattern is alined with the cross hair. As these fringes will always be parallel with the cross hair and perpendicular to the long dimension of the gage-block image, no other tilting adjustment is required.

4. The micrometer screw is advanced until the black fringe of the gage-block pattern is alined with the cross hair. A reading is taken of the scale. This may be either 0.9770 or 0.0230, depending upon which arm contains the gage block of length L_1 . In this interferometer it is known from the design that movement of the wedge platform to the right (fig. 2) increases the optical path, and that the left arm is increased at a greater rate than the right arm. Therefore, if the reading is 0.9770, the longer gage block, L_1 , is in the left arm and

$$L_1 = L_2 + 0.0002000(0.9770 - 0.5000)$$

$$L_1 = L_2 + 95.4 \text{ microinches.}$$

To conclude the description of the component parts and the operational procedure, it may be well to consider some of the advantages

possessed by this instrument. While some of those listed below are common to other methods of interference comparison, it has other advantages that make it unique as an interference comparator. It is believed that these advantages make it necessary seriously to consider this interferometer as a practical comparator:

1. No measuring pressure is exerted on the gage-block surfaces.
2. It has good precision combined with an adequate range.
3. No prior knowledge of the difference in length of the gage blocks is required to avoid misinterpretation of the interference order.
4. The measuring procedure is relatively simple, and the need for training of the operator is kept to a minimum. No estimation of fringe fractions is involved.
5. The air paths and gage blocks are in close proximity at the time of measurement.
6. The parts are stable and, with the exception of the light source and the auxiliary plate, should last for years without replacement or maintenance.
7. Once assembled, the instrument does not require special monochromatic light sources.
8. Effects of vibration are reduced as the interferometer is sensitive only to vibrations tending to tilt the auxiliary plate with respect to the prism about an axis perpendicular to the plane of the drawing in figure 2.
9. The interferometer can accommodate a turret-type auxiliary plate for convenient comparison of more than one pair of gage blocks.
10. Although time does not permit discussion here, it is possible to measure absolute lengths with this interferometer by replacing the white-light source with a monochromatic source.

5. A High-Sensitivity Interferometer for Measurement of Phase Shift and Other Applications

By J. B. Saunders¹

An interferometer arrangement is described that permits greater sensitivity than has previously been available. Its principal application is the measurement of thin films and also absolute phase shifts at reflection. With highly reflective surfaces, such as fresh silver, as many as fifty reflections may be used and, in such case, one order of interference will correspond to approximately one hundredth part of a wavelength of the light used. A method for applying this interferometer to the measurement of phase shift of light from metallic surfaces and for the comparison of gage blocks is described.

1. Introduction

The accuracy of measurement attainable in interferometry is limited by the inability of the operator to duplicate settings on the centers of the interference fringes. The principles of multiple reflections introduced by Fabry and Perot,² and further developed by Tolansky,³ reduce the error of fringe readings by reducing the width of the fringes relative to their separations. However, asymmetry of the curve for light distribution about a minimum (center of narrow dark fringes) increases the difficulty of locating the minimum or darkest point.

This paper describes another method of multiple reflections that reduces the error in results due to error of reading the fringe fractions. Its applications are limited to small order differences and to small changes in order. Some of these applications are as follows: (1) Comparison of gage blocks, (2) variations of index of refraction in transparent fluids and solids, (3) thermal expansion, (4) thicknesses of thin films, and (5) the electromagnetic phase shift of light when reflected at a boundary. Numbers one and five of the above mentioned applications are of vital importance in the measurement of gage blocks. The phase shift (5) varies with wave length, with roughness or finish of surface, and from metal to metal.

The discussion of this paper is primarily directed to the measurement of phase shifts of light vectors at reflection. This involves measurements of wringing films (separation of gage block and the base plate to which it is wrung), surface-finish effects, and measurement of small differences between gage blocks. Because these quantities are often of the same order of magnitude as the errors in determination of fringe fractions, errors in the reading of fringes are very important.

2. Phase Shift of Light at Reflection

When light is propagated by transmission through any medium the phase change of the electric vector is uniform, continuous, and con-

¹ National Bureau of Standards, Washington.

² Ch. Fabry and A. Perot, *Ann chim. phys.* **22**, 564 (1901).

³ S. Tolansky, *Multiple-beam interferometry of surface films* (Oxford, Clarendon Press, 1948).

stant with time. If we represent time, measured from the time of emission, by T (fig. 1, A) the phase, β , of the vibrating light vector is proportional to T . That is, $\beta = kT$, where k is a constant of proportionality. If at some later time, T_1 , (fig. 1, B) the light is incident upon the polished surface of a dielectric, the light beam is divided

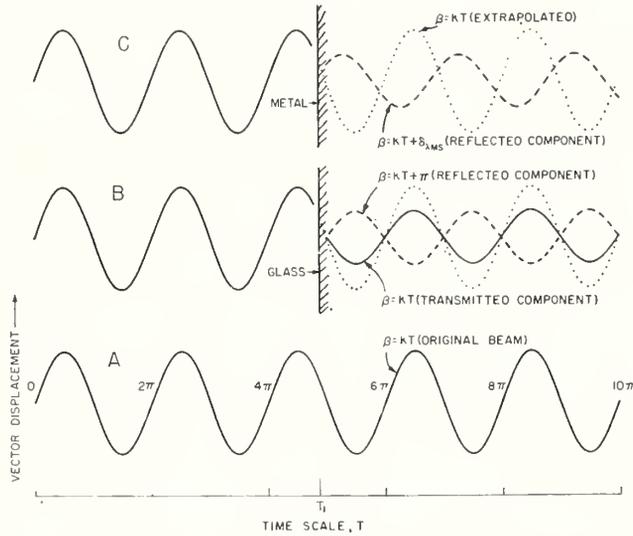


FIGURE 1. Change in phase of light vectors with time, and at boundaries.

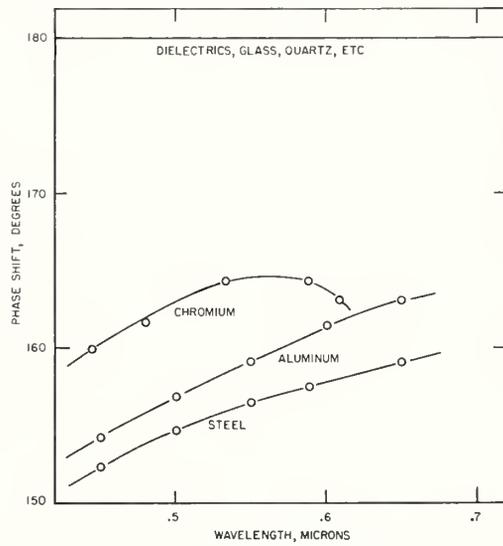


FIGURE 2. Change in phase of light vectors at reflection from metals and dielectrics.

into a reflected and a transmitted component. The phase of the transmitted component remains proportional to time T . The reflected component suffers a large increase in rate of phase change that lasts for a very short time during the reflection, in the neighborhood of T_1 . The directions of the component beams are not shown in figure 1. No geometrical directions are indicated. The abscissas represent time, and the ordinates represent amplitudes of the vector displacements.

ments. The differential phase change between the transmitted and reflected components, for dielectrics, is precisely π radians for all wavelengths of light. The phase shift for metals ($\delta_{\lambda ms}$ in fig. 1, C) is always less than π radians.

If the face of the reflecting surface has scratches, sleek marks, pits, etc., the quantity T_1 differs for different parts of a wave front, assumed parallel to the surface. This introduces a spread in values of the phase, β , for the reflected component beam. The observed phase will be some form of an average. However, as scratches, sleek marks, and pits do not affect the position of the measured geometrical surface, the phase change resulting from them is properly considered as due to the finish of the surface.

A graph, showing variations in phase shift, δ , at reflection with wavelengths, λ , for several materials, is given in figure 2. The change in β for steel between wavelengths $\lambda=0.4471 \mu$ (helium violet) and $\lambda=0.7065 \mu$ (helium red) is approximately 7.9° or more than one-fiftieth (0.022) of an order of interference. When measuring a 1-mm gage block, this change in order equals that caused by a change in atmospheric pressure of 15 mm of mercury, or to that caused by a change of 0.4°C in the temperature of the gage block.

3. Methods of Measurements

Several interference methods of measuring the phase of light at reflection have been used. In making such measurements with most methods the thickness of the "wringing film", the effects of surface roughness, and small differences in gage blocks require consideration. The quantities being measured in all of these methods are so small that other errors of measurement prevent the attainment of high accuracy in the results. The arrangement described herein increases the magnitudes of the fringe shift and related effects, relative to other errors, by using multiple reflections from the samples.

Consider two plane reflecting surfaces, M_1 and M_2 (fig. 3), that are adjusted to form a small angle α between them. If mirror M_1 receives a collimated beam of light, incident at an angle φ that is greater than α , the beam will be reflected back and forth between the mirrors. The angle of incidence decreases by an amount α at each successive reflection. At the N th reflection it is $[\varphi - (N-1)\alpha]$. If φ is adjusted to equal an integral multiple of α , the beam eventually becomes normal to one surface; after which the angle becomes negative, increasing in the negative direction until it becomes $-\varphi$; after which the beam returns along the incident path.

If N equals the total number of reflections and the angle of last incidence is equal to the negative of the 1st incidence, then $\varphi - (N-1)\alpha = -\varphi$, or

$$2\varphi = (N-1)\alpha. \quad (1)$$

The light appears to be reflected from M_3 , which is an image of M_1 , if $(N-1)/2$ is even, and is an image of M_2 if $(N-1)/2$ is odd. In the interferometer arrangement, to be described here, surface M_3 represents an end mirror and receives, normally, one of the component beams that produce interference.

In order to arrange the elements properly, it is important to know the magnitudes of the following defined quantities, shown in figure 3:

dicular to the plane of incidence. These values were computed from Minor's⁴ determination of optical constants and electromagnetic theory. It is seen that δ_{S20} (the value of δ_S at 20° incidence) differs from δ_{S0} by 1.3° for light polarized perpendicular to the plane of incidence. Similarly, $\delta_{P0} - \delta_{P20} = 1.4^\circ$ and $\delta_{S20} - \delta_{P20} = 2.7^\circ$, which corresponds to only 0.0075 fringe. The quantity $(\delta_S - \delta_P)$ represents a spread in phase shift caused by the coexistence of two superimposed sets of fringes that are not quite in phase with each other. The total spread is the summation, $\Sigma(\delta_S - \delta_P)$, for all reflections from the samples. If 7 reflections are used, there will be 2 reflections at 20° incidence, 2 reflections at $13\frac{1}{2}^\circ$ incidence, 2 at $6\frac{2}{3}^\circ$, and 1 at 0° or normal. The

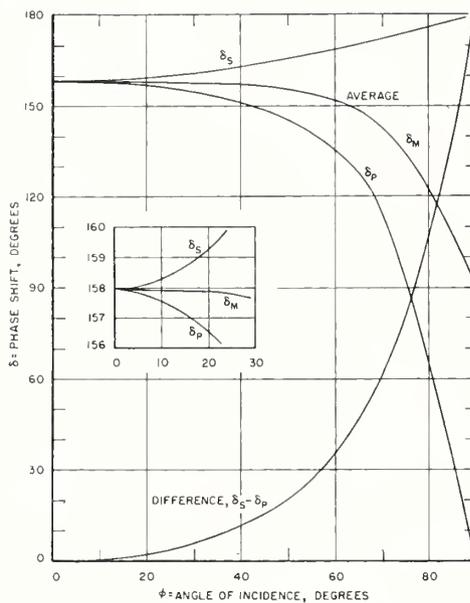


FIGURE 4. Variation of phase shift at reflection with angle of incidence.

The subscripts S, P, and M refer, respectively, to light polarized normal to the plane of incidence, parallel to the plane of incidence, and to the average or unpolarized light.

total spread is found to be 3.76° ; or the change in the order of interference at any point due to changing from light that is polarized in the plane of incidence to light polarized normal to it is approximately 0.01 of a fringe. The observed fringe position, with unpolarized light, is the average. Figure 4 shows that δ_M remains very nearly constant for values of ϕ less than 20° . Consequently, no appreciable error is introduced by the change of phase with angle of incidence, provided ϕ does not exceed 20° .

The number of reflections, N , is obtained by direct observation of images of a small light source. Figure 5 shows the array of images of a light at L , seen by looking into mirror M_1 . The subscript, v , of any image I_v represents the number of reflections from the two mirrors.

4. Measurement of Phase Shift at Reflection

One method of measuring phase shifts at reflection from metal gage blocks will be described. A wedge of small angle, α , and appropriate thickness is made with high-quality surfaces (see fig. 6, A).

⁴ R. S. Minor, Ann. phys. **10**, 581 (1903).

Three holes are cut through the wedge, as shown in figure 6, B. A high-reflectance plane mirror, M_2 , is contacted to the upper surface of the wedge, and three gage blocks are contacted to the lower surface of the wedge; each block covering one of the three holes in the wedge. This places the three upper surfaces of the three gage blocks coplanar, if the wringing films are equal. We shall assume equality of the wringing films. This condition is practically attained if the blocks are "optically contacted" to the wedge.

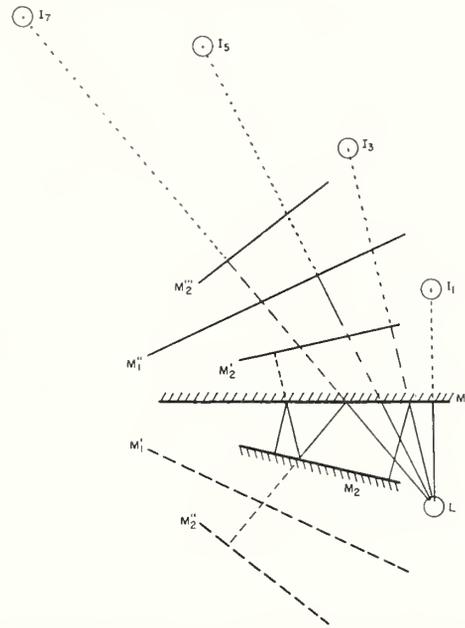


FIGURE 5. *Multiple imagery from multiple reflection of light between two mirrors.*

The subscripts of I represent the number of reflections suffered by light that produces the corresponding images.

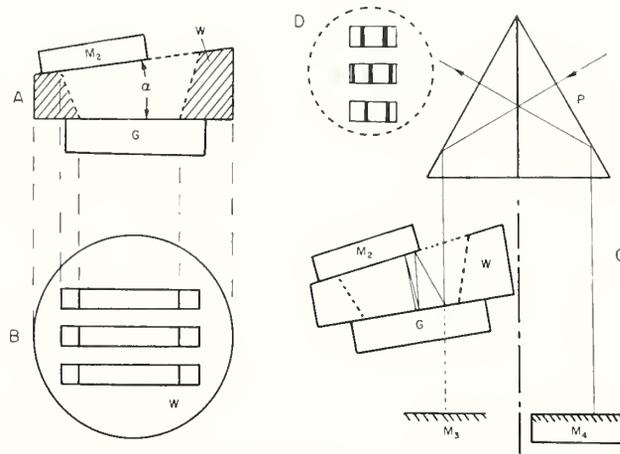


FIGURE 6. *Arrangement of an interferometer for measurement of phase shift of light at reflection.*

W is a wedge, M_2 a plane, high reflectance mirror, M_3 an image of M_2 , M_4 an end mirror, G a gage block, α the angle between gage blocks and mirror M_2 . B is a top view of W. C is a side view of a Kösters double prism interferometer. D shows the relative shifts of fringes that arise from differences in phase shift at reflection.

If this combination is introduced into one arm of an interferometer, as is shown in figure 6, C, interference may be obtained. The fringes (fig. 6, D) produced by interference of the two component beams, over the area of the unobstructed part of the beam that is reflected from the gage blocks and mirror M_2 combination, will form a single set of straight fringes if the phase shifts at reflection from the three gage blocks are equal. However, if one of the gage blocks is made of quartz (or glass) and is uncoated, its phase shift will differ from that of the metal gage blocks. There will be a shift in the fringe pattern, as is indicated in figure 6, D, which is a measure of the difference in phase shift at reflection between a dielectric and the metal surface of the gage block. If the light has suffered N reflections from the gage blocks, the magnitude of the fringe shift will be N times that for one reflection. If the metal blocks are of steel and the surface of one of them is a high quality optical surface, free from finish marks, (hereafter designated "the standard") the phase difference between it and the quartz will be approximately 22 degrees. The relative displacement of the observed fringe pattern will, accordingly, be $22 N$ degrees. If it is assumed that $N=10$; $22 N$ will equal 220 degrees, or 0.6 fringe—a quantity that may be measured 10 times as accurately as other methods of measuring phase shifts permit.

After having measured the phase shift of the standard metal surface, the phase-shift difference between it and a sample gage block (indicated here by the second metal gage block) is similarly measured. The purpose of using a standard is to obtain a higher reflectance and better observation conditions than are obtainable with the relatively low reflectances possible with uncoated quartz or glass. After having calibrated the standard surface, the quartz block may be replaced with sample surfaces to be measured. The particular arrangement shown in figure 6 permits two specimens to be tested simultaneously. Obviously the method permits an increase in the number that can be mounted together for simultaneous tests.

A more elegant arrangement is obtained by rotating the wedge of figure 6, C, 90 degrees from the position shown and adjusting the lateral position until the images of the specimen and standard coincide—eliminating mirror M_4 completely. The absolute order of interference, determined with polychromatic (white) light, is then a measure of the difference in phase.

5. Comparison of Gage Blocks

Another application of the above principle of multiple reflections is that of comparing gage blocks with standards. For this interferometer, the two gage blocks (a standard and the unknown to be tested) are wrung onto a baseplate in the conventional manner (see fig. 7). Mirror M_2 is adjusted to form a small wedge between it and the tops of the two gage blocks. If the top surfaces of the gage blocks are similar and the wringing films assumed equal, the observed relative shift in the fringe pattern is a measure of the difference in thickness of the two gage blocks.

In order to correlate observed orders of interference with differences in thickness of gage blocks, the top surfaces of the two blocks and that of mirror M_2 are projected to the vertices of the wedges between them (see fig. 8). Since, in general the gage blocks are not perfect parallels, their upper surfaces will usually produce different angles

with the surface of mirror M_2 , in figure 7. Also, these angles usually differ from the angles between the baseplate and mirror M_2 . The details of the mathematics are too extensive to be included here. However, if we define d as the difference in thickness of the two blocks at the point of first incidence, ϵ the angle between their faces (difference in wedge angles), γ the observed angle between the two interfering beams, F the observed order of interference, h the maximum separation between mirror M_2 and the gage blocks, measured

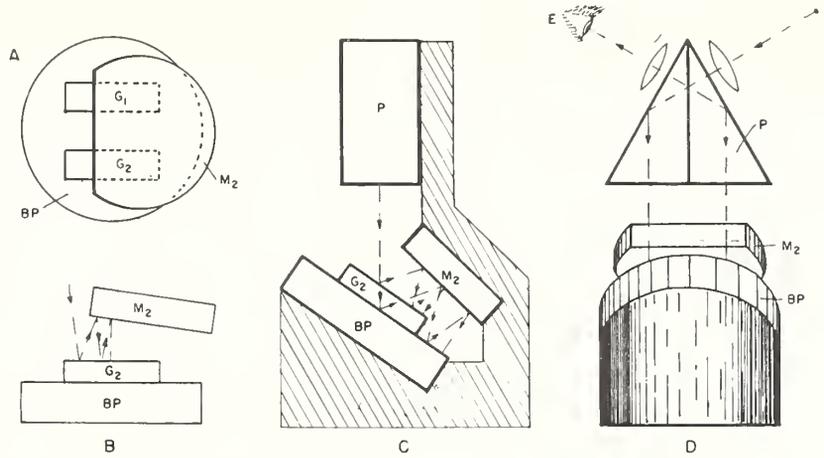


FIGURE 7. Plan of interferometer for comparison of gage blocks.

G_1 , G_2 are gage blocks (standard and sample); BP is the baseplate to which the blocks are wrung; M_2 , a plane high reflectance mirror; P a Kösters double prism beam divider. A and B are, respectively, a top and a side view of the combination of the mirror, baseplate and gage block. C and D are two horizontal side views of the entire interferometer, at right angles to each other.

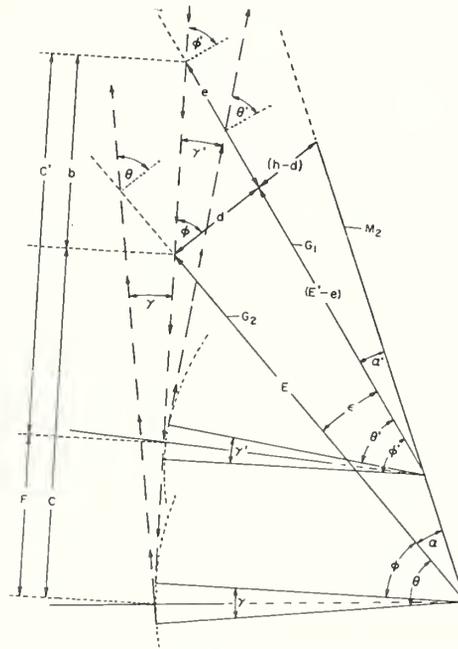


FIGURE 8. Geometrical diagram of optics of "increased-sensitivity interferometer."

The working equations for the interferometer are derived from this figure. Distances and angles are indicated. The lines marked M_2 , G_1 , and G_2 correspond to mirror M_2 and the gage blocks indicated in figure 7.

from the edge of M_2 adjacent to the aperture, ϕ the angle of first incidence of the beam on the gage blocks, and α the angle between the gage blocks and mirror M_2 ; then

$$d = \frac{F \sin \alpha}{\sin(\phi + \alpha)} + \frac{h\gamma \cos \alpha}{(\phi + \alpha) \sin(\phi + \alpha)} (\alpha \cot \alpha \sin \phi - \phi \cos \phi) \quad (3)$$

and

$$\epsilon = \frac{\alpha\gamma}{\phi + \alpha} \quad (4)$$

The second term in eq (3) vanishes when γ becomes zero. This condition is satisfied when measuring thicknesses of evaporated films that are uniform in thickness, and when the two gage blocks have equal wedges that are properly oriented with respect to M_2 . Because α is usually small, the coefficient of γ in eq (3) will always be comparable in value to that of the coefficient of F . If one-tenth of a fringe error in F is considered insignificant and γ is of this order of magnitude, then the second term of eq (3) becomes negligible. In any case, γ may be measured as precisely as F . Consequently, the principles of this instrument also afford a precision method for testing and measuring the parallelism of surfaces.

An interferometer of the above description is under construction. Measurement of phase shifts at reflection is contemplated for a first application.

6. An Improved Angle Interferometer

R. E. Sugg¹

1. Introduction

The relation that length gages bear to the precise measurement of angles is apparent when the use of gage blocks with sine bars is considered. All the opportunities for error inherent in stacked blocks are present in this method of angle measurement and also in the use of angle blocks. In addition, observational errors may be greater than incurred in length measurement.

Our experience, substantiated by others² has been that claims made for the accuracy of angle blocks to ± 1 sec of arc and for dividing heads and rotary tables to ± 2 to 5 sec are often excessive. In order to use these media for measurement or positioning in the tolerance range of 1 to 5 sec, a means of calibration is required with an inherent accuracy to an order of magnitude greater (i. e., ± 0.1 sec). This is an exceedingly small quantity, being approximately 1/13,000,000 part of a circle, and may be visualized as the angle subtended by the edge of a sheet of writing paper at 500 ft. At the edge of a 4-in.-diameter circle this amounts to less than a millionth of an inch, and, as usual when dealing in microinches, the use of an interferometer is indicated.

An interferometer for angle measurement was developed by T. J. O'Donnell, several years ago.³ A similar instrument was constructed by Moore Special Tool Company for use in calibrating the precision rotary tables they manufacture. The instrument to be described is a modification of these designs to achieve freedom from error due to variations in ambient conditions, to assure ease of use, and to provide simplification in manufacture.

2. Principle of the Angle Interferometer

So far as we know, the use of interferometers for the direct measurement of angles has not been published, and a description is in order. It may be seen from figure 1 that the instrument is basically a Michelson interferometer in which the reflecting mirror in one of the light paths is replaced with two mirrors, one above the other on a rotary table, and inclined to each other at the angle desired for measuring or indexing. In order to set this angle to the required accuracy, the following steps are to be followed:

1 With the table set on zero and the mirrors set to the approximate angle, the mirrors are rotated until the required fringe pattern appears from either the upper or lower mirror.

¹ E. I. du Pont de Nemours & Co., Wilmington, Del.

² C. F. Bruce and W. A. F. Cuninghame, Measurement of angle by interferometry, Australian J. Appl. Sci. **1**, 243 (1950).

³ T. J. O'Donnell, The angle step plate interferometer (Physical Sciences Development Shops, Univ. Chicago, Oct. 27 and 28, 1952) (unpublished).

2. The table is then rotated the exact angle of the mirrors until the fringe pattern appears from the second mirror.
3. The mirrors are then rotated counter to the table rotation until the pattern appears from the first mirror.
4. Repetition of the above steps is continued until the table has been rotated 360° or some multiple of 360° , plus or minus the amount of error in the mirror angle multiplied by the number of steps taken.
5. Compensation for the error may be made by resetting the mirrors or by arithmetic elimination.

It is apparent that the angle interferometer is, therefore, merely an ultrasensitive circle divider. Its accuracy is largely dependent upon the number of steps taken during the process of error determination. Accuracy is also dependent upon the magnitude of temperature variations during the setting and measuring cycle, sensitivity of

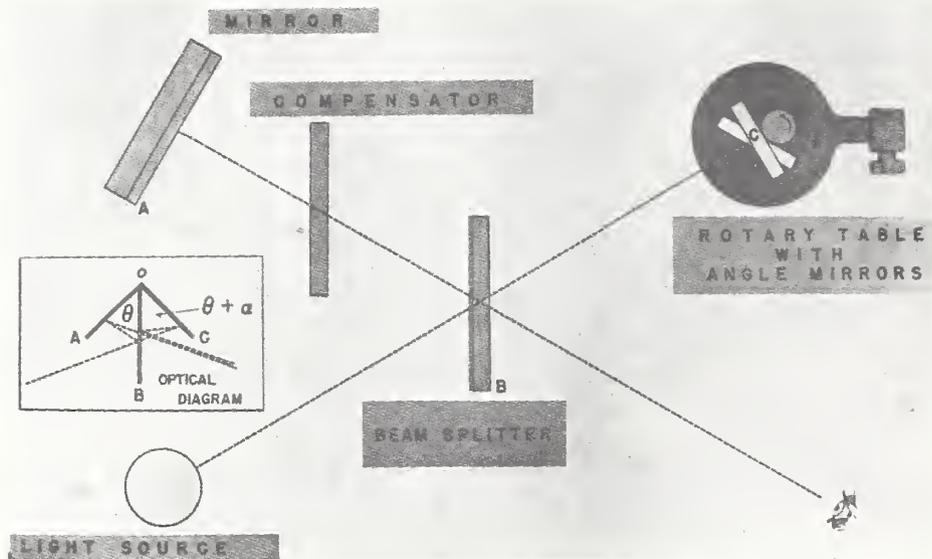


FIGURE 1. Diagram of the angle interferometer.

components to temperature variations, and to the optical resolution. The effects of temperature changes are essentially the same as those encountered in any gaging work, but the effects of resolution as determined by the aperture of the system, or effective mirror size, do need explanation.

Referring to figure 1, the back mirror, A, and the beam splitter, B, make an angle θ , which is matched as closely as possible in setting rotatable mirror C. The residual error, α , which is always undetectable, is determined by the length OC , which is the width of rotatable mirror seen in reflection from the various mirror surfaces. This arises from the fact that fringe deviation can only be accurately estimated to about two-tenths of the spacing and, as a second of arc is 4.85×10^{-6} radians, simple geometry determines that if OC is 1 in., α will be about ± 0.443 sec when the green line of mercury is used as a light source. α may be reduced to ± 0.1 sec by making the length of the rotatable mirror about 5 in. and the size of the other glass parts comparable. Of course, sensitivity may also be increased by a more precise method of determining fringe location than by visual observa-

tion of monochromatic fringes, but this complicates the system and will not be discussed now.

3. Design and Construction Details

Figure 2 shows the angle interferometer set up for use. In order to minimize temperature effects, all metal parts are made of Invar, heat treated to provide nominally a zero coefficient of expansion.⁴ Not entirely relying upon the attainment of a zero coefficient, however, the apparatus for holding, adjusting, and rotating the angle mirrors has been made with as few parts, as compact, and as symmetrical as possible. This is the real heart of the instrument, since a shift of a few millionths of an inch in some parts during use could introduce an error greater than the accuracy attempted in measuring or indexing.

As previously indicated, the width of the angle mirrors is 5 in. The back mirror and the plates are 6 in. in diameter with all mirrors made of fused quartz and the interferometer plates of selected crown



FIGURE 2. *Angle interferometer set up for use.*

The light source appears at the left with the angle mirrors and rotary mechanism at the right.

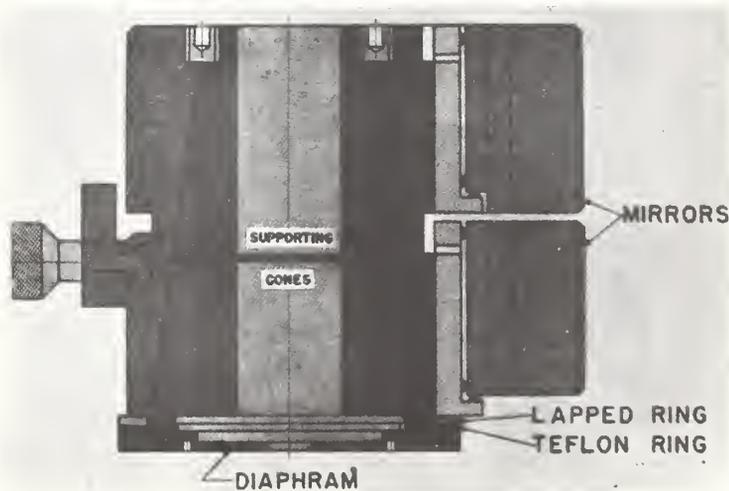


FIGURE 3. *Cross section of angle mirror assembly.*

The equipment for fine adjustments is not shown.

⁴ B. S. Lement, B. L. Averbach, and Morris Cohen, The dimensional behavior of Invar, *Trans. Am. Soc. Metals*, 43, 1072 (1951).

glass. Flatness and parallelism were held to tolerances better than $\frac{1}{20}$ wavelength.

A cross section of the angle mirror assembly is shown in figure 3. Provision is made for fastening the unit to the rotary table through a diaphragm to prevent any distortion of critical parts. The carrier for the bottom mirror rests principally upon a thin Teflon ring (0.002 in. thick) and partly upon the lower cone. The upper cone, attached to the lower carrier, provides a similar seating arrangement for the upper mirror carrier. The cones are plated with 0.001 in. thick porous chromium of the channel type. Before assembly, watch oil was applied to the chrome and then wiped as dry as possible.

Proper weight distribution between cone and base was achieved by lapping the Invar ring, shown above the Teflon, until smooth rotation without any tendency for "stick-slip" action was achieved with absolute positioning of the mirrors. Use of the full mirror width is achieved by mounting the mirrors with spring clips inserted in grooves cut into their edges near their back surfaces. This also minimizes distortion from mounting and from changes conceivably occurring in the metal parts.

4. Results and Conclusions

The instrument has just been completed and checked, but has not yet been placed in service for measuring, calibration, or indexing. An interesting feature is that it is essentially self-checking—by necessity, because no other standard is sufficiently accurate; and by nature,

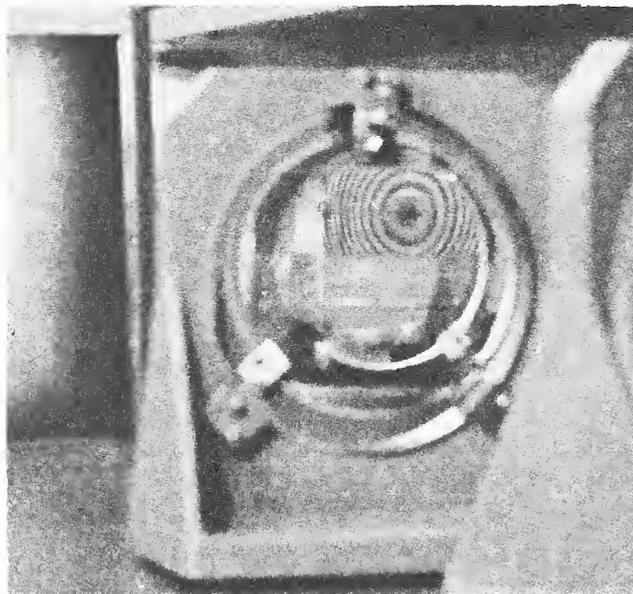


FIGURE 4. *Fringe pattern as seen when the top mirror is in position.*

because deviations of 0.1 sec may be detected by a suitable number of steps in closing the circle over a multiple of 360° .

The fringe pattern as seen by the eye is shown in figure 4. Zero position of the mirrors is determined when no change in the fringes occurs as the eye is moved to sweep the center of the pattern from

one edge of the mirror to the other. A deviation of one second is shown by two fringes appearing or disappearing in the center, with other deviations showing proportional changes.

Stability of the instrument was checked by setting the mirrors to zero angle (within ± 0.1 sec.). No change could be noted over a 2-hr period, which might be a normal usage time for determining a setting and making a measurement. Over a week end no change could be detected in the position between the angle mirrors, but a drift of 2 sec occurred in the unit as a whole. This was probably in the bed on which the instrument was mounted or in the rotary table. No attempt was made to control the temperature closely, and variations of 50 deg F or more have been deliberately introduced by directing a stream of hot air on one side of the angle mirror assembly without noticeable effect.

It is expected that the angle interferometer will find widest use in the calibration of rotary tables and checking of angle blocks. However, it should also be suitable for direct measurement of many precise machined parts, and it is expected that under some conditions it may be usable for direct indexing in precision machining operations.

Without the previous development work done by T. J. O'Donnell, Physical Sciences Development Shops, University of Chicago, the design and construction of our instrument could not have been undertaken. We are indebted to him and to Fred C. Victory of the Moore Special Tool Company, Bridgeport, Conn., for the construction details of their interferometers and for valuable suggestions which led to our design.

L. C. Eichner of L. C. Eichner Instruments, Clifton, N. J., contributed greatly to the design and constructed the mechanical components. Peter Lenart, Jr., Ferson Optical Company, Ocean Springs, Miss., did an excellent job in grinding the plates and mirrors to tolerances more exacting than the normal requirement for an interferometer.

7. Effect of Unstable Thermal Conditions During the Testing of Long Gage Blocks

By J. C. Moody¹

Metrologists generally agree that a principal source of error in the making of precision measurements is the variation in temperature between the piece to be measured and the measuring reference. Factors such as vibration, lack of cleanliness, and crowded working conditions also contribute to the measuring errors, but fortunately these can be easily understood and controlled. Temperature problems are not equally simple.

Uncontrolled heat transfer by convection, conduction, and radiation directly affects the temperature of everything in the measuring area. These are fundamental factors, and metrologists at the top measuring echelon—the National Bureau of Standards—consider them all in their efforts to reach the sixth decimal place by means of interferometry.

But what of the industrial laboratory where long gage blocks are tested against masters certified by the Bureau to ± 0.000001 inch per inch of length? The thermal factors so carefully considered by the Bureau also affect the accuracy of work done in industrial laboratories. However, it is both impractical and unnecessary for such laboratories to carry their work to the same degree of closeness as is needed by the Bureau.

The studies described here were undertaken by Sandia Corporation primarily in an effort to standardize a simplified but reliable procedure for the testing of long gage blocks. The principal variables affecting this operation are temperature, geometric shape of the subjects, the relative location of the master and sample during the thermal-equilibrium period, the length of the thermal-equilibrium period, and the finish and color of the surfaces. It was assumed at the outset that if a quantitative measure of the length caused by these variables could be clearly established, industrial gage laboratories could use this information to increase their accuracy in measuring long gage blocks.

A light-colored, 20-in. Hoke gage block that had been certified by the National Bureau of Standards to be 20.000040 in. long was selected as the master for this test; a darker-colored, 20-in. working Hoke gage block was selected as the sample. A 24-in. comparator equipped with a 10,000 \times magnification millimeter box with a full scale of 0.0005 in., each graduation 0.00001 in., was the measuring device used. An accurate temperature recorder completed the required equipment.

The first step was to determine the difference in length between the master and sample. Since the temperature of the measuring room remained constant within $\frac{1}{2}$ deg F during the 12 hr immediately preceding the start of the work day, the nearest approach to thermal equilib-

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rium occurred just at the beginning of each work day. To take advantage of this fact, the test pieces were set up for calibration in the afternoon and readings taken at the start of the work day. These preliminary tests were repeated daily for a week and showed that the sample was 0.000030 in. longer than the master.

During the first day after this preliminary calibration had been completed, the temperature in the measuring room was erratically cycled through an extreme temperature variation of 6 deg F. The master and the sample were spaced 5 ft apart on the work bench between tests, and readings were taken at intervals of approximately one hour throughout the day. Under these extremely unfavorable conditions, a maximum deviation of -0.00007 in. from the established value of the sample was obtained.

The identical procedure was repeated the next day except that the temperature was controlled to a gradual rise of 3 deg F from 7:30 A. M. to 4:30 P. M. In spite of this greatly reduced temperature variation, the maximum deviation of -0.00007 in. from the established value was again recorded.

In an effort to determine the cause for this behavior, on the following day the relative positions of the master and sample between tests were reversed. The temperature was again controlled to a gradual rise of 3 deg F during the day. This time a maximum deviation of $+0.00010$ in. from the established value was recorded. The fact that the deviation in this test was in the opposite direction from that found the previous day indicated that thermal conditions differed between the two areas 5 ft apart in which the subjects were kept between the tests. This indication was confirmed by velometer tests which showed the air velocity in the one area to be greater by a factor of 5 than that in the other area.

For the fourth test, the temperature was again allowed to increase 3 deg F during the day, but this time the subjects were stored immediately adjacent to each other in that area of the bench where the air currents were negligible. For the first 4 hr the established difference of $+0.00003$ in. was read on each hourly test. But the reading taken at 1 P. M. showed a difference of $+0.00006$ in., indicating a change of $+0.00003$ in. from the established value. This change remained constant throughout the rest of the day.

Those results indicated that the conditions allowed to prevail during this test were not adequate to insure minimum measuring error. But two interesting questions were raised by the test: First, why was the change in length preceded by a lag of 4 to 5 hr? Second, why did the change occur so abruptly and then stabilize?

A review of the results obtained to this point suggested that the effect of the difference in color between the sample and the master on the magnitude of over-all error was considerable. In an attempt to evaluate this effect, another test was undertaken. The temperature control unit of the measuring room was allowed to function normally so that the room temperature was held at 68 to 68.5° F.

Both gage blocks were set up on the base of the measuring machine and were left there throughout the test so as to minimize the necessary handling. With all the lights left on in the measuring room, a gradual growth of 0.00001 in. in the sample was observed. When the overhead lighting was reduced, the size of the deviation diminished accordingly. Apparently this effect was a result of the difference in reflectance values between the darker colored sample and the lighter master.

More radiant heat was being absorbed by the darker subject, causing it to expand more than did the lighter. Here then is a culprit easily overlooked—radiant heat from overhead lights.

To bring these experiments to a conclusion, one more test was necessary. The conditions of the previous test were duplicated except that all lights were turned off except one small unit in the corner which gave only the minimum light needed to read the instruments. Under these conditions, the results of the hourly tests were consistent. The

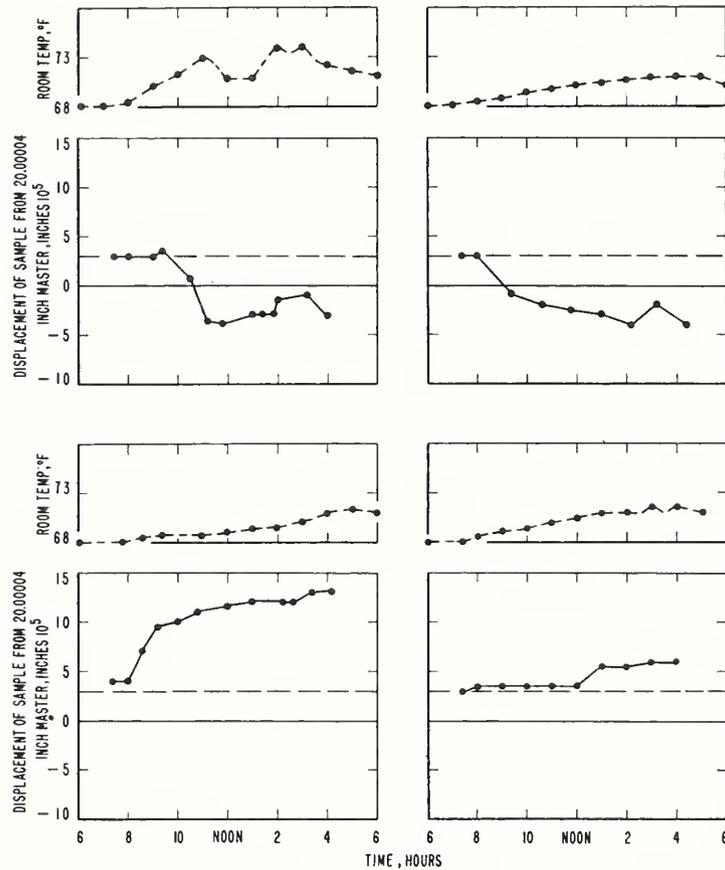


FIGURE 1.

length of the sample remained 0.00003 in. greater than that of the master.

On the basis of this series of tests, a technique for the testing of long gage blocks was established. This technique requires: (1) that the master and sample be allowed to stabilize thermally in an area where the thermal conditions are uniform; (2) that the subjects be placed on the comparator at least 1 hr before calibration; (3) that the temperature in the measuring area be held stable within $\frac{1}{2}$ deg F; (4) that the subjects be protected from direct illumination; (5) that the master and sample be of the same geometric shape; and (6) that the measuring area be carefully studied for possible thermal instability due to air currents and due to radiation from the walls or from other heat emitting sources in the measuring room.

A practical test was conducted the following week. The master and the sample were taken out of storage, thoroughly cleaned, and left on the work bench for 24 hr to stabilize. The next morning the lights were reduced to a bare minimum and the gage blocks set up on the comparator base. A reading was taken 1 hr later. The sample measured 0.00003 in. longer than the master, the exact difference established at the outset of these experiments.

8. Secular Length Changes of Gage Blocks During Twenty-five Years

By Walter B. Emerson¹

Secular changes in length during twenty-five years were determined for nineteen steel end gage blocks of known composition and heat treatment. Although the composition and heat treatment of all gage blocks were reportedly the same, some of the blocks appear stabilized after an initial decrease in length, whereas others continue to increase either at uniform or reduced rates.

The material commonly used for gage blocks is hardened steel, but if this material is not stabilized by proper heat treatment, large secular changes in the gages may occur. Changes in length of steel blocks subjected to various heat treatments have previously been observed over relatively short periods, but to my knowledge no accelerated aging test has been devised that can definitely be correlated with changes that may take place in a gage block under normal conditions over a long period. Changes that have occurred in NBS gage blocks during 25 years may therefore be of interest and possibly of value.

A series of four 1-in., two 2-in., three 4-in., two 50-mm, and two 100-mm AA quality Johansson steel gage blocks (set 1010) of known composition and heat treatment² was purchased in 1929. These were selected by C. E. Johansson for excellence of planeness, parallelism, and surface finish. Their lengths were determined upon receipt and at irregular intervals thereafter. In addition to these gage blocks, four 1-in., one 2-in., and one 4-in. Johansson blocks of probably the same composition and heat treatment were obtained in 1927 (set 410). Data on these are included in this report.

Absolute lengths of the gage blocks were determined by the interferometric method described by Peters and Boyd.³ This is essentially a modification of the Fabry-Perot method, using the gage blocks as separators for the interferometer plates. The four 1-in. blocks obtained in 1927 (set 410) were calibrated by this method and then sent to the National Physical Laboratory, Physikalisch-Technische Reichsanstalt, and the Bureau International des Poids et Mesures to determine the uniformity of interferential calibrations. Measurements of the individual laboratories differed from the mean by 0 to 1 μ in. Excellence of agreement of the international laboratories was further confirmed by calibrations of decimeter end gages in 1935. These gages were made of fused quartz to minimize the effect of temperature. The comparisons gave the same lengths to an average deviation of 0.016 μ (1 part in 6 million) and to a probable deviation much less.

Gage blocks of the same material and surface finish, and of the

¹ National Bureau of Standards, Washington.

² Confidential data furnished by Edward C. E. Johansson.

³ C. G. Peters and H. S. Boyd, Interference methods for standardizing and testing precision gage blocks, BS Sci. Pap. 17, 677 (1922) S436.

same approximate length may be accurately compared by the more rapid Fizeau method (see footnote 3).¹

Lengths here reported are based upon combinations of the two methods. Absolute lengths of a few of the gage blocks were determined by the Fabry-Perot method. Lengths of the other gage blocks were then obtained by comparison with these. This procedure may involve an error of perhaps 1 to 2 μ in. in some gage blocks, as for example when a 4-in. gage block is determined by comparison with a combination of two 2-in. gage blocks, the length of one of the 2-in. gage blocks having been determined by the Fabry-Perot method.

Another factor to consider is wear resulting from wringing. Rolt⁴ has shown that lengths decreased 2 μ in. by 200 wringings for gage blocks with surface finish comparable to these and thereafter remained constant. Practically all change results from the first 50 wringings. It is considered probable that most of the wear of the NBS gage blocks took place within 5 years after the initial measurements.

Figures 1, 2, 3, and 4 give differences from nominal lengths during a period of approximately 25 years for the set 410 and the set 1010 gage blocks.

Figure 1 shows increases in length ranging from 3 to 21 μ in. for set 410 (1 in.) gage blocks. The rate of increase appears to be maintained after 25 years. Any wear of the gage blocks during the first few years is masked by the tendency of the gage blocks to increase in length. Set 1010 (1 in.) gage blocks Nos. 1, 2, and 3 show a small initial decrease in length and then remain quite constant. Number 4 increased 12 μ in. and was still increasing but at a less rapid rate after 25 years.

The 2-in. gage blocks, Nos. 1 (set 1010) and 2 (set 410), (fig. 2), showed a decrease of 4 μ in. after 7 years and then remained constant or possibly increased slightly, whereas No. 3 (set 1010) increased for 25 years, although at a less rapid rate since 1942. The 1951 value for this gage is somewhat questionable as it was obtained from comparisons that involved errors of wringing that could increase the determined length 1 to 2 μ in., whereas the 1955 value is based upon direct absolute determinations.

All 4-in. gage blocks (fig. 3) grew at fairly constant but different rates for the individual gages from 1929 to date, and give no indication of leveling off.

The 100- and 50-mm gage blocks (set 1010) (fig. 4) decreased 0.160 μ (6 to 7 μ in.) from 1929 to 1936 and thereafter remained constant. This initial decrease in length appears greater than would be expected from wear only.

At the suggestion of E. S. Rowland and his associates, at The Timken Roller Bearing Company, two gage blocks (2 in. No. 1 and 1 in. No. 1, set 1010) that appear to be stabilized and two (2 in. No. 3 and 1 in. No. 4 set 1010) that appear to be increasing in length were sent to their laboratory in 1955 for determinations of retained austenite and residual stress.⁵ The results of their findings were presented at the Symposium and are given in this Circular.

⁴ F. H. Rolt, Gages and fine measurements, vol. 1, p. 169, 170, and 171, (Maemillan and Co. Ltd., 1929).

⁵ Determination of retained austenite required that an area approximately $\frac{3}{4} \times \frac{3}{4}$ in. be etched on one side of the gage blocks. Etching appears to have changed the direction and magnitude of the slope of the gage-block surfaces. Measurements at the Bureau indicated that previous to etching, the length of the gage blocks at the etched side was slightly greater than that at the opposite side. After etching, the length at the etched side was the lesser. The observed changes in differences of length at the two sides of the gage-blocks before and after etching were: 2 in. No. 3, 15 μ in.; 2 in. No. 1, 7 μ in.; 1 in. No. 4 set 1010, 18 μ in.; 1 in. No. 1 set 1010, 8 μ in. The differences were greater for gage blocks that were changing in length than for those that appear to be stabilized.

The length changes observed in the present gage blocks are small in comparison with several other gage blocks measured. Unfortunately the composition and stabilization procedures for the latter are not known.

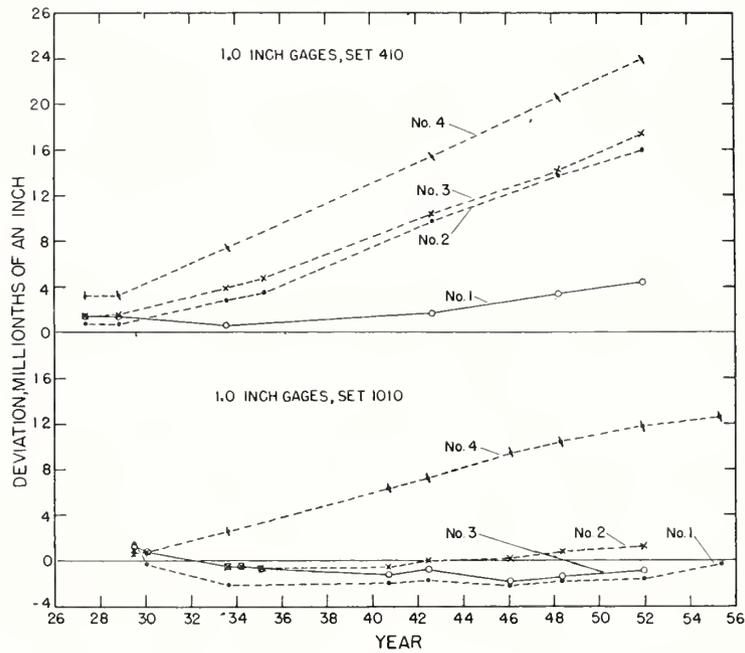


FIGURE 1. Length changes of 1-inch gage blocks.

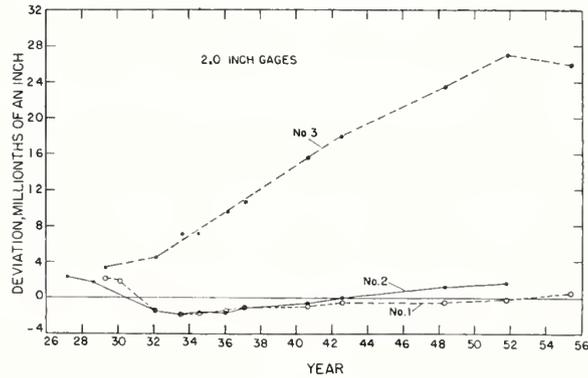


FIGURE 2. Length changes of 2-inch gage blocks.

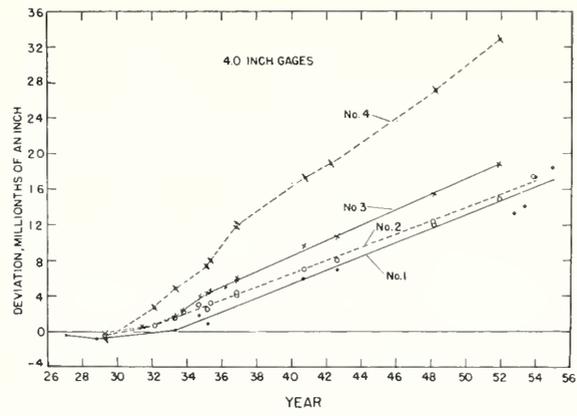


FIGURE 3. Length changes of 4-inch gage blocks.

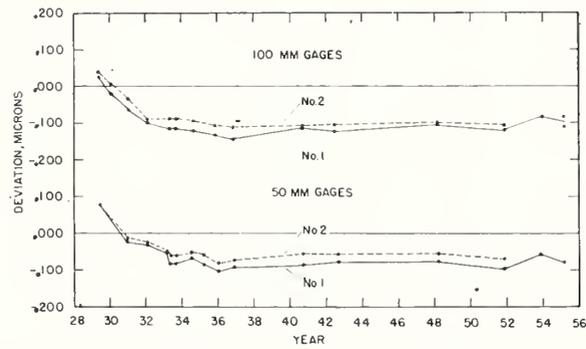


FIGURE 4. Length changes of 100- and 50-millimeter gage blocks.

9. Retained Austenite and Residual Stress Measurements on Certain Gage Blocks

By A. L. Christensen¹

The Timken Roller Bearing Company laboratory was given the opportunity of examining four gage blocks whose length changes have been carefully followed for the past 25 years or so. Two of the gages are nominally 2 in. in length and two, 1 in. One each of the two different sized gages has remained remarkably stable in length during this period of time, whereas the other two have grown uniformly in length in an amount totalling approximately 13 $\mu\text{in./in.}$ The blocks were prepared from 1.3-percent-carbon steel at the Ford Motor Company in 1929.

It was the purpose of this examination to determine if there were residual stress differences or variations in retained austenite contents in the blocks that possibly could account for their rather marked difference in growth behavior. Retained austenite is an unstable nonequilibrium phase in hard steels, with a propensity to transform to martensite with an accompanying increase in volume. Relaxation of residual stress may also cause distortion of the steel samples, and in this connection it is of interest to note that martensite is normally considered to be strain nucleated, and, hence, any plastic flow that might occur has the double effect of stress relaxation and austenite transformation. In other words, transformation of austenite may alter the stress pattern, and relaxation of the stress pattern may in turn influence the austenite to martensite transformation.

The measurements of residual stress and retained austenite in these gage blocks were made by the use of X-ray diffraction techniques, and they are described briefly before presenting the results.

The measurement of stress by X-ray is, as in other techniques, a measurement of strain and not stress (fig. 1). The interplanar distance, d , of a selected family of crystalline planes in the phase under study is used as an internal indicator of strain present, and in the two-exposure method illustrated here is determined at two or more ψ angular orientations of those planes to the direction in which it is desired to measure the stress. The angles normally used are 0° and 45° , or 0° and 60° .

These d values may then be equated to the stress by means of a theoretically derived expression, one form of which is shown in figure 2, or by means of a simple constant of proportionality, usually called the stress constant. In the derivation of this expression it is assumed that the value of E , Young's modulus, and ν , Poisson's constant, remains independent of the orientation of the lattice planes to the direction of stress, or that the material is isotropic. However, if the

¹ The Timken Roller Bearing Co., Canton, Ohio.

stress constant is experimentally ascertained, lack of isotropy does not impair the usefulness of the method.

There is, on the other hand, a problem that exists in the measurement of stress in hard steels, which is not ordinarily present. We note in figure 3, that whereas the diffraction lines are well defined as a whole, they are broad and possess ill-defined peaks, which make

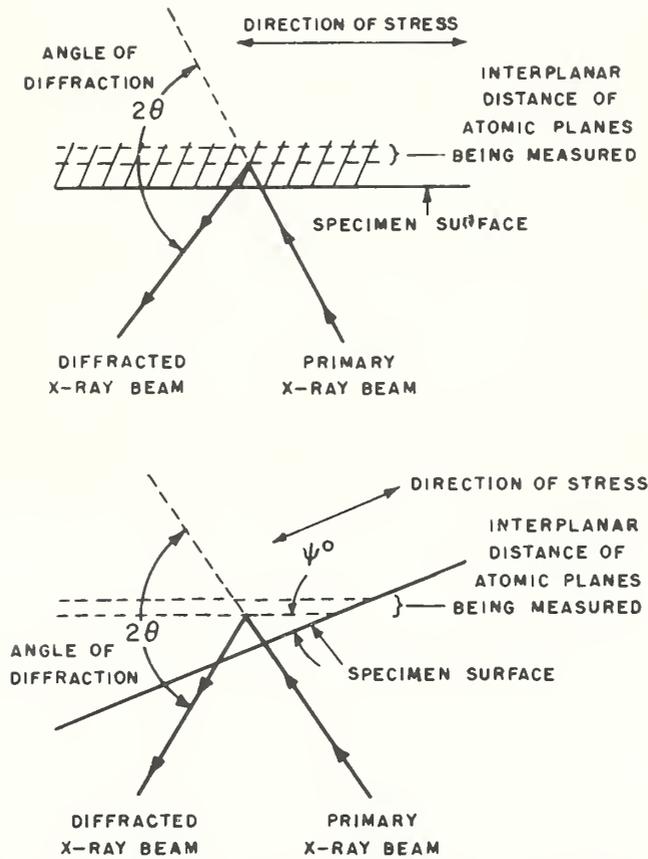
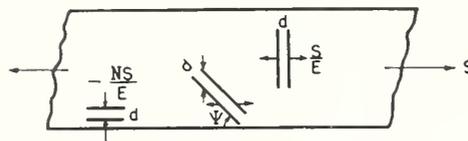


FIGURE 1. Orientation of measured lattice planes to direction of stress.

$$d_{\psi} = \left[\frac{(1+N)}{E} d_0 S \right] \sin^2 \psi + d_0 \left(1 - \frac{NS}{E} \right)$$

OR $d_{\psi} = C \sin^2 \psi + B$



$$S = \left(\frac{d_{\psi} - d_1}{d_1} \right) \left(\frac{E}{1+N} \right) \frac{1}{\sin^2 \psi}$$

OR $S = K \Delta d$

FIGURE 2. Expression for relation of residual stress to interplanar distance.

it difficult to determine their positions with sufficient precision for adequate stress measurement. Fortunately, in stress measurement, we are not concerned with absolute line positions but only in the relative line position obtained at each ψ angle used. Thus, we may take advantage of the fact that the line sides are linear over a fair range and extrapolate them to their peaks for their relative positions. But one complication yet remains. A change in line symmetry is observed as the sample is shifted from one ψ angle to another, and

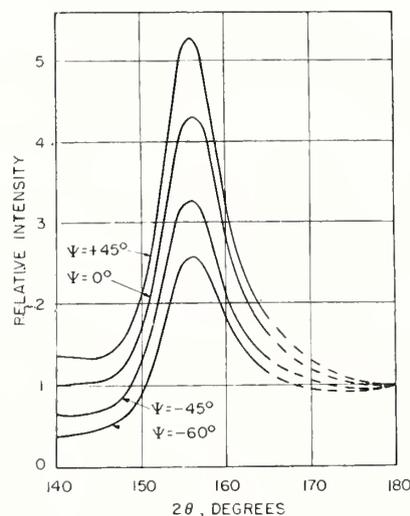


FIGURE 3. Variation of line shape with angle ψ .

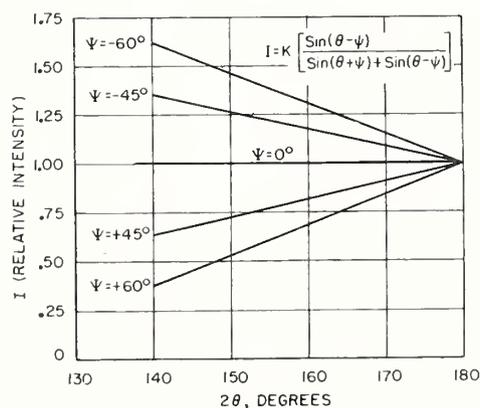


FIGURE 4. Absorption effect.

this variation in symmetry is sufficient to cause an appreciable error in the measured line position.

Analysis of the cause for this change in line symmetry discloses that the X-ray beam absorption within the sample results in the diffracted X-ray intensity becoming a function of the diffraction angle 2θ (fig. 4), and that this function is different for each ψ angle. Consequently, suitable corrections may be applied to the intensity readings obtained and an accuracy of 2 or 3 thousand pounds per square inch in stress measurement can be achieved.

The measurement of retained austenite is far simpler than the measurement of stress (fig. 5). The integrated intensity or total area under a line diffracted from the austenite phase is determined and compared to an area under a line from the ferrite or martensite phase. The ratio of these two areas multiplied by a suitable constant gives the percentage of austenite present. It is evident that determining the true line intensities necessitates subtracting out the

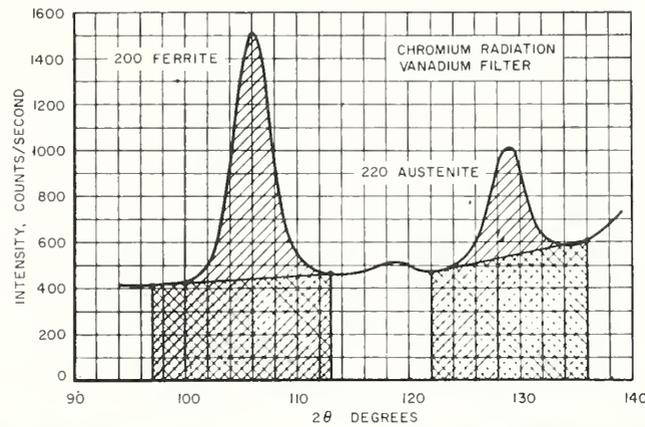


FIGURE 5. Retained austenite measurement.

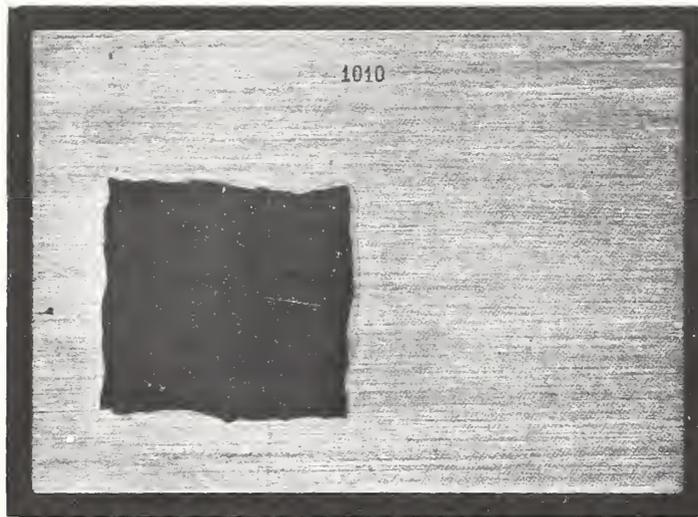


FIGURE 6. Two-inch gage block No. 1010.

background, and it is equally obvious that the background level cannot always be accurately established. Hence, ordinarily one uses as many lines of both the austenitic and ferritic phases as possible to average out this uncertainty. However, in the measurements made on these blocks, only the two lines illustrated here were used. Therefore, we hesitate to claim an absolute accuracy greater than plus or minus 10 percent of the austenite present, although on a relative basis the results are very much better than this.

Figure 6 is a photograph of one of the 2-in. blocks examined. The black area is the area in which the measurements were taken and is the result of etching approximately four thousandths stock from the

surface to get below the effects of grinding. The etching was done in a solution of 5 percent nitric acid, 95 percent water, at room temperature.

Measurements of stress were made, before and after the stock removal, in the direction of the grinding and also transverse to this direction. Although it has been commonly believed that grinding induces tensile stresses, experience at Timken has indicated that good grinding practice invariably introduces compressive stresses of the order to 100,000 lb/in.² in the direction of grinding and somewhat higher transverse to this direction. These grinding stresses normally do not penetrate more than 0.004 in. deep.

It is observed in table 1 that our expectations of the residual stresses in the as-ground surfaces were borne out. However, 0.004-in. stock removal was insufficient to remove the effect of grinding—at least in the 1-in. blocks—because through-hardened pieces of this size and geometry ought to be slightly in tension on the surface. Time did not permit examination of the stress condition at greater depths. Nevertheless, we believe the results indicate first that the grinding of the 1-in. blocks was somewhat different from the grinding of the 2-in. blocks, and second, that there is no significant stress variation between the two blocks that remained stable and the two that grew approximately 13 μ in./in.

TABLE 1. *Stress measurements*

Block	Nominal length	Approximate change in length	Surface stress (as-received)		Stress (0.004-in. stock removed)	
			Longitudinal	Transverse	Longitudinal	Transverse
	<i>in.</i>	μ <i>in./in.</i>	<i>lb./in.²</i>	<i>lb./in.²</i>	<i>lb./in.²</i>	<i>lb./in.²</i>
2-B4-2	1	<-1	-143,000	-191,000	-25,000	-4,000
4-B4-2	1	+13	-152,000	-203,000	-20,000	-6,000
2-B1-6	2	<+1	-136,000	-199,000	-5,000	-2,000
1-B1-5	2	+13	-141,000	-203,000	+1,000	+4,000

TABLE 2. *Retained austenite measurements*

Block	Nominal length	Approximate change in length	Surface austenite (as-received)	Austenite (0.004-in. stock removed)
	<i>in.</i>	μ <i>in./in.</i>	%	%
2-B4-2	1	<-1	8.6 to 8.7	10.1 to 10.9
4-B4-2	1	+13	9.3 to 9.5	14.0 to 14.0
2-B1-6	2	<+1	6.7 to 7.1	10.3 to 10.4
1-B1-5	2	+13	13.7 to 13.9	18.8 to 19.8

The retained-austenite measurements, however, are considered significant. It is observed in table 2 that at a depth of 0.004 in., the retained-austenite contents of the two blocks that increased 13 μ in./in. are appreciably higher than in the other two blocks. Those measurements taken on the as-ground surface do not indicate this marked difference in the instance of the 1-in. blocks, but this fact is unimportant because grinding causes transformation of the austenite.

These data are obviously superficial and fragmentary but lead one to speculate that the difference in growth behavior was caused largely by variation in retained austenite, and had all blocks been treated to reduce the retained austenite below 10 percent, they would have all remained fairly stable and behaved pretty much alike.

10. Dimensional Instability in Gage Blocks

By Frederick C. Victory¹

With the increasing accent on higher accuracy in gage-block measurement, both comparative and absolute, in terms of wavelengths of light by means of interferometry, it seems inconsistent that more effective steps have not been introduced to insure the permanence of such accuracy. Indeed, transient accuracy can be, and often is, more dangerous than known and acknowledged errors in gage blocks.

Physically measurable errors in geometry, poor surface finish, and deviation from nominal size can be allowed for in the use of gage blocks. The more subtle, and frequently greater errors resulting from permanent dimensional instability present a hazard of unsuspected dimensional change in blocks, after measurement and certification.

Hardened steel, in addition to its well-ordered obedience to the laws of thermal expansion, is subject to erratic and entirely unrelated dimensional variations of considerable magnitude. These changes can be either shrinkage or growth and may occur under the following conditions:

1. A slow, progressive change, continuing for months or years, which may start immediately after hardening or at any time thereafter.
2. An instantaneous and complete change at any time after hardening.
3. A reversal of direction of change at any time after hardening.

It is well recognized that a variety of unstable constituents may be produced during the hardening cycle of steel, depending upon the analysis and the temperature : time relationship. Some of these constituents are transitory, and are not normally carried down to room temperature, except in unstable condition. Other constituents are considered end products of the reaction and are, in themselves, unstable.

The entire problem of dimensional instability may be resolved in terms of the crystalline structure of these constituents as affected by the atomic rearrangement within them. Recent research² on the problem has revealed by dilatometric and X-ray diffraction examination that transition phases during the cycle result in shifts in atomic arrangement within the structure and are attended by volumetric changes in the steel.

The two offending constituents in hardened steel are austenite retained at room temperature after quenching and untempered, or tetragonal, martensite.

At this point consider the reaction of steel to the hardening cycle, for example, an oil-hardening steel of the low-alloy type.

¹ Moore Special Tool Co., Bridgeport, Conn.

² The physics of metals, Frederick Seitz (McGraw-Hill Book Co., New York, N. Y., 1943).

Upon being heated from room temperature, at which it exists in the annealed state as a spheroidized structure, the steel obeys the laws of thermal expansion until a so-called critical temperature is reached. At this point the steel undergoes a marked change in crystalline structure, entering what is known as the gamma phase, the product being austenite. This transformation is evidenced by several observable phenomena, the one pertinent to the discussion being decrease in atomic volume, or shrinkage. To restate this, austenite is less voluminous than the structure from which it is formed³ (see fig. 1).

On the cooling side of the cycle, the behavior of the steel is, to a great extent, governed by the rate of cooling. If allowed to cool slowly the steel reexpands to its normal volume for that temperature, at a point somewhat below the temperature that marked its original transformation. In this case the resulting product will be pearlite. Continued cooling to room temperature will restore its volume to that before treatment.

But if cooling is sufficiently rapid, as in the case of a quench, re-expansion at the secondary critical temperature (an isothermal reaction) is avoided, and austenite is carried down to a much lower temperature. At this point (approximately 400° F) a direct transformation from austenite to martensite begins and continues until room temperature is reached or cooling is otherwise interrupted. This austenite-martensite reaction is, again, a result of atomic displacement within the crystalline structure, and is accompanied by an increase in volume (see fig. 1) to an extent exceeding that to which steel owes its hardness characteristic, and in this stage, it is unstable.

In the untempered stage, martensite has a tetragonal crystalline structure, which tends to transform, over a period of time, to a stable cubic structure of appreciably smaller volume. This accounts for the shrinkage phenomenon in hardened steels that have been inadequately or improperly tempered.

Theoretically the tempering cycle specified for most low-alloy steels carries this reaction through to a reasonable degree of completion, rendering the steel relatively immune to this form of instability, and restoring it to its original volume at room temperature. Low-temperature tempering and too short a time at heat, which occur accidentally or in an attempt to attain maximum hardness, will result in something less than complete conversion and stability.

From the standpoints of maximum hardness and stability, it would be highly desirable to have the austenite to martensite transformation continue to completion. Unfortunately this is almost never the case. Because of variations in temperature and effectiveness of the quench, and a degree of reluctance on the part of the austenite to transform, a percentage of the unstable austenite with its smaller crystalline structure is carried down to room temperature. Here it can exist only precariously, and if not triggered into a sudden transformation by some form of shock, will gradually decompose into bainite; in either case with a resulting increase in volume which causes dimensional growth in the steel.

This retained austenite may vary from 2 to 35 percent, or more, depending on the specific analysis of the steel and conditions obtaining during the hardening cycle. The gravity of the problem becomes

³ B. S. Lemen, B. L. Averback, and M. Cohen, 'The dimensional stability of steel,' Part IV—Tool steels, *Trans. Am. Soc. Metals* **41** 1061 (1949).

apparent when one learns (see footnote 3) that conversion of less than 1 percent of the steel's volume will result in a growth of 0.0001 in. per inch of length.

At this point it may be noted that hardened steel almost inevitably contains two unstable constituents affecting it dimensionally in opposing directions, and to varying degrees, depending on proportion and the presence of any external triggering stimulus such as physical or thermal shock. Occasionally, and by chance, they may establish, between themselves, a precarious equilibrium, or the predominant one will control the direction of change for a time until stabilized or exhausted.

At this point it might be appropriate to consider the practical consequence of such instability in gage blocks by citing specific case—examples:

Case A

Specimen: A class "A," 4-in. gage block.

Conditions: Used for somewhat less than 1 year in a temperature-controlled room as a master block for reference.

Observation: At the end of a year comparison showed a suspected growth of 0.00004 in., verified by the National Bureau of Standards. The manufacturer replaced this block on a no-charge basis.

Conclusion: The growth could only have resulted from the decomposition of unstable, retained austenite. Whether or not a significant percentage still remained was not determined.

Case B

Specimen: An 18-in. gage, measured and certified by the National Bureau of Standards.

Observation: At the end of 2 years storage in a temperature controlled room this gage was flown to England for measurement by the National Physical Laboratory, where it was certified as being 0.0002 in. longer than before, after conversion from English to American inches had been taken into account. Naturally this brought up a question as to the validity of the new length determination. Subsequent measurement by the National Bureau of Standards confirmed the fact that the gage had, indeed, grown slightly over two ten-thousandths inch. Whether or not this was an isothermal reaction, i. e., at room temperature, or had been the result of the low temperature (-45° F) encountered in the baggage compartment of the aircraft could not be determined.

Conclusion: Frequent periodic measurements of this gage clearly showed that it is still continuing to grow and is useless as a master.

Case C

Specimen: An 81-piece class B set of gage blocks, of which not more than a half a dozen blocks were ever used during a period of 2 years.

Observation: Casual comparisons against master blocks indicated that a few of the blocks were undersize. The entire set was returned to the manufacturer for measurement and certification. The resulting certificate proved to be a rather amazing and revealing document. Each block was short by many times the tolerance for that class of block, in nearly a direct proportion to its size. This value ranged from 0.000034 in. for a 0.50-in. block to 0.000596 in. for a 3-in. block.

Conclusion: Because the original certification of this set and the subsequent certification revealing the error were both made by the manufacturer, it is impossible to state which of two possible conditions might fit the case. Either the entire set of blocks (assumed to be representative) had shrunk as a result of the isothermal conversion of untempered, tetragonal martensite, or the masters with which they had been compared had grown an equal amount as a result of decomposed austenite. It is somewhat more likely that the former explanation is the more valid.

Although a number of additional examples could be cited, they would merely be repetitious and contribute nothing new to the discussion. More to the point would be a brief consideration as to what may be done to cope with this serious problem.

The subject of stabilizing hardened steel is highly controversial and in many cases is regarded as a closely guarded secret. For a time, subzero treatment was considered most effective. Manufacturers of commercial refrigerating equipment and their disciples proposed this as the panacea for all stabilizing problems. Unfortunately the answer is not that simple.

Although it is true that subzero treatment, as an immediate continuation of the initial quench, is capable of converting a large percentage of retained austenite to martensite, there are certain practical limitations to this method.

The thermal shock of being reduced to -150° F is very likely to produce fractures and cracks in the steel. As a result a tempering operation is introduced between the quench and the subzero treatment, with the unfortunate result of rendering the latter largely ineffective. In this case the austenite becomes sufficiently stabilized to resist conversion by low temperature but will still decompose isothermally.

An additional source of trouble results from this method. The conversion product of austenite under subzero conditions is tetragonal, unstable martensite, previously cited as being responsible for the shrinkage sometimes noted in gage blocks. It is a reasonable assumption that this sort of cycle was responsible for the shrinkage noted in the 81-block set previously discussed. At best, the subzero cycle can only be expected to affect one of the two unstable constituents found in hardened steel.

Conversely, ordinary tempering at the temperature:time ratio recommended by steel manufacturers can be expected only partially to effect conversion of the other unstable constituent. Unfortunately, these two treatments are largely incompatible in combination.

Recent research at several of our leading technical institutes has revealed that a somewhat unorthodox tempering cycle provides an effective method of simultaneously coping with both undesirable constituents.

Although it is not the purpose of this discussion to recommend specific cycles for each type of steel, one example will serve to illustrate the point. In the case of an oil-hardening steel of the analysis 0.90 C, 1.70 Mn, and 0.25 Si, the conventional treatment would be to quench from $1,420^{\circ}$ to $1,440^{\circ}$ F and draw for 1 hour at 350° F. This would result in a commercially acceptable structure for ordinary tool work, but not for gage blocks.

If, however, the austenitizing temperature is carefully controlled at the low side of the range, say 1,425° F, ±5 deg F (no small problem in itself), a smaller percentage of austenite will be carried down to room temperature than otherwise would be the case. Then, with a draw of 400° to 410° F continued for 18 hours, the remaining austenite is completely converted to stable bainite, and the tetragonal martensite is simultaneously converted to stable cubic martensite. The resulting structure, although its hardness is about three points lower than maximum for this analysis on the Rockwell C scale, will be entirely stable dimensionally.

Potential stability or instability can be determined within varying degrees of accuracy by several methods, destructive and nondestructive.

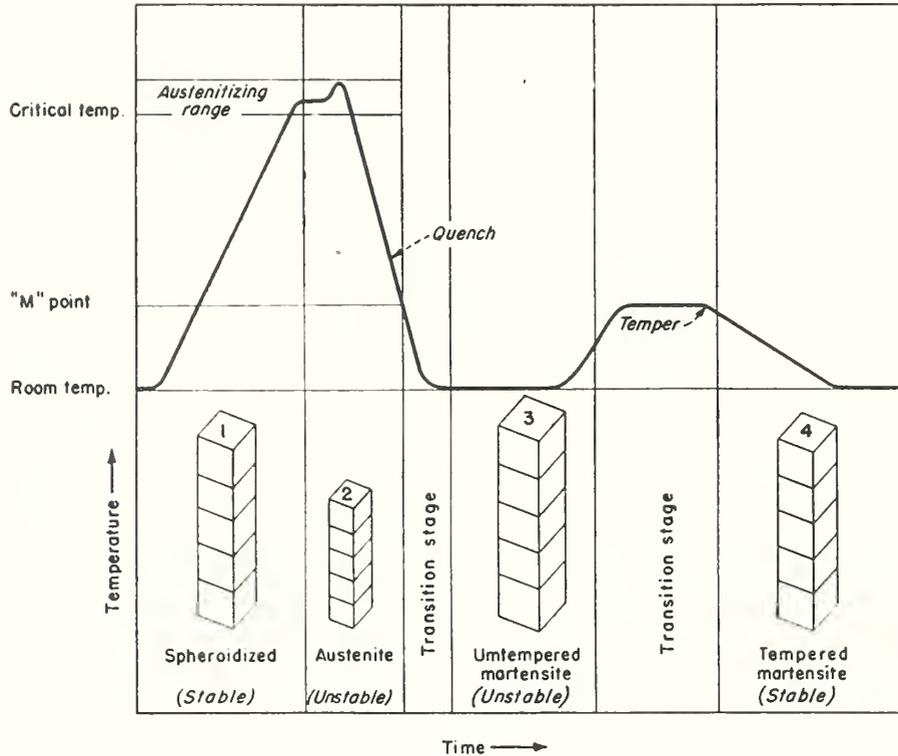


FIGURE 1.

Volume changes occur in steel, according to the content of various crystalline structures. Here is shown the situation resulting from a theoretically ideal hardening cycle. Structures 1 and 4 are stable and equal in size (chart not drawn to scale).

tive. At one time it was the practice of the National Bureau of Standards to determine this dilatometrically by boiling the specimen at 212° F for 24 hours and remeasuring. If no change in length occurred, the gage was assumed to be stable. The writer's experience indicates that this cycle is inadequate and inconclusive. The minimum temperature at which one can be certain of the effective conversion of semistabilized austenite is 390° F for most oil-hardening steels, consequently a more realistic and revealing test would be to subject the specimen to a 4-hour cycle in an oil bath at 400° to 410° F and then redetermine the length. The X-ray diffraction examination method is capable of determining the amount of retained austenite within about 0.5-percent accuracy. Surface examination with a micro-

scope reveals much as to the crystalline structure but seems to be less accurate than either of the previous methods. One additional method seems to offer much promise, both as to the accuracy of result and economy of use. It is based on the ability to measure electronically the differences in magnetic permeability of the specimens as affected by varying percentages of austenite and untempered martensite. The method involves comparison against standard specimens of known values.

In conclusion, it might be said that industry, in its faith and reliance on the gage block as a practical and convenient standard of linear measurement, needs and deserves a practical assurance of the permanence of its accuracy, disregarding the factor of wear.

11. Surface Characteristics of Gage Blocks

By A. G. Strang¹

The primary function of a gage block is to provide an accurate standard of length. Its accuracy, singly or in combination, is governed essentially by the planeness, parallelism, and microfinish of the surfaces. The contact surfaces must also be free from large pits, deep scratches, excessive porosity, and burrs. The quality of the surface microfinish largely governs the ability of the gage blocks to wring together, the resistance to wear, and to some extent the accuracy of length measurement by both mechanical and interferometric methods.

With the improved methods of manufacturing and grading of abrasive particles and with the wide variety of abrasives and suspending lubricants on the market, gage block manufacturers now have a simpler job in selecting suitable materials which will give them the desired surface finish. Obviously the better wringing surfaces are those that approach a true plane.

Gage blocks, to wring well, must be free from fuzz or burrs, which project above the true plane. Scratches below the plane are of secondary importance. We know that careful stoning of gage blocks frequently improves the wringing tremendously because it wears off the microscopic burrs. This is necessary on many new blocks if they are to be wrung to a clean quartz surface. Furthermore, gage blocks with excessive fuzz will decrease in length rapidly until several micro-inches of fractured metal are worn off.

There is still considerable difficulty in accurately rating comparatively smooth surfaces, especially the finish encountered on gage blocks. The tracer-type surface analyzer² used at the Bureau produces a graph, or chart, of the surface and also indicates the arithmetical average deviation from the mean plane, that is, the plane where all the peaks are leveled off to fill in the valleys. The arithmetical average deviation of the surface irregularities is defined as the average value, in microinches, of the departure of the profile from a meanline or centerline, whether above or below it, throughout the traversing length. In other words, it is the sum of all the areas enclosed between the profile and the meanline, divided by the traversing length, and is expressed by the formula

$$Y = \frac{1}{l} \int_{x=0}^{x=l} |y| dx,$$

where Y is the arithmetical average deviation, or height, and l is the length of surface traversed.

¹ National Bureau of Standards, Washington.

² American Standard B46.1, Surface roughness, waviness, and lay, American Society of Mechanical Engineers, New York, N. Y. (1955).

In Europe, this deviation is called centerline-average. To prevent confusion in this paper between arithmetical average, abbreviated AA, and Grade AA gage blocks, I have chosen to use centerline-average, abbreviated C. L. A. The centerline-average values are not too reliable for these fine finishes. In order to get the best meter reading or chart, the stylus must never trace over the same path.

The diamond stylus is a four-sided 90° pyramid truncated to 0.0001-in. width at the end. Measurements indicate that the end is rounded to approximately a 0.0001-in. radius, and therefore, this stylus can reach to the bottom of nearly all of the scratches on gage blocks.

The average width of the scratches on AA gage blocks varies from ³ 20 μin. on mirror-finished gage blocks to 80 μin. on the rougher finishes. A graph should show the true peak to valley depths of the narrow scratches but seldom does. However, it is usually possible to see a definite difference between gage blocks of different roughness. With a load of 0.1g on the tracing point, it is possible that inertia and/or friction in the pivot assembly of the tracer may prevent the point from faithfully following all the irregularities of a surface at the speed of traverse which is usually used. By reducing the tracing speed from 0.0025 in. to 0.0005 in. per second, it is possible to produce a chart which more faithfully records the profile of the scratches. By adding a correction for indentation of the diamond point into the peaks, a more reliable peak-to-valley distance is obtained.

The following figures illustrate gage-block surfaces having good and bad characteristics. The selection of gage blocks was made from sets produced by the world's leading manufacturers. Some illustrations are not characteristic of the product of the manufacturer but in most cases they are.

Figure 1 shows interference micrographs of AA steel gage blocks. The bands on the blocks are interference fringes produced by the predominant green spectrum line of thallium. The half-wavelength is 10.7 μin. Fringe displacements are proportional to elevations of surface irregularities. For example, a fringe displacement of ½ fringe downward would indicate a scratch or hole 5.3 μin. deep. Each of the photomicrographs covers an area 5.4 mils in width and 7.1 mils in length. This group of pictures shows extremes in the surface roughness of AA steel gage blocks. Note that there is a wide variation in the surface roughness of the products of different manufacturers.

Figure 2 shows interference micrographs of grade A steel gage blocks manufactured by the same four companies but note the difference in finishes. The gage block of manufacturer 1 is much rougher than the AA grade gage block in figure 1, which indicates that this manufacturer produces AA gage blocks rather than selects them from the factory run. The finish on the A grade gage block, produced by manufacturer 2, is about the same as for the AA gage block shown in figure 1 whereas the finish on the A grade gage block of manufacturer 4 is better than that on the AA gage block.

Graphs of the preceding eight gage blocks, where the trace of the surface analyzer traverses a distance of approximately 150 mils, are shown in figure 3. The coordinate spacing corresponds to 2 μin. in the vertical direction and 2 mils in the horizontal direction. Here we have

³ One microinch, abbreviated 1 μin., equals 0.000001 in.

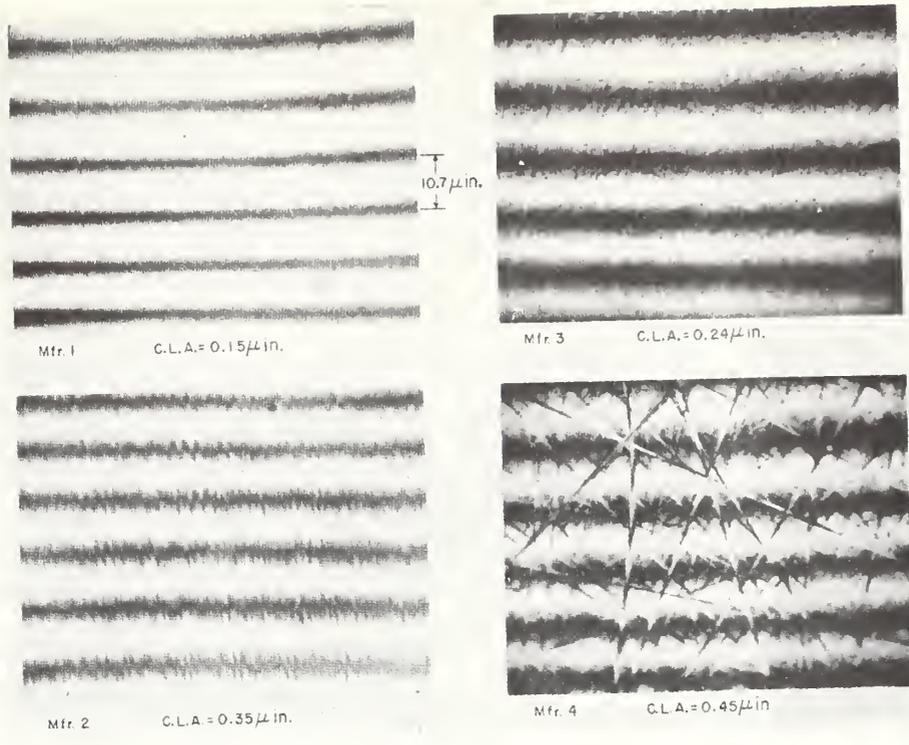


FIGURE 1. *Microfinish on grade AA steel gage blocks.*

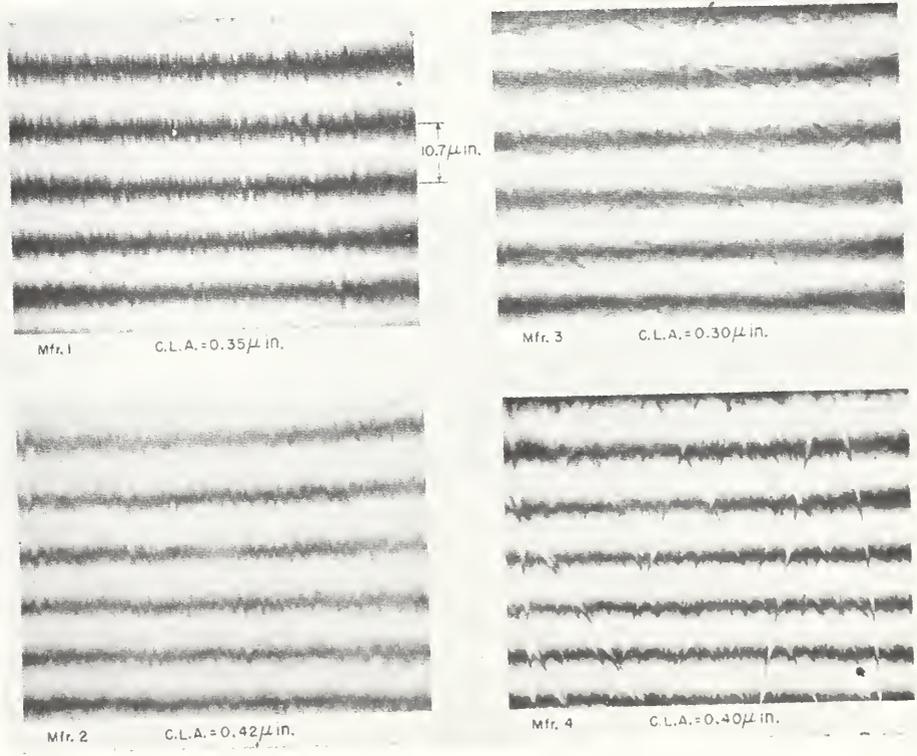


FIGURE 2. *Microfinish on grade A steel gage blocks.*

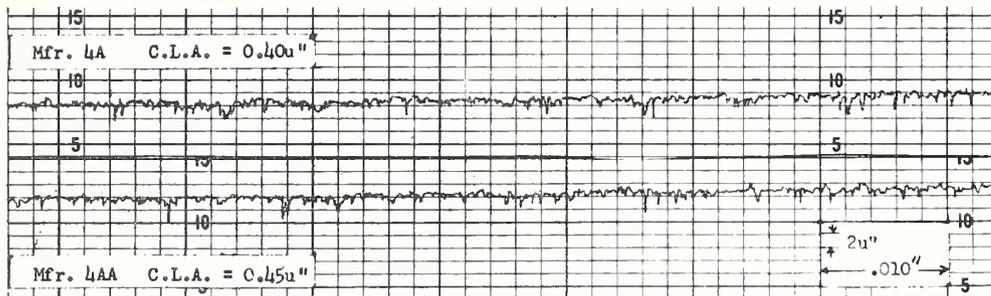
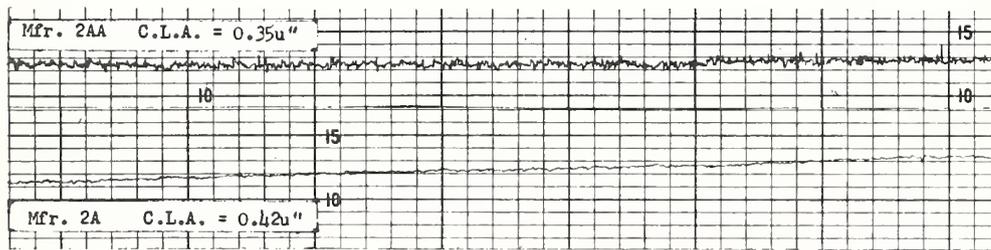
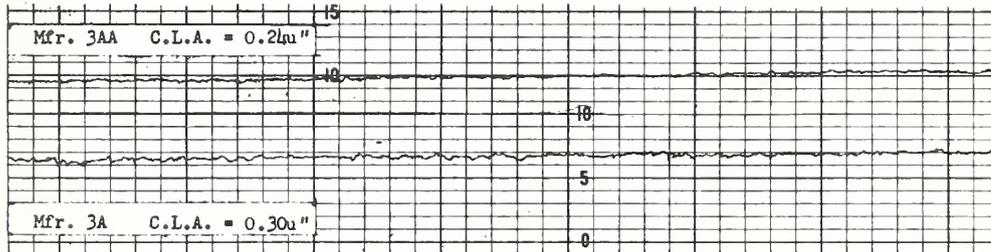
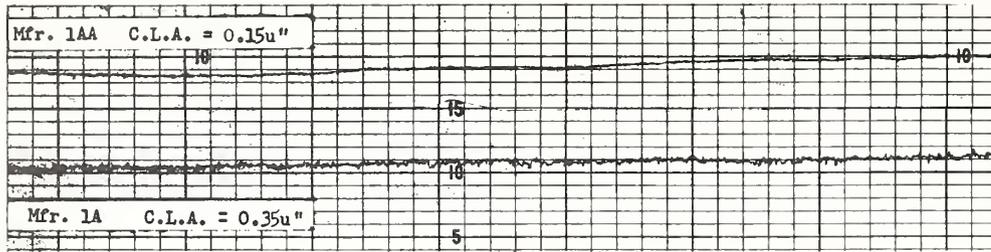


FIGURE 3. Graph of microfinish on grade A and AA steel gage blocks.

examples of apparently conflicting data. The charts do not indicate the narrow deep scratches but they show that there is a definite difference between the finishes. In chart 1AA of figure 3, the mirror finish shows a simple wavy surface with no scratches because the stylus indentation of $0.8 \mu\text{in.}$ is nearly equal to the depth of the scratches which average $1 \mu\text{in.}$ In chart 1A, the predominant depth is $2.5 \mu\text{in.}$ In chart 3AA, the predominant depth of scratch is $1.7 \mu\text{in.}$ and in 3A, $2 \mu\text{in.}$ For the AA gage block, produced by manufacturer 2, the C. L. A. value is less, the chart is rougher, and the predominant scratch depth is greater than for the A gage block. The scratch width is slightly wider on the AA gage block and, therefore, the tracer picked up greater depths and produced a rougher chart. The predominant depth of

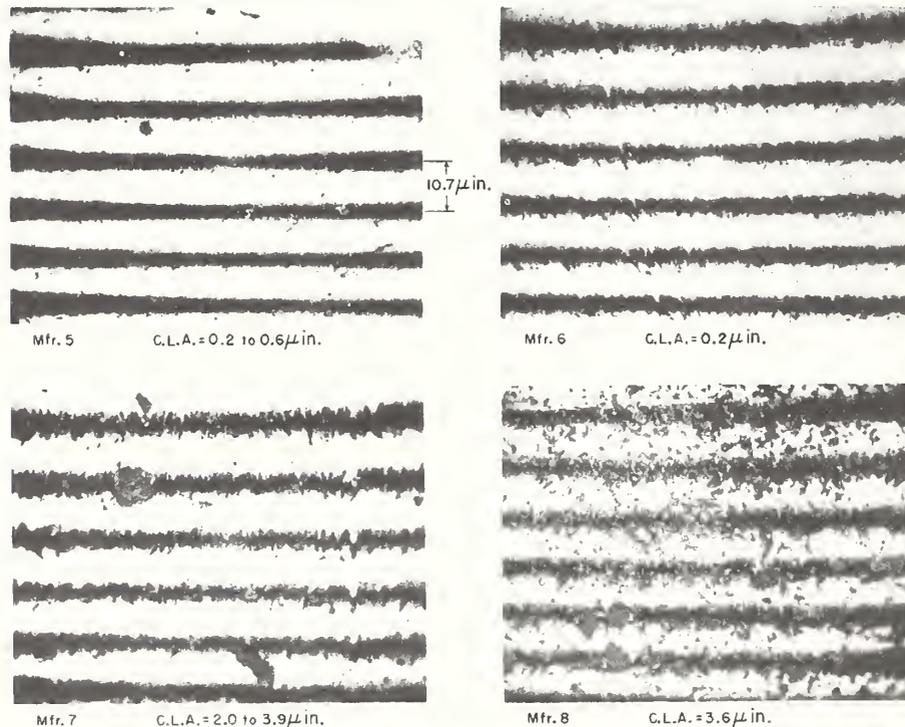


FIGURE 4. Microfinish on grade A and AA tungsten carbide gage blocks.

scratches on gage block 4AA is $4.0 \mu\text{in.}$ and only $1.5 \mu\text{in.}$ on the 4A gage block, but their centerline-average values only differ by $0.05 \mu\text{in.}$ Due to the random lapping, the tracer can often travel a short distance along the scratches and thus record a more accurate depth.

The microfinishes on tungsten carbide gage blocks are illustrated in figure 4. The scratch depth on all carbide gage blocks is very shallow (seldom over $2 \mu\text{in.}$) and there is very little fuzz. This accounts for their good wringing characteristics. The major problem is defective carbide having excessive voids. The large void in the block of manufacturer 7 is about 0.0007 in. in diameter.

The corresponding charts in figure 5 indicate the size (both width and depth) of the voids. On the gage block of manufacturer 7 there is a hole $.0008 \text{ in.}$ in diameter and over $27 \mu\text{in.}$ deep. Surface imperfections give large and varied readings of C. L. A. which do not repre-

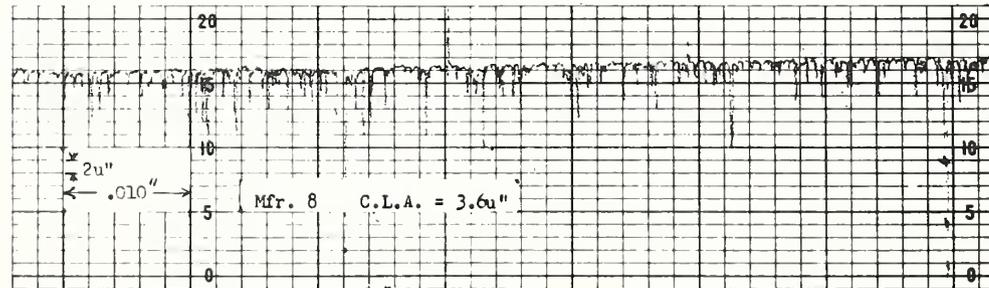
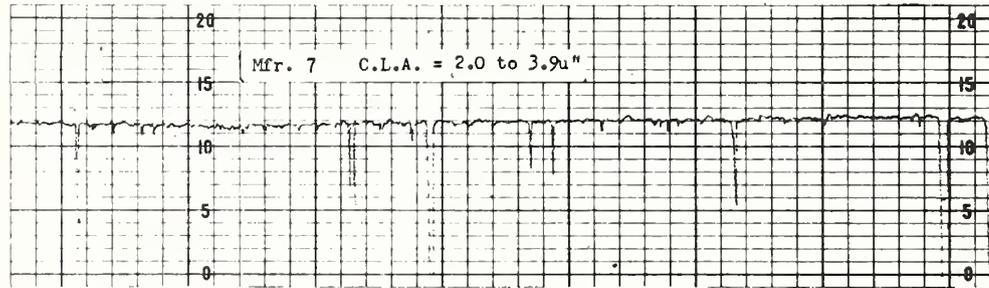
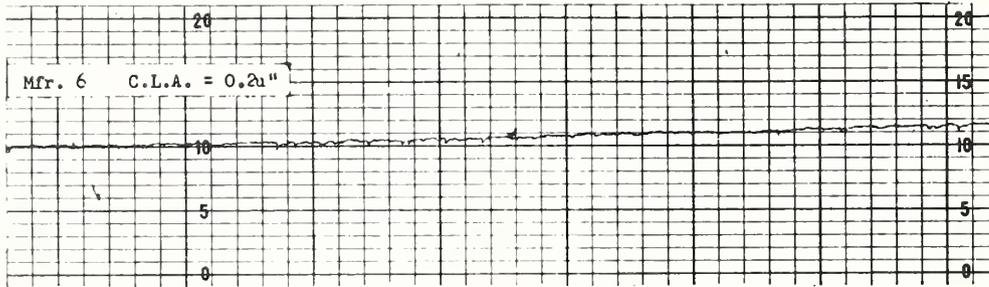
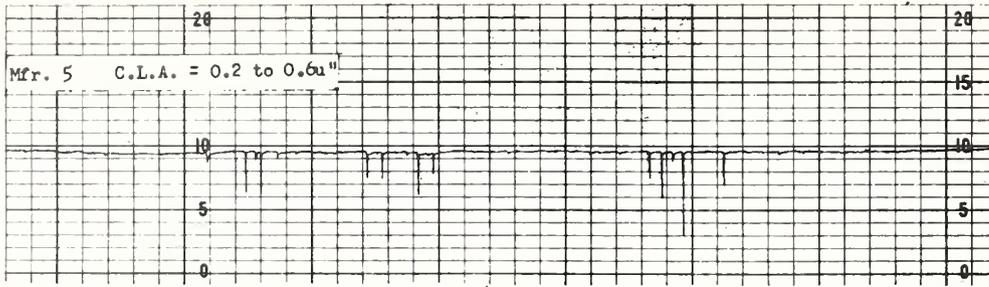


FIGURE 5. Graph of microfinish on grade A and AA tungsten carbide gage blocks.

sent the real surface roughness. Care should be exercised in using porous carbide gage blocks for accurate gaging with a mechanical indicator as it is possible for a contact point having a small radius to partially penetrate such holes.

Surface finishes on chromium-plated gage blocks are shown in figure 6. Chromium-plated gage blocks are popular for their wear characteristics and corrosion resistance but unpopular where good wringing is an important factor. The gage block of manufacturer 10 shows why some chrome-plated gage blocks fail to wring well. The wringing surface is a series of hills and valleys of irregular heights. When the surface roughness is reduced as shown in the interference

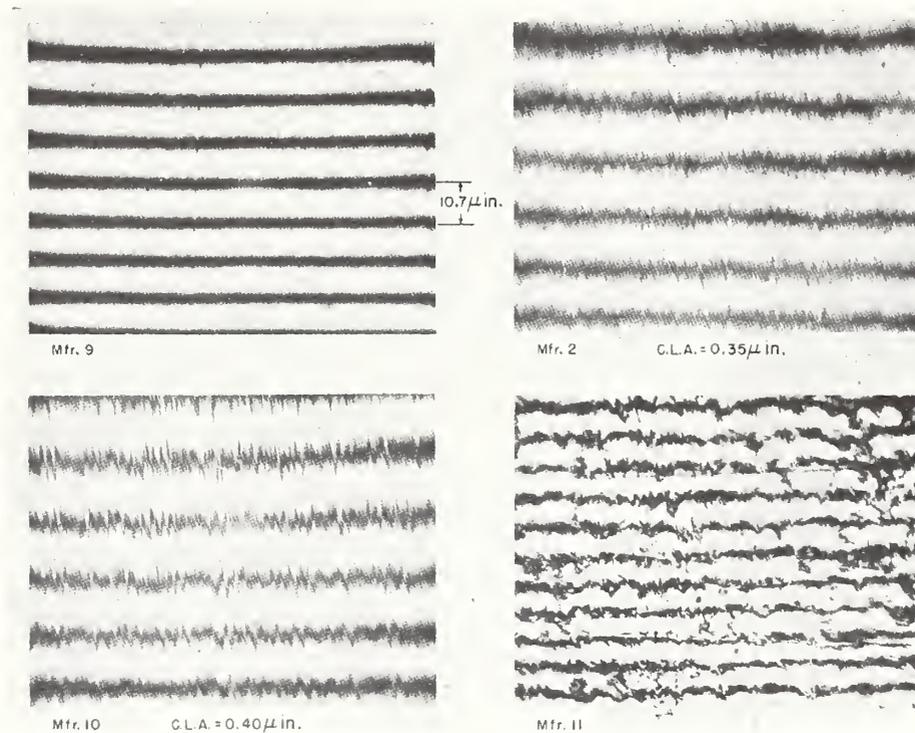


FIGURE 6. Microfinish on grade A and AA chromium-plated gage blocks.

micrographs of the gage blocks of manufacturers 9 and 2, and the surface is plane, these gage blocks wring very well.

Objectionable gaging surfaces are illustrated in figures 7 and 8. The steel surfaces of the gage blocks of manufacturers 12 and 13 are not satisfactory. The first surface has excessive pits for steel, and the second shows oxidation caused by excessive surface temperature during the final lapping operation. A micrograph of a fresh steel surface has a bright background with clean, well-defined dark lines or abrasive scratches. Figure 7 (manufacturer 14) shows a chromium carbide AA gage block with a large pit (size 5.4 by 2.5 mils) in the surface. The hole is 38 μ in. deep. Smaller pits (1 mil in diameter) are often found. Figure 7 (manufacturer 8) shows a very porous surface such as is found on some tungsten carbide gage blocks. Figure 8 (manufacturer 2) illustrates a crazed chrome-plated surface probably caused by high surface stresses. Figure 8 (manufacturer 11) illustrates

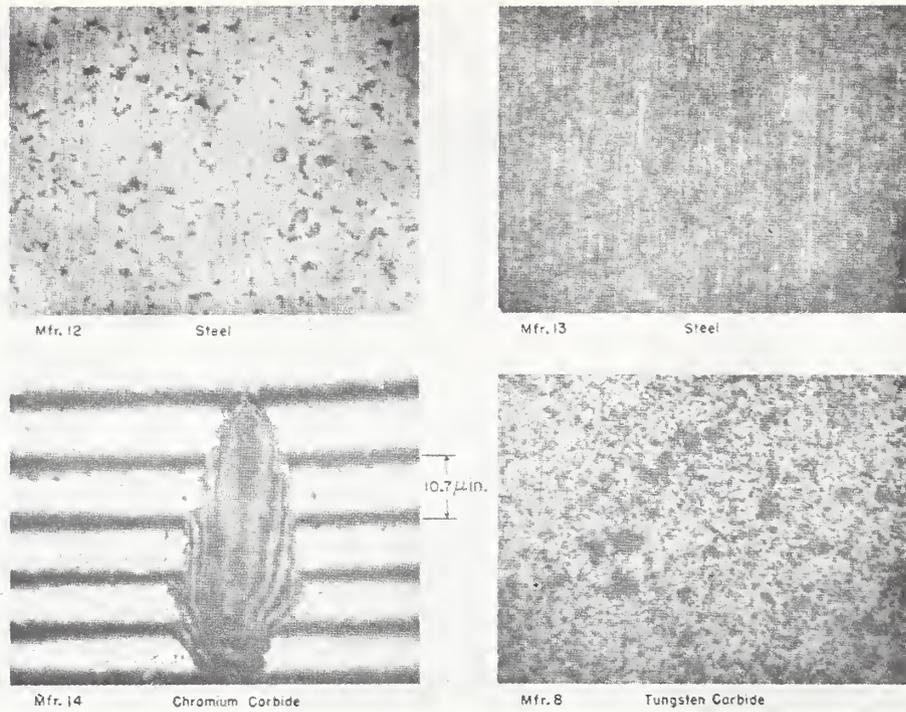


FIGURE 7. *Surface characteristics on gage blocks.*

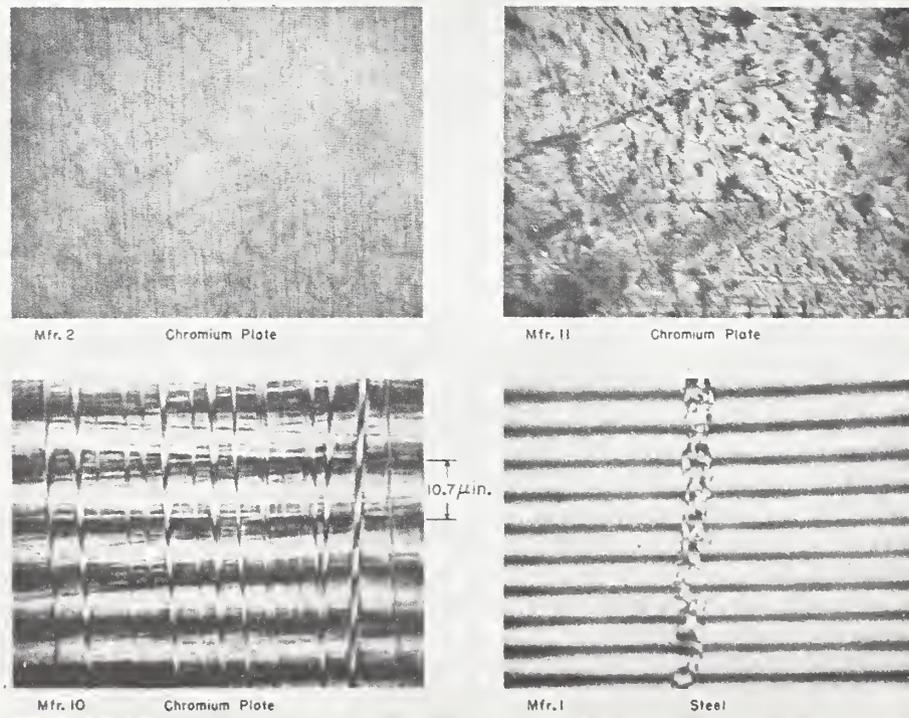


FIGURE 8. *Surface characteristics on gage blocks.*

a poorly bonded chrome-plate. Figure 8 (manufacturer 10) indicates that fairly large abrasive particles were lodged in this rough chromium surface because the tracer shoes of a surface indicator dislodged them and caused the large scratches. The striated fringe pattern is caused by the rough surface finish which has a predominant scratch depth of $6 \mu\text{in.}$ The blemish on the mirror finished steel gage block shown in figure 8 (manufacturer 1) was produced when the skid of a surface analyzer traversed the surface. The torn surface is $7 \mu\text{in.}$ deep in places. Similar damage was not produced on rougher finished gage blocks.

The ease with which many mirror finished surfaces can be scratched leads us to believe that the hardness of a very thin layer, perhaps

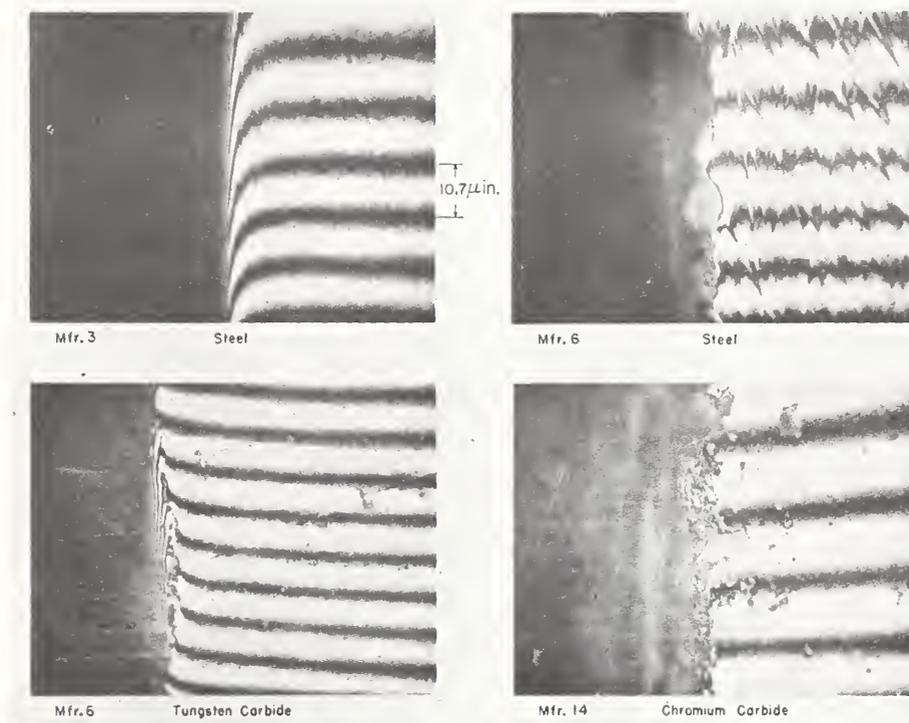


FIGURE 9. *Edge conditions on grade AA gage blocks.*

3 to $7 \mu\text{in.}$ deep, is appreciably less than in the rest of the gage block. We think that some annealing of a steel surface with a decrease in hardness may occur during a lapping operation as the surface approaches a mirror finish.

Figure 9 shows the variety of edge conditions found on AA gage blocks. The gage block of manufacturer 3 illustrates the ideal contour of an edge. This type of edge will not scratch another gage block when blocks are wrung together. Grinding a beveled or radius edge will not produce this smooth edge but stoning will. The edge on the tungsten carbide gage block has a burr raised $11 \mu\text{in.}$ above the gaging surface; thus the surface will not wring to another gage block surface. This type of edge has been found on other manufacturers' steel and carbide gage blocks. The chromium carbide gage block has a fractured and crumbly edge.

It is believed that ragged edges are the greatest source of potential damage to gage block surfaces. Sometimes small pieces of metal break off the edges and get between the wringing surfaces. Many cases were found where scratches on gage blocks start at a ragged portion of an edge. Furthermore, it is extremely easy to produce a

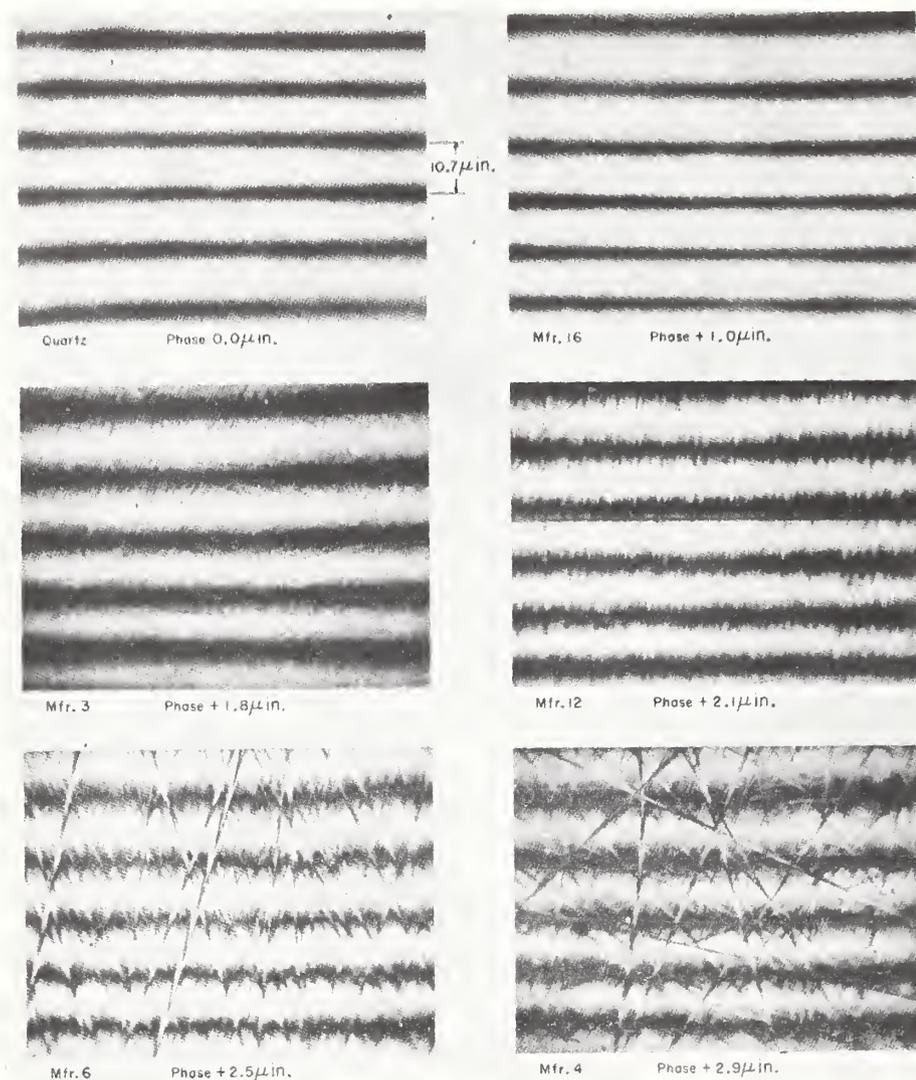


FIGURE 10. Comparison of surface roughness and phase change correction on quartz and steel gage blocks.

burr on an edge of this type. It is hardly more than necessary to touch such an edge against a hard surface to produce a burr. The burr may be only a few microinches high but it generally cannot be entirely removed by stoning and usually adversely affects the wringing quality of the gage blocks.

Previous speakers have discussed phase change correction. This correction is necessary because light appears to penetrate a metallic surface to a slight extent before it is reflected. The effect is partly due to the theoretical electromagnetic phase change which occurs

when light is reflected from a material which is a conductor of electricity and partly due to the effect of surface roughness. The phase change at reflection from a nonconductor such as glass or quartz conventionally is considered to be zero. The phase correction for a highly polished steel surface is $+0.8 \mu\text{in.}$ When a surface is not perfectly smooth, a further correction is necessary to compensate for the penetration of the light into the irregularities. This correction increases in proportion to the surface roughness and varies from less than 0.5 to over $2.0 \mu\text{in.}$ The true phase correction ($+0.8 \mu\text{in.}$ for steel) and the correction for surface roughness are usually summed together as the phase correction. The interference micrographs in figure 10 compare the surface roughness on gage blocks with total measured phase change correction. The table below summarizes the data.

Mfr. number	Composition	Measured phase change correction	Predominant scratch depth	Centerline—average height
--	Quartz.....	$\mu\text{in.}$ 0.0	$\mu\text{in.}$ <1.0	$\mu\text{in.}$ 0.05
16	Steel.....	+1.0	<1.0	.07
3	+1.8	1.7	.24
12	+2.1	2.2	.28
6	+2.5	3.0	.35
4	+2.9	4.0	.45

It is interesting to note that the sum of one half of the predominant scratch depth on steel and the theoretical phase change correction for steel ($+0.8 \mu\text{in.}$) are in close agreement with the measured phase change correction. In most sets of gage blocks, it is possible to find a $1 \mu\text{in.}$ spread in phase change correction, therefore, it is essential that the manufacturer produce a very uniform surface finish on all AA gage blocks.

12. Questions and Suggestions With Regard to Gage Blocks

By Edvard Johansson¹

The papers read yesterday and today have dealt with the scientific measurement of gage blocks. I feel that something should be said regarding the standardization of gage blocks from the standpoint of the manufacturers. I shall therefore deal more with standardization and questions of interest in the mutual work on gage blocks of the national laboratories and the manufacturers of precision gages. Thanks to very close mutual work with leading scientists of different national standards laboratories, we have today an almost perfect length standard in the gage block.

Many new ideas and suggestions regarding improvement of gage blocks come almost every day. New materials, new shapes, and new types of accessories are a few of these. We can find, in addition to the ordinary rectangular type, cylindrical, square (with and without center hole), heavy duty, and even triangular-shaped gage blocks. No wonder then, that the scientists responsible for the measurement of these gage blocks would like to standardize only a few conveniently-shaped types of gage blocks.

The first combination set of gage blocks was used at the Royal Rifle Factory in the town of Eskilstuna, Sweden. This set is now in the possession of the C. E. Johansson Co. in the same town and these are real heavy-duty blocks. Such gage blocks are therefore not a recent development. They were soon found to be too heavy and were superseded by a lighter type, the conventional rectangular type gage block. Later, during the first world war, the round type was developed here at the National Bureau of Standards. Experience in the use of gage blocks during the past 60 years has shown that there is no real need for gage blocks having shapes other than the conventional rectangular and square types.

The mutual work of the national laboratories in creating and adopting a common international standard for gage blocks will be of great value to both manufacturer and user. In this work the main question is the standardization of the tolerances for the length, the flatness, and the parallelism of the gage blocks. The tolerances must be specified so that they include the accuracy to which the national laboratories are able to determine the length in question. Regardless of the accuracy claimed by any gage-block manufacturer for his product, the length of a gage block cannot be known more accurately than the uncertainty in the measurement specified by the laboratory that calibrates the block.

The first tolerances system for gage blocks was made symmetrical and progressive to assure that a combination of gage blocks would

¹ C. E. Johansson Gage Co., Dearborn, Mich.

not exceed the tolerance limits for a solid gage of the same nominal size. Later on, a nonsymmetrical tolerance system was used to some extent. This was to permit greater wear and extend the life of the gage blocks. The British standard, by way of example, stipulates for the inspection grade a tolerance of $+7$ and -3 μ in. The tolerance for the work shop grade is $+10$ and -5 μ in. for gage blocks up to 1 in. in length. If a 1-in. combination is made up with two gage blocks from the inspection set and these two gage blocks are made with the allowable wear tolerance $+7$ μ in., we find that the length of this combination is outside the allowable maximum tolerance limit of $+10$ μ in. for a single 1-in. gage block of the work shop grade. Thus the inspection grade has actually become less accurate than the work shop grade. The application of such a nonsymmetrical tolerance system to gage blocks causes confusion as to the grade of accuracy. When a plus tolerance from 2 to 3 times the minus tolerance is specified, most of the gage blocks in a set will be oversize. With respect to the calibration and reference grades having symmetrical tolerances, the plus tolerance blocks will, in effect, represent a new and larger measuring unit. Nothing is more confusing than these nonsymmetrical tolerances, especially in consideration of the work on the unification of the two existing inch systems, the American and the British inch.

More than 25 years ago the first step was taken to unite the two existing inch systems into one international inch system. The basic length of this system is almost exactly midway between the American and the British inch systems. For industrial purposes this international inch was adopted in 1933 by the American Standards Association and some years later was also adopted in England. The international inch has not yet been accepted legally in either country, and the progress of the work toward unification of the two very closely related inch systems seems to be very slow, even after 25 years of consideration.

I take this opportunity to express myself in the interest of my company and many other manufacturers of precision gages, and request that the National Bureau of Standards and the National Physical Laboratory of England do everything possible to expedite the adoption of a common definition of the inch. A common inch system would certainly be unanimously recognized in other countries.

In the definition of the length of a gage block a reference point on the measuring surface is specified. This reference point is differently defined in different specifications. Users of gage blocks as well as manufacturers usually specify the length at the center of the measuring surface on rectangular gages. The certified deviations from nominal length in a certificate of calibration should, therefore, refer to the center of the measuring surface of rectangular gage blocks and, on square gages, to a point midway between the edge of the center hole and the edge of the side bearing the nominal size marking. It would be less confusing for users and manufacturers of gage blocks if the center and midway points on the measuring surfaces were accepted as standard reference points for the length.

With regard to the material used in the manufacture of gage blocks, I shall discuss only chromium plate. It is essential that a very hard type of the chromium plating be applied, and that it be deposited on the hardened steel in such a manner as to insure against chipping and peeling in normal use. Chipping and peeling may occur if the chromium plate is too thin although otherwise meeting

all requirements. This may happen when the plunger of a measuring device is brought in contact with the chromium plated surface in a conventional manner. The tip of the plunger penetrates the surface of the gage block to some extent, the amount depending partly on the elasticity of the material of which the gage block is made. When this occurs brittle chrome may crack. If the deposit is too thin, sliding of the gage block under the plunger may tear the chrome film. A minimum thickness of the chromium deposit is therefore essential to support the plunger without cracking the chromium film. An investigation of this matter would be of interest and a recommendation should be included in specifications with regard to the minimum thickness of the chromium deposit. It would be of interest to know if any one here has had any experience with regard to a required minimum thickness of the plating.

A specification for gage blocks should include a requirement that all reports of the calibration of gage blocks specifically state the accuracy of the reported values. Certificates list the deviation from the nominal length of a gage block in microinches, but the accuracy to which the calibration was made is usually not stated. If, by the way of example, a 1-in. gage block of "A" grade is certified to be 1 in. plus 4 μ in. and the accuracy of the calibration is plus or minus 3 μ in., the 1 in. gage block may be any value between 7 and 1 μ in. longer than the nominal length. In the former case the error in the length of the gage block is outside the permissible limit for a grade "A" block. From the manufacturer's standpoint it is necessary that a certificate issued by a national laboratory state the accuracy of the calibration.

When gage blocks are measured by interferometric methods, either absolutely or by comparison, the calibration certificates should list the correction factors that have been used for parallax and for the change of phase. The latter, to a large extent, depends upon the surface finish of the two surfaces involved. A manufacturer of gage blocks who uses interferometric methods needs to know the correction factors that have been applied.

Regarding the squareness of the measuring surfaces to the sides, it is desirable that a common international standard be adopted. The German requirements in DIN 861 are acceptable whereas the British requirements in British Standard 888; 1950 are unnecessarily restrictive.

Considering the very close tolerances that prevail nowadays, it is essential that the manufacturers of gage blocks work very closely with the national laboratories, especially with regard to the performance of gage blocks, and this meeting, at the invitation of the National Bureau of Standards, is a step in the right direction. I hope that such meetings can be held more frequently in the future in order to create a better and closer understanding between the manufacturers and the users of gage blocks on one hand and the national laboratories on the other.

13. Gage-Block Surveillance

By M. S. Hoskins ¹

In the calibration and surveillance of over 1,000 sets of gage blocks used by 23 military organizations and many manufacturing contractors over the past several years, it is apparent that there are definite needs for the clarification of certain misconceptions and for constructive suggestions for improving the surveillance, care, and proper use of gage blocks in the laboratory and shop. At Sheffield we need and demand the best in gage-block surveillance for the two hundred and some sets in use throughout our own plants and for the sets that we calibrate for our wide variety of industrial customers. With the increasing demands for higher precision in industrial dimensional standards, and with continually improved methods of calibrating gage blocks, the proper handling of blocks in use must be controlled more thoroughly; first, to insure the accuracy needed for certifying the smaller tolerances, and second, to insure the practicality of recalibration, which, of course, becomes exceedingly more difficult as the blocks are more used and abused.

My analysis is intended to be constructive and not a reflection upon any user of gage blocks, and all of my remarks pertain to the used block. I will say at the start that there has been considerably better thinking in the last few years, especially in the realm of better gage-block inspection procedures, and there is a decided trend to what I like to call "laboratory affection" for this precision instrument known as the gage block. However, I feel that there is an area for greater precision appreciation, and so I should like to point out more specifically some misconceptions and poor usage practices that sometimes occur.

One of the longest standing beliefs, and the one that causes probably more inaccuracies in measurement than any other, is the belief that once a set of blocks is purchased, it is perfect and will remain perfect forever. This, of course, is not true, even if the set has never been used, because of possible dimensional instability. Before joining the Sheffield organization, I checked badly worn sets that had been in use for as long as 15 years with no calibration whatsoever. However, the user of one of these sets would not believe that his set was far out of specifications until he was actually shown. His comment of "Oh, but those are precision gage blocks" was answered and corrected by mine, which was simply, "No, they *were* precision gage blocks." A rather unique—or at least I hope it is unique—condition occurred in one of the old uncalibrated sets. Evidently, three of the blocks from this set had been lost, and substituted in their places were three pieces of steel stock carefully etched with nominal size and more or less lapped. A rough mechanical measurement of size found them to be

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about 0.003 in. undersize and quite out of parallel. Despite my being convinced that they were merely to hide the empty spaces, I found that these pieces were actually being used with the other blocks in the set. These old uncalibrated sets and homemade blocks are fortunately in a minority. However, many, many sets have been inspected that were worn far beyond all practical use for which they were intended. This was mainly the result of neglecting to have the sets recalibrated periodically under a schedule based on amount of use of the blocks and the accuracy required to do the job.

Even after calibration and recalibration requirements are understood, it is occasionally difficult to make some inspectors and others understand that the calibration report is to be used. The smaller the tolerance, the more important the report of size. One extreme case of such neglect was that of the probably well-intentioned but inexperienced foreman of a group of final inspectors who, after he received current calibration reports for all of the sets in his department, carefully put the reports in a nice, clean folder and locked them up in his desk drawer so they wouldn't get dirty. The locked-up report is, of course, more unusual than the occasional inspector who only consults the calibration report when he is expressly warned of the errors caused by inaccuracies of his block combinations. The calibration report should be kept in plain sight in the work area.

Another misconception occurs often enough to be mentioned, that is, that "gage blocks is gage blocks." Occasionally, the use of identical buildups from several sets cannot be avoided; then it is important to avoid putting the blocks back into the wrong cases. Inspection discipline should prohibit this negligence, and having all of the blocks marked with individual identification or serial numbers, helps to avoid such confusion.

After a department recognizes the merit in having its gage blocks periodically calibrated, and even when the inspector intelligently uses his calibration report, problems still remain. Gage blocks may be used as parallels or shims in fancy set-ups, or the blocks may be clamped down to a surface plate or angle plate with C-clamps to make sure they stay put. These special uses were explained to me when I started to investigate why some blocks had objectionable terraces and circular scratches on them. Most of the other markings, nicks, scratches, and burrs—of which there were an amazing number—naturally came from dropping, wiping the blocks on dirty rags and paper, laying the gaging surfaces down on a gritty, dirty workbench, and from using the blocks on freshly lapped material without first carefully cleaning it. Rust and acid stains came from finger prints being allowed to remain on the blocks and from eating salted peanuts or fruit while working with the blocks. One other easily recognizable marring of the block comes from using the gage blocks to set snap gages without using wear blocks. The aforementioned damages are not necessary and should be thoroughly eliminated from the work methods and procedures.

A final point to be made is that the proper wringing of two or more gage blocks together is an art which must not be neglected. The gage blocks should be wrung together only when they are clean and no single block has burrs of *any* amount that will scratch or mar the surface of any other block. Then the blocks are to be wrung, not forced or pushed together. Briefly, an initial light, circular motion that will begin the wringing if the blocks are clean, and then adding

very slight pressure into full engagement, is the correct way to wring blocks.

Referring to my previous statement about improvements in thinking, people are definitely bringing up to date their ideas as to the accuracy and importance of gage blocks. They have begun to ask more questions about what should be done and how to go about it. They have become more insistent that gage blocks be checked and have been quick to bring in individual blocks for certification when questions of dimensional accuracy have arisen with a contractor or government inspector. One man went a little too far in the improvement angle, I think—maybe not—maybe our inspection methods will go that far soon—but I had one request to explain just how we went about measuring the depth of germs on the gage blocks, as it was evident to this man that there were germs present. Through another man's misuse of the word 'contaminated,' he had been led to believe that this was done. At least he was aware that the gage blocks were subjected to high precision measurement. A check revealed that he was not trying to be humorous.

A program of calibration must provide different techniques of measurement for different qualities of blocks. A set of the highest accuracy possible, for use of the standards laboratory, would be designated a reference or referee set. The ideal situation would be to have no block in this set with a flatness or parallelism error greater than 0.000001 in. and with its size within 0.000002 or 0.000003 in. of nominal. The next quality would be a master set for the standards laboratory, whose accuracy is within 0.000002 in. for flatness and parallelism and whose size is within 0.000005 in. of nominal. The technique for measuring or calibrating these sets would be by the absolute method. The absolute method, of course, yields the highest accuracy in length measurement and is done by comparing the blocks with several wave lengths of light used as length standards and with corrections made for any difference from the norm of temperature, barometric pressure, vapor pressure, and acceleration of gravity. In our new Eli Whitney Metrology Laboratory at Sheffield we have the Kösters absolute interferometer and the Zeiss Opton interferometer to accomplish this type of measurement, and these facilities are continuously made available by Sheffield to private industries along with its other precision measuring equipment. For application of this method, it is essential that the flatness and parallelism errors of the block to be tested be no more than 0.000003 in., otherwise it would be a waste of time and money to try to measure blocks by this method. The larger the flatness and parallelism error, the greater the observational error, and soon the observational error may exceed the specified tolerance.

The best accuracy for the next group, which could be called a standard set for the gage laboratory, would be a limit of 0.000005 in. for flatness and parallelism errors and no more than 0.000007 in. deviation from nominal size. In all of these categories the deviation from nominal size is of less concern than flatness and parallelism errors. Size may deviate from nominal and not really affect the quality of the block at all. Following the standard set would be the working set for the inspection room for use on parts. This working set accuracy should be within 0.000010 in. for flatness, parallelism, and length. Both the standard and working sets could be measured by a careful and trained inspector applying the comparison method with a high magnification external comparator.

Allowing for important exceptions that may exist in any one plant, any blocks over the above tolerances or specifications, and not exceeding 0.000020 in. in flatness and parallelism errors, when provided with a deviation chart could be relegated to the shop as a shop set.

As to the handling of gage blocks, it should be the responsibility of every laboratory, inspection room, and shop foreman to educate every person using gage blocks in proper cleanliness habits, handling procedures, and wringing methods. Gage blocks are highly precise instruments and should be handled as such with no margin for error.

With regard to a recalibration schedule, a good rule of the thumb would possibly be:

Reference sets—recalibration every 6 months.

Master sets—every 3 months.

Standard or inspection sets—every 2 to 3 months.

Shop sets—every 9 months to 1 year—with the exception of those shop sets used in abrasive areas, for such sets would probably need very frequent reinspection.

A facility either within the home organization or on the outside, set up with the proper equipment and trained personnel to perform these difficult types of calibration should be provided, and a calibration schedule should be set up and followed.

I have pointed out many malpractices and have suggested a few methods to overcome the problems. Let me then, briefly, sum it all up. Gage blocks must be calibrated, and by a method indicated by their accuracy requirements. The accuracy requirements must be set up according to the use of the blocks and the amounts of the tolerances of the work. The calibration report must be used, and the report reevaluated as often as surveillance shows it to be necessary—a surveillance that checks use and abuse, wear and damage, and is cognizant of the tolerances being checked by these blocks.

Gages and measuring equipment for the dimensional control of modern mass production require constant surveillance to insure their continuous accuracy. Gage blocks occupy the keystone position in the precision structure that assures the dimensional conformity and interchangeability of parts that flow into the mass assembly of items that make our national economic abundance and the weapons of our national defense. Just as we accomplish the surveillance of the accuracy of our watches by periodic checks against radio time signals or authoritative clocks, so also does each gage or item of measuring equipment require periodic checking against accurately known standards. In the American production scene, gage blocks are found almost universally in use as the authoritative dimensional standards, from the tool room to the high level standards laboratories. Gage blocks are the vital surveillance tools in all echelons of precision dimensional control, and they must be subjected to rigid surveillance themselves to assure the effectiveness of all tools for precision dimensional inspection.

14. Concerning the Revision of the German Standard DIN 861, Parallel End Standards (Gage Blocks)

By E. Engelhard¹

A new version of the German standard for gage blocks and their accessories became effective in Germany some months ago.

Specifications for gage blocks in Germany were first established in 1927. Since that time gage blocks have come much more into use, with the requirements today for accuracy on the one hand and for precision in manufacturing gage blocks on the other hand, being remarkably higher. Thus the former German specifications have not fully agreed with the actual situation for many years. About 1930 when the ISA system of tolerances for fits, with not less than 16 grades of fits having a minimum tolerance of 0.5μ (0.00002 in.), was introduced in German industry, it became apparent that new finer specifications for gage blocks would soon be necessary. Objections to the introduction of the new system of fits made by gage manufacturers at that time could only be answered by promising new specifications for gage blocks with finer grades. The specification was prepared in 1931 but unfortunately was not available before this year due to difficulties during and after the war. As a new specification for gage blocks is also being prepared in this country, perhaps it may be timely to look at the specification now used in Germany and especially at some of the considerations on which it is based.

In the new version of the German standard, "gage blocks" DIN 861, three points are dealt with: (1) definitions, (2) shape of gage blocks, and (3) tolerances.

Concerning the first item the following statements are made in DIN 861: (1) The length at the center m (fig. 1), which requires no explanation, is defined as the perpendicular distance of the center of the gage block surface A from the plane surface of an auxiliary plate B made from the same material as the gage and wrung to the other gage block surface A', the surface of the auxiliary plate B being of the same quality as the surface A. This version agrees fully with the definition of the length of a gage block that is accepted in all countries in accordance with an ISO recommendation. Note that this definition involves the thickness of a wringing film. This is important, in order to avoid complications when gage blocks are wrung together. (2) The error in the length at the center f_m is consequently defined by the difference between the length at the center and the nominal length, $f_m = m - l$. (3) The third definition of the standard concerns the length at any point of the surface of the gage block indicated by b (fig. 2). The length at any point of the gage block surface, in consequence of the definition of the length

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at the center m , is defined as the vertical distance of any point on the gage block surface A from the plane surface of an auxiliary plate wrung to the other surface of the gage block under the conditions given by the definition of the length at the center; for example, b_1 and b_2 are the values of length for the points 1 and 2, respectively, of gage-block surface A. (4) The deviation f_b at any point of the gage-block surface is consequently defined by the difference between

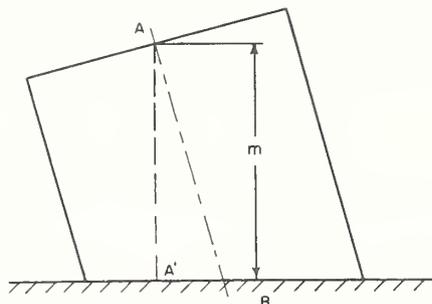
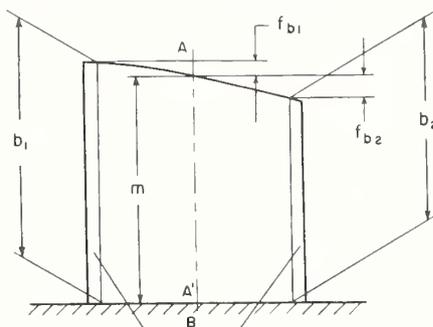


FIGURE 1.

the length at any point, b , and the length at the center, m , $f_b = b - m$. The geometrical conditions of the gage-block surface, including flatness and parallelism, are completely determined by the value of the deviation f_b at any point of the surface.

The preceding four definitions of length at the center, m , error of the center, f_m , length at any point of the surface, b , and deviation at any point on the surface from the length at the center, f_b , which are the most important dimensions of a gage block, were, in principle,



EDGE ZONE
FIGURE 2.

specified in the former version of DIN 861. (5) A definition of obliquity (fig. 3) has been added in the new standard.

According to the new DIN 861, obliquity of gage blocks is defined by s , s being the deviation in perpendicularity of the side faces relative to every measuring surface. Therefore, for every side face, two values, s_1 and s_2 , of the obliquity are taken into consideration, s_1 being the obliquity of one side face relative to the lower surface, s_2 the obliquity of the same side face relative to the upper measuring surface of the gage block. Altogether eight different values are possible for the four side faces of one gage block. Limitation of obliquity is necessary, otherwise combinations of gage blocks might not fit in the holders as specified by the standard. In order to determine obliquity it is recommended that the gage block in question be placed on a surface plate

with one side face set against a straightedge clamped to the plate surface. The location in a horizontal plane of this side face of the gage block should then be determined by a suitable method, for example, by a mechanical test indicator also fixed on the plate. The gage block is then replaced by a square and a reading of the test indicator made in the same manner, the difference of the two readings corresponding to s . (6) The next definition in DIN 861 concerns wringing. Wringing is undoubtedly one of the most essential features of a gage block, because it permits the formation of lengths in very fine increments. Therefore, in preparing a specification for gage blocks, it is recom-

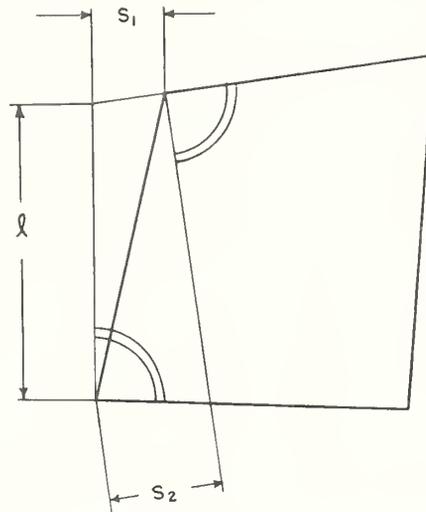


FIGURE 3.

mended that the requirements as to wringing be very exactly defined. DIN 861 explicitly states that the adhesion of surface must not be obtained by supplementary substances and that close, i. e., optical contact, as judged by means of an optical flat, must be obtained over the whole surface.

Part 1 of DIN 861 covers definitions. Part 2 covers the shape of gage blocks. First, it should be mentioned that only gage blocks made of steel, the coefficient of thermal expansion being $(11.5 \pm 1.5) \cdot 10^{-6}$ per deg C, and diamond pyramid hardness number not less than 800 kg/mm^2 are specified in the German gage-block standard. Furthermore, in this part of DIN 861, the nominal cross section for different lengths of gage blocks is stated. The cross section, in general, is rectangular, 9 mm in width and 20 and 30 mm in length for gage blocks up to 0.5 and 10 mm, respectively, and 9 mm in width and 35 mm in length for longer gage blocks. Gage blocks having other cross sections are allowed provided the area is the same as in the case of the rectangular section. However, gage blocks of other than rectangular section are quite unusual in Germany. A further stipulation of DIN 861 requires manufacturers to mark gage blocks with their trademark.

After defining or stating the nominal sizes for gage blocks in parts 1 and 2 of DIN 861, the essential tolerances are indicated in part 3 (fig. 4). This table represents the allowable values (1) for the errors in length at the center f_m , (2) for the deviation of length at any point of the surface f_b , and (3) for the obliquity s , all values expressed in

microns (0.00004 in.), the reference temperature being, as usual, 20° C (68° F).

Nominal Size mm	Grade of Accuracy 0			Grade of Accuracy I			Grade of Accuracy II			Grade of Accuracy III		
	f_m	f_b	s	f_m	f_b	s	f_m	f_b	s	f_m	f_b	s
0.1	—	—	—	0.2	0.15	—	0.5	0.25	—	1.0	0.5	—
0.5	0.1	0.1	50	0.2	0.15	60	0.5	0.25	75	1.0	0.5	90
10	0.12	0.1	50	0.25	0.15	60	0.6	0.25	75	1.2	0.5	90
20	0.14	0.1	50	0.3	0.15	60	0.7	0.25	80	1.4	0.5	95
30	0.16	0.1	50	0.35	0.15	65	0.8	0.25	80	1.6	0.5	100
40	0.18	0.1	50	0.4	0.15	65	0.9	0.25	85	1.8	0.5	100
50	0.20	0.1	50	0.45	0.15	65	1.0	0.25	85	2.0	0.5	100
60	0.22	0.1	55	0.5	0.15	65	1.1	0.25	85	2.2	0.5	110
70	0.24	0.1	55	0.55	0.15	70	1.2	0.30	90	2.4	0.6	110
80	0.26	0.1	60	0.6	0.2	70	1.3	0.30	90	2.6	0.6	110
90	0.28	0.1	60	0.65	0.2	70	1.4	0.30	95	2.8	0.6	110
100	0.3	0.1	60	0.7	0.2	70	1.5	0.30	95	3	0.6	120
150	0.4	0.1	65	0.95	0.2	80	2.0	0.30	110	4	0.6	130
200	0.5	0.1	70	1.2	0.2	85	2.5	0.35	120	5	0.7	140
300	0.7	0.15	80	1.7	0.25	95	3.5	0.40	140	7	0.7	170
400	0.9	0.15	90	2.2	0.25	110	4.5	0.45	160	9	0.8	190
500	1.1	0.15	100	2.7	0.3	120	5.5	0.45	180	11	0.9	200
600	1.3	0.2	110	3.2	0.3	130	6.5	0.5	200	13	1.0	250
700	1.5	0.2	120	3.7	0.35	140	7.5	0.6	200	15	1.1	250
800	1.7	0.2	130	4.2	0.35	160	8.5	0.6	250	17	1.1	300
900	1.9	0.2	140	4.7	0.4	170	9.5	0.7	250	19	1.2	300
1000	2	0.25	150	5	0.4	180	10	0.7	300	20	1.3	350
1500	3	0.3	190	7.5	0.5	250	15	0.9	400	30	1.7	450
2000	4	0.4	250	10	0.7	300	20	1.1	500	40	2	600
3000	6	0.5	350	15	0.9	400	30	1.6	700	60	3	850
4000	8	0.65	450	20	1.1	550	40	2	900	80	4	1100

FIGURE 4.

The table covers a range in length from 0.1 mm up to 4,000 mm and four grades of accuracy 0, I, II, and III, the grades I and II being nearly the same as in the previous version of DIN 861. Two grades, a higher one, namely 0, and a lower one, namely III, have been added in the new version. Using the designations usually specified in this country, grade 0, I, and II correspond rather closely to AA, to A, and to B precision. There is no equivalent of grade III in this country. Take, for example, the tolerances for a gage block 100 mm, or nearly 4 in., in length. The allowable tolerances for the length at the center are, according to DIN 861:

	0	I	II	III
DIN 861	$\pm 0.3 \mu$	$\pm 0.7 \mu$	$\pm 1.5 \mu$	$\pm 3 \mu$
	± 0.000012 in.	± 0.000028 in.	± 0.000060 in.	± 0.000120 in.

and according to American classification

American standard	AA	A	B
	± 0.000008 in.	± 0.000016 in.	± 0.000032 in.

The tolerances in length for every accuracy grade as stated by DIN 861; are nearly twice as large as for the preceding grade, the tolerances for grade I being twice as large as for grade 0, for grade II twice as large as for grade I, and so on. This classification of gage blocks in four grades, as stated in the German specifications, takes the usual organization of industrial measurement technique into

account. According to the common use of gage blocks in industry, the four accuracy grades 0, I, II, and III could be designated in the same row—as is done in the British standard—as reference grade, calibration grade, inspection grade, and workshop grade, the latter being provided for use in workshops, the inspection grade for controlling gages used in the workshops, the calibration grade for calibration of inspection equipment, and finally the reference grade for the purposes of central metrology laboratories in official institutions or in large plants. The new classification of gage blocks in four grades, instead of in only two as in the previous edition of DIN 861, satisfies the requirements of gage manufacturers for more accurate gage blocks that arose from the higher requirements of the ISA system of tolerances. The introduction of the accuracy grade 0 takes the general improvement in manufacturing gages into account. For many years the situation in Germany was such that gage blocks of high quality were, in general, underrated with regard to quality because they were classed with gage blocks of much lower quality, the highest accuracy grade having been grade I until a short time ago.

With regard to the deviations in length at any point of the surface of the gage block, a comparison with the American specification is impossible without further consideration, because in this country separate requirements are listed for parallelism and flatness, whereas the German standard specifies only one value for the surface errors, this being the deviation in the length at any point of the surface f_b .

One might think that this method of specifying surface errors is not very convenient because it makes a difference whether a surface deviation, for example, of $\pm 0.6 \mu$, as allowed for a 100-mm gage block in grade III, is due to lack of parallelism only, or whether it is due to lack of flatness only. In the first case, the surface could be of the highest quality with regard to planeness, whereas in the second case, the allowable deviation being due to lack of flatness only, would certainly result in a surface so distorted that it could not be wrung to another plane. The German standards committee discussed the question of whether it is preferable to state separately the tolerances for parallelism and flatness or to define only a single limit for surface errors. The final conclusion was that, in general, it is impossible to separate errors in parallelism and errors of flatness in practice, and therefore they should not be specified separately. Strictly speaking, parallelism is connected to the geometrical concept of plane surfaces and if surfaces are not plane, parallelism has no significance. Consider a gage-block surface with an error in flatness. A correct statement of the error in parallelism perhaps could be made in the following manner: A plane surface may be defined in such a manner that the summed squares of the deviations of each point of this surface from the real surface are a minimum. Parallelism consequently should be determined relative to this ideal plane. Naturally such a method is not practical. Therefore the German specification does not have separate requirements for parallelism and flatness but only the values of f_b , i. e., the values for the permissible deviations in length at any point on the surface with errors in parallelism and of flatness both included. But this does not mean that the surface of a gage block may have errors in flatness of the full amount of the allowable value for the surface deviations f_b , for example $\pm 0.6 \mu$ for a 100-mm gage block as stated for the accuracy grade III, because it is explicitly stated in DIN 861, that the gage blocks have to satisfy the wringing require-

ment and this would be impossible if the error in flatness were larger than, let us say, a few tenths of a micron. Thus the decision that gage blocks must satisfy the conditions of wringing, in addition to the limitation on the values of f_b , represents, in reality, a sufficient restriction with regard to the surface errors, and has the advantage of being very easily recognized.

The situation is quite similar with regard to microflatness. The German specifications on gage blocks do not contain any exact information in this respect, in spite of the fact that it is more and more customary for block manufacturers to give information on the surface finish. DIN 861 requires only that the surface finish must be good enough to allow wringing of the gage blocks. There are two reasons for doing so: First, it was felt by the German standards committee that it was too early to specify definite requirements for surface finish, the situation of this field being, in general, not yet clear enough. Second, there was no unanimity of opinion with regard to the degree of surface finish that would be most suitable in practice. The common opinion seems to be that the highest degree of finish should be sought in every case. But objections are often made that surfaces with a very high finish, in practice, are too easily damaged. Indeed, before the war gage blocks of very high quality, but with lower surface finish, were manufactured in Germany explicitly for this reason. In general, the most practical method of specifying surface finish seems to be a requirement as to wringing quality.

With regard to the allowable values of the obliquity, there is little of importance to say. As may be seen from figure 4, the values of s , in general, are rather low, for instance the highest allowable value of the obliquity s for a gage block 100 mm, or about 4 in., in length, for accuracy grade III, is 120μ or nearly 0.1 mm. The highest possible value of $1,100 \mu$ or more than 1 mm is allowed for a 4,000-mm gage block in grade III.

In general, the new version of DIN 861 contains only specifications, decisions, or statements that are absolutely necessary, no more and no less. In preparing the new standard consideration was given to the fact that gage block manufacturers must inspect their product for compliance with every requirement of the standard. That is of interest not only to gage-block manufacturers but also to the official institutions authorized to inspect gage blocks such as the PTB (Physico-Technical Institute of the Federal Republic) in Germany. The official inspection of gage blocks at the PTB is based entirely on the requirements of DIN 861. The pertinent certificates not only deal with the determined values for length or the other specified sizes of gage blocks but also state explicitly that the gage blocks satisfy the requirements of one of the accuracy grades of DIN 861. Gage blocks that do not fulfill the requirements of the standard are not certified. Thus, by means of DIN 861, not only manufacturing but also official inspection of gage blocks is regulated in Germany, this standard representing a generally acknowledged and commonly used criterion in judging the quality of gage blocks. Naturally no one in Germany is of the opinion that the present specification for gage blocks is perfect, but it is hoped that the present version will satisfy all essential requirements for 10 or 20 years, in spite of the fact that desires for still higher accuracy and specific requirements concerning the surface finish have already been expressed.

15. Proposed Federal Specification for Gage Blocks, Attachments, and Accessories

By I. H. Fullmer¹

[The draft specification, which is now available as Federal Specification GGG-G-15 from the Business Service Center, General Services Administration, had been previously distributed to those in attendance. The following is a condensed record of Mr. Fullmer's comments on the specification.]

This is a purchase specification for the Federal government. To give you a little of the background and history of it: We have for a long time felt that there ought to be a Federal specification for gage blocks, but as you are well aware, there are so many controversial features to such a specification that it never was undertaken until the Navy Bureau of Ships was given responsibility for the purchase of items of this kind for the entire Department of Defense. A large part of the work that is represented in this document was done by W. V. Hurley of the Bureau of Ships, who is here in the audience. We commented at length on the first draft, which was submitted to NBS, and since then we have been working closely together on formulating the specification.

The scope is that the specification is applicable to precision gage blocks in either the English or metric systems for uses as follows: (1) Laboratory standards for calibrating other gage blocks, (2) laboratory standards for the inspection of other types of gages, (3) inspection gages in tool shop projects, and (4) work gages for laying out and setting up work with high accuracy for machining operations.

The classification covers three shapes: Rectangular, square with center hole, and square without center hole. There are the usual recognized grades of accuracy: AA, A, and B.

Materials specified are steel, chrome-plated steel, and carbides—chromium carbide, tungsten carbide, and other carbides.

In tables I and II are given the sizes of blocks that are generally recognized as standard in the industry. There is an advantage in stipulating these, as the listing will prevent to some extent the specification of odd-size blocks, which present a special problem in measurement. Then is listed a series of sets of blocks, that is, the sizes that go into any given set. There are seven generally recognized sets of blocks stipulated, based on a survey of government requirements made by Mr. Hurley.

You have heard the discussion of the German standard for blocks and you were furnished translations of the German standard. There is also available a British standard for gage blocks, the latest revision being in 1950.

Referring to paragraph 3.4.3 and the requirement that the case shall be marked with the manufacturers' name or trademark, the

¹ National Bureau of Standards, Washington.

British have this additional requirement: "If the sizes of the gages, as engraved upon them, are not visible when the gages are in their respective compartments, each size shall be marked in the case, immediately adjacent to the appropriate compartment". That is really quite general practice, but it might be well to stipulate it in our specification.

Next, paragraph 3.5.3.1, Surface defects: "Wringing and gaging surfaces shall be free from all burrs, however slight, sharp corners, and other defects which may affect accuracy or serviceability. The edges of wringing surfaces shall be smoothly rounded (not sharp nor beveled) and free from ragged grind marks. The porosity of carbide gage blocks shall be such that no pit on a wringing surface shall exceed 0.001 inch in diameter, width, or length." The British specification definitely specifies that a rounding of the edge shall be equivalent to a radius of 0.015 in. Whether or not it is desirable to stipulate a definite radius for that rounding, I am not certain. It seems to me it would be a rather difficult specification to enforce.

In our next paragraph, 3.5.3.2, Parallelism, flatness, and surface finish: "Gage blocks, attachments, and accessories shall have their wringing surfaces flat, parallel with each other, and smooth within the tolerances specified in table III. For sizes of blocks less than 0.100 inch the tolerances for flatness and parallelism are applicable when the block is wrung to an optical flat. Wringing surfaces of grade AA gage blocks, which are concave from the middle to an edge to a depth exceeding 0.000002 inch shall be subject to rejection. (See 4.4.3.) The finish of gaging surfaces shall meet the requirements of table III when tested by means of a surface roughness analyzer of known accuracy. (See 4.3.1.)" We felt that the stipulation regarding concavity was necessary, particularly on the reference grade of blocks, because such blocks do not wring satisfactorily, and when the edge is turned up in that way one is liable to damage other blocks when wringing the gages. Our practice in this country, at least here at the Bureau, has been to check flatness of gages not wrung down if they are 0.1 in. in size or longer. The British practice is to wring them down up to 0.160 in. Our specification is considerably more rigid, then, in respect to flatness because it will not accept blocks that are warped. Of course, warped blocks again present a difficulty in wringing. When the blocks are thinner than 0.1 in. they often cannot be readily wrung down, and actually it is almost impossible to obtain a thin block that is not warped. The warping in general extends to about the 0.115 in. size, but from 0.1 to 0.115 in. the warping is not serious; the blocks generally wring down satisfactorily. They are usually within the tolerance specified for flatness.

Turning to table III we might examine the tolerances specified. These tolerances compare quite closely with those specified in the British standard, there being differences of one or two millionths inch in certain categories. With regard to surface finish specifications, I think we should consider that a requirement for wringability might serve as a substitute for flatness and surface finish requirements, as Dr. Engelhard pointed out this morning. However, our practice is well established along the lines laid down and seems to be working satisfactorily. In any case, I think that a wringability test is an important test to make because it uncovers a variety of defects.

Referring to the last column of table III and the referenced footnote, which states that "all scratches shall not exceed the depth specified except that random scratches not exceeding twice the specified depth may occur at intervals of 0.001 in. or greater": the interval of 0.001 in. represents about one-half of the diameter of the field seen in a microinterferometer. A series of scratches, which are somewhat deeper than the average specified, can be tolerated if there are reasonable spaces separating them.

Table IV, tolerances on lengths of gage blocks, conforms with practice except that we have specified a biased bilateral tolerance, more plus tolerance than minus. As you heard this morning, there is some objection to specifying tolerances in that way. It is a matter of a choice of two evils. We tried balanced bilateral tolerances. If one were buying a B block with a tolerance of ± 0.000008 in. and then allowed 0.000003 in. observational error, one might get a block 0.000011 in. short, that is a block which is worn out before it is used. For that reason we have given a plus bias to the tolerances, and we feel that in general, the random sizes that we will get in a set of blocks will not all be excessively plus. Some will be minus and, in general, the combinations are not expected to be biased plus to any detrimental degree.

Referring to paragraph 3.5.3.3, Squareness of sides, we have specified a tolerance of 5 min. When the blocks are supported in a horizontal position, if the sides are appreciably out-of-square there may be difficulty. This tolerance of 5 min we found to be more or less representative of blocks which we check. The British specification is 0.001 in. over the length of the gage. In a 1-in. length that amounts to 3.5 min compared with our tolerance of 5 min. Specifying it as they do, the specification becomes more rigid as the length increases.

I call your attention to the fact that we do require that a manufacturer's certificate of inspection be furnished with each set. This is desirable because it assures that the set has been inspected properly. Also, for the person who inspects it for the government, a discrepancy would give an indication that perhaps the measurements of a given block should be repeated.

Under paragraph 3.4.2; "Cases shall be so designed that when the case is open the blocks, attachments, or accessories are readily removable, but when closed and fastened they shall be firmly held in place. The means of fastening the lid shall be of such strength and positive action that the lid will not open during shipment as the result of either breakage or opening of the fasteners. Unless otherwise specified in the invitations for bids, contract, or order the case dimensions shall be in conformance with manufacturer's standard practice." For some time sets of blocks that were shipped to the Bureau for calibration were often received in damaged condition, either because the clasps on the cases broke or they were not fastened securely.

We have stated a requirement for wringability of blocks under "Inspection", paragraph 4.3.2: "Either as a separate test, or in the course of testing the parallelism as required under 4.4 and 4.5, the wringing quality of all blocks shall be examined. When wringing or gaging surfaces are wiped clean with a dry cloth or chamois, not less than 95 percent of the surface shall wring down on a transparent (quartz or glass) optical flat, as indicated by the absence of color between the wrung surfaces when viewed in white light." We feel that the block should wring down over the entire surface, but we can

tolerate a spot that does not, provided that the block is within the flatness tolerance.

In the inspection of blocks we feel that all AA and A blocks should be inspected, that is, the entire set of blocks. Most of this inspection will be performed by government laboratories other than the National Bureau of Standards. The Bureau is not at present in a position to undertake what we might term acceptance inspection of gage blocks. Our function is to provide calibrated masters where they are needed. Occasionally we have had the experience that a set has been shipped here by the manufacturer, and it has been stated that the agency ordering the set has required a certificate from the Bureau before the set is accepted. Usually that is done without the foreknowledge of the Bureau and is a practice that should be avoided. If the buying agency of the government wants an inspection by the Bureau, the agency should order it directly from the Bureau.

In the inspection of B blocks we have tried to set up what might be called a sampling plan. Grade B blocks do not require 100-percent inspection of the set. We have put in this stipulation that "the 2-, 3-, and 4-inch blocks shall be measured individually and a sample of 15 gage blocks shall be drawn at random from the set, of sizes to and including .1 inch. The length correction of each block shall be determined in accordance with 4.4.1. The set shall be regarded as conforming to the requirements of this specification for length if both: (a) All individual errors are within the interval -8.4 to $+12.4$ microinches, or this interval times the length in the case of the 2-, 3-, and 4-inch blocks; (b) after subtracting 2 microinches from each error for the blocks in the random sample and regarding these as whole numbers, the sum of their squares is less than 213."

This specification was worked out after we spent hours with members of the Mathematics Division and statistical experts from the Bureau of Ships. Mr. Marthens is going to present some charts later showing the basis of that specification.

In addition to this test of 15 blocks, we would require what is called a composite test. "A composite test involving length, parallelism, flatness, and wringing quality shall be made as follows: Nine groups of blocks shall be formed from a set, each group consisting of from 8 to 10 blocks each. The blocks of each group shall be wrung together and the combined length of each group measured. The set shall be regarded as conforming to the requirements of this specification if: (a) Each of the 9 groups has a correction of less than 28 microinches, or (b) 8 groups have corrections less than 28 microinches and the remaining group, having a correction larger than 28 microinches, has not more than one block outside of the tolerance for length when each block of that group is measured."

Another requirement in the specification which might be discussed further is that for hardness. It has been traditional in this country to require a hardness of Rockwell C65 on steel gage blocks as a minimum. As was brought out here in the discussion yesterday morning, blocks that are not quite so hard are more likely to be stable than blocks hardened to C65. Thus there is again a choice as to which is more tolerable, lesser hardness with greater stability or higher hardness and increased wear resistance with less stability.

16. Discussion

Discussion relating to papers Nos. 7 and 11

QUESTION: Do you substitute the average depth for what is commonly known as a root mean square?

MR. STRANG: The trend in this country now is to eliminate the rms value. This has been accepted in most industries. Arithmetical average seems to be a simpler value to understand. We are using, and many European countries are using, what is called the center line average, or arithmetical average.

MR. HAVEN: Arithmetical average has been officially adopted in this country, Mr. Curtis.

MR. STRANG: Theoretically there is about a 10 percent difference between the two values. Practically you usually obtain about the same value using instruments which indicate in rms or arithmetical average units.

MR. HAVEN: Are there any other questions?

DR. GARDNER: I don't have a question but I wish to refer to Mr. Moody's paper. I enjoyed it very much. There was one thing he mentioned which I think should have been emphasized a bit more, perhaps. That is, that this radiant energy that heats these gages is not necessarily from light bulbs. A good bit of the radiant energy comes from material that is at such a relatively low temperature one can't see it at all. We were working, for instance, in a room that had one wall separating the room from the hall and one wall separating the room from the earth. The difference in temperature of these two walls gave a radiation effect which caused us quite a bit of trouble until we eliminated it by use of aluminum foil. Mr. Moody mentioned this effect but he talked so much more about lights that I wanted to emphasize that it isn't only the sources of visible radiation in the room that cause this heating effect.

MR. HAVEN: I think that everyone who has worked in laboratories has found that lights do have an effect on gage standards. I am glad Dr. Gardner brought out the effects of other sources of radiant energy.

QUESTION: From the standpoint of acceptance and inspection of blocks, would you care to indicate your preference for instrumentation, the tracer type instrument or microinterferometer or a combination?

MR. STRANG: I think that if a tracer type instrument with a slower tracing speed were available, I would prefer it. Average deviations from a mean line obtained with the microinterferometer are usually about twice the values obtained with a tracer type instrument. I think the center line average values shown in the slides are too low. If the tracer could move slowly, I believe it would give fairly good values. If it moves too fast, there is some loss in the indicated depth. With microinterferometers that are available now the scratch width won't show up unless you increase the magnification of the instrument or make enlargements. The charts obtained with a tracer type

instrument are much easier to interpret than the fringe patterns of the microinterferometer.

QUESTION: How often would the tracer point have to be checked if it were used continuously?

MR. STRANG: We haven't worn a tracer point to any appreciable extent so I can't answer that question. From what we have heard they last a long time.

MR. A. W. YOUNG: I would like to point out two things. Someone here mentioned a tracer stylus of 30 microinches radius which wore very rapidly. We have checked tracer styli periodically and I haven't observed too much change in them. As far as the stylus entering at a slower speed, Dr. Rolt pointed out in his paper that they have made comparative tests on gage blocks and in that case what was done—I believe Eric Lindberg of General Motors Research has done some of the same work—was to slow up the motor drive by using a manual control. You can get a much slower manual drive which would allow the stylus to enter at a very slow speed. All that is required is a simple suction attachment.

MR. HAVEN: I think we have to recognize that the Talysurf is a general purpose instrument. The finish on gage blocks represents a rather extreme application and calls for special methods.

Are there any other questions? (None)

Discussion following papers Nos. 4 and 12

MR. HAVEN: Are there any questions?

QUESTION: I wonder if you could tell me the movement of the micrometer screw for the total two ten-thousandths of an inch range of this interferometer?

MR. T. R. YOUNG: The total travel is one inch.

QUESTION: One inch on the micrometer represents 2 ten-thousandths of an inch in the length of the gage?

MR. YOUNG: Yes.

QUESTION: Thank you.

MR. HAVEN: Are there other questions?

DR. GARDNER: Mr. Chairman, I have very much enjoyed the presentation of Mr. Johansson and agree with him on essentially all points. I think if the National Physical Laboratory and the National Bureau of Standards had the privilege of deciding between the American and British inches this matter would have been settled long since. Unfortunately, it is not so simple. I don't know what the British problem is but here we have to convince Congress that this change should be made. That isn't always the most direct method of procedure. The ASA standardization is admittedly sort of a stop-gap, but it is very surprising the respect some people have for these inches which we think are sort of outworn and outmoded. I would not want this assembly to go away believing that the failure to come to an agreement on these inches is an indication of the failure of scientific men or of the two national laboratories. It is purely a matter of legal complications which are much more than you would imagine.

Regarding the chromium plating, I believe the National Bureau of Standards, in another section, has developed a very ingenious instrument for measuring the thickness of chromium plating, particularly when it is applied to a substance such as steel. We can measure these chromium layers without mutilating them and without the

slightest difficulty. I don't know whether we have reached an opinion here as to the most desirable thickness of chromium or not, but it can be readily measured once we have decided on the matter. I believe the Bureau certificates now give the measurement at the center of a gage block instead of at the edge. As to the accuracy and precision, we are constantly trying to improve both here. We have a rather extensive development program in progress and the instrument that you have seen today is one of our steps in progressing towards a more precise and accurate method of determining the length of gage blocks.

There is a committee in the Bureau that is considering this whole question of how to state accuracy and precision and what we should say, and I think before very long our reports will be perfectly adequate in that respect. It is not an easy problem. To make a definite statement of the precision of the measurement which we can stand by and to make a definite statement of the accuracy is not simple. In fact, it is impossible, but we can make statements that will be true perhaps 99 percent of the time.

Mr. HAVEN: Are there further comments or questions?

VOICE: I don't think that the user should be expected to accept a grade A block that is in error by 7 millionths, because, as I understand it, Dr. Gardner just stated that the 3 millionths observational error is more or less in there to reduce controversies and that the size that is given for the block is usually more accurate. I am afraid that some of the manufacturers are taking advantage of the uncertainty in measurement. I never liked it and I don't think we should be expected to accept blocks that are in error by the tolerance plus the full amount of the uncertainty.

Mr. FULLMER: I might say that the practice of the Bureau has changed in recent years with regard to certification of blocks. During the war particularly, when acceptance of blocks by the government was based on our calibration, we actually listed the criteria for the various grades of blocks and applied the observational errors to the tolerance. We have discontinued that practice entirely. Our calibration reports now merely state the size of the block, the errors, etc. There is no judgment passed on the block as to whether it should be accepted or rejected by the users.

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INTERFERENCE METHODS FOR STANDARDIZING
AND TESTING PRECISION GAGE BLOCKS

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INTERFERENCE METHODS FOR STANDARDIZING AND TESTING PRECISION GAGE BLOCKS

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ABSTRACT

Precision gages, which are blocks of metal (usually steel), having two opposite faces plane, parallel, and a specified distance apart, are used in the shop as reference end standards for checking micrometers and other measuring instruments, and also as distance pieces or size blocks for precise mechanical work. The extensive use of precision gages necessitated by the small tolerances allowed in the manufacture of interchangeable machine parts has required more accurately determined end standards and more rapid and precise methods for comparing gages with these standards than have been previously available.

Since comparisons of end standards with line standards by means of micrometer-microscopes and of precision gages with end standards by means of contact instruments are subject to appreciable errors, methods which make use of the interference of light waves were used in making these measurements. With the interference methods described in this article the planeness and parallelism errors of precision surfaces can be measured, and the length of standard gages can be determined by direct comparison with the standard light waves with an uncertainty of not more than a few millionths of an inch. The errors of other gages can be determined by comparison with these calibrated standards with equal precision. This process makes the standard light waves, which have been determined to one part in four or five million relative to the international meter the standards of length for this work.

The apparatus used for calibrating standards and comparing other gages with these standards is illustrated by line drawings and thoroughly explained.

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I. INTRODUCTION

Precision gages, which are blocks of metal (usually steel) having two opposite faces plane, parallel, and a specified distance apart are used in the shop as reference end standards for checking micrometers and other measuring instruments, and also as distance pieces or size blocks for precise mechanical work. The extensive use of precision gages necessitated by the small tolerances allowed in the manufacture of interchangeable machine parts, and the development of the process of making these gages with errors of construction that seldom amount to 0.5 micron (0.00002 inch)—in most cases to not more than 0.25 micron (0.00001 inch)—have created a need for more rapid and precise methods of test and also for more accurately determined reference standards than have been available. Since tests of the accuracy of precision gages can be most readily made by comparison with secondary end standards of the same size and shape, gage blocks with very nearly perfect surfaces can be used in the testing laboratory as secondary end standards when they have been accurately calibrated relative to some fundamental standard of length and when it has been proved that they retain their dimensions for a considerable period of time.

The fundamental standard of length, the International Prototype Meter, is a line standard whose length is the distance between two lines ruled on a platinum-iridium bar carefully preserved in a special vault at the International Bureau of Weights and Measures near Paris. A duplicate of this bar, Meter No. 27, which is kept at the Bureau of Standards, is the primary standard of length for the United States. Other similar bars, graduated in various subdivisions of a meter and calibrated relative to the primary standards, are used as secondary or working standards. In calibrating these secondary and intermediate line standards errors of 0.2 micron

(0.000008 inch) are possible, due to imperfections in the ruled lines.

The end standards of the Bureau of Standards have been calibrated heretofore by comparison with these line standards by the methods of Fizeau¹ and Fischer.² These methods, requiring the use of microscopes, are subject to errors of measurement which may amount to 0.5 micron (0.00002 inch).

The comparisons between the secondary end standards and the precision gages are usually made with contact comparators subject to errors of 0.5 micron (0.00002 inch).

Inasmuch as the tolerance on some machine work is ± 2.5 microns (± 0.0001 inch), requiring the precision gage of the shop to be correct within 0.25 to 0.5 micron (one or two hundred-thousandths of an inch), this degree of precision in the gages requires the secondary end standards of the testing laboratory to be correct within a few hundredths of a micron (millionths of an inch). This tolerance also requires that the errors of measurement in making the comparison between the gages and the standards should likewise be limited to a few hundredths of a micron. It is impossible to attain this precision in measuring the irregularities in the surfaces of gages or end standards with any micrometric apparatus, in calibrating end standards by comparison with line standards, or in making comparisons between end standards and precision gages with any contact apparatus, because in each case the error of measurement may be several times the allowable error. The error of measurement in the step from the standard meter to the gage should not be more than 0.02 to 0.06 micron (one to three millionths of an inch).

In December, 1917, we undertook to develop an optical method for making these measurements, and in a short time, by applying methods which make use of the interference of light waves, succeeded in solving the problem.³

II. OPTICAL METHOD

Instead of calibrating the end standards by direct comparison with line standards derived from the meter the lengths of standard light waves were chosen as the primary standards. These

¹ *Travaux et Mémoires du Bureau International des Poids et Mesures*, 10.

² *Bull. Phil. Soc. of Washington*, 18, p. 241; 1898.

³ Reports on these methods have been given at the following meetings: Peters, American Society of Mechanical Engineers, New York, Dec. 5, 1918. Peters and Boyd, American Physical Society, Washington; Apr. 25, 1919; American Society of Mechanical Engineers, Washington, May 30, 1919; and Philosophical Society of Washington, May 29, 1920. A popular article on the methods was published by Peters and Boyd in the *American Machinist*, Sept. 30 and Oct. 7, 1920.

waves possess all the necessary properties of fundamental units of length, the most important of which are constancy, reproducibility, accuracy of measurement, and ease of application. As to their constancy, reproducibility, and accuracy of determination little need be said. The wave lengths of red, yellow, and green radiations from cadmium have been determined by direct comparison with the standard meter by Michelson ⁴ in 1893 and re-determined by Fabry and Benoit ⁵ in 1907, the values obtained from these two independent determinations agreeing to one part in sixteen million when corrected to similar conditions.

Numerous comparisons made by spectroscopists between these fundamental wave lengths and the wave lengths emitted by other luminous substances prove that the light waves are the most dependable length units known. Their work has placed at our disposal a large number of light sources which can be easily obtained and operated and which emit radiations whose wave lengths are known to one part in four or five million. The only remaining requirement is the easy application of these waves in the calibration of end standards.

In making this application incandescent neon and helium gases, giving wave lengths ranging from 0.40 to 0.75 micron (0.000016 to 0.000030 inch) were used. These have been determined⁶ with an accuracy of one part in four or five million and found to be exactly reproducible within the limits of observational error. For an accuracy of one millionth of an inch a comparison with these waves is therefore exactly equivalent to a comparison with the standard meter.

Three sets of carefully selected gages, each containing 81 blocks of various lengths from 0.05 to 4 inches, were chosen as the secondary end standards for gage calibration by interference methods. The planeness and parallelism of the surfaces and length of the gages were determined by means of the interference fringes seen in monochromatic light. To test for planeness, a glass true plane was placed over each gage surface and the curvature of the interference fringes in monochromatic light was determined by means of the Pulfrich instrument described below. Each gage was then made the separator for two interferometer plates, thus forming a Fabry and Perot interferometer. Lack of parallelism of the sur-

⁴ Michelson, *Travaux et Mémoires du Bureau International des Poids et Mesures*, 2; 1895.

⁵ Fabry and Benoit, *Travaux et Mémoires*, 15; 1913.

⁶ Burns, Meggers, and Merrill, *B. S. Sci. Papers*, No. 329; 1918.

faces was determined on moving the eye across the plates by the expansion or contraction of the circular (Haidinger) rings, produced when the interferometer was illuminated with radiation from a helium source. The distance between the interferometer plates was determined from measurements on the diameters of these rings. From this determination, by applying corrections for the density of the air, lack of parallelism of the gage surfaces, and thickness of the metallic film on the interferometer plates, the length of the gage was obtained.

Several independent determinations made the same day on a given gage agreed to 0.025 micron (0.000001 inch). Intercomparisons between similar gages from different sets and between combinations of different gages proved that errors in the determinations of these gages were in no case greater than 0.07 micron (0.000003 inch), and these errors were due in most cases to irregularities in the surfaces of the gages. With perfect gages and accurately controlled conditions the precision of the measurements should be comparable with the highest precision obtainable in wave-length measurements.

After these end standards were calibrated a large number of precision gages were compared with them by means of an interferometer comparator. With this instrument one person is able to test the planeness and parallelism of the surfaces and length of about 100 gages per day with an uncertainty of not more than 0.07 micron (0.000003 inch). During the past year about 30 000 precision gages have been tested at the Bureau of Standards by two workers, which shows that the method is sufficiently rapid for quantity work.

Thus it has been possible to originate, by the method fully described below, end standards directly from the standard light waves and to compare large numbers of commercial gages with these standards by means of light interference, with an accuracy far greater than can be attained in the manufacturing shops of the country.

III. INTERFERENCE OF LIGHT

The sensation of light is due to transverse waves radiated by luminous bodies. These waves vary in length, giving rise to different color sensations. The range of the wave lengths visible to the eye is from about 0.4 micron (0.000016 inch) for blue light to 0.7 micron (0.000028 inch) for red.

If two trains of waves from one point in a source having traversed different paths fall upon a point on the retina of the eye, the resultant amplitude of vibration determines the brightness. If they are "in step," maximum brightness results. If, however, the troughs of the one arrive with the crests of the other, destructive interference takes place, resulting in relative darkness. If the two trains travel different distances, so that the difference in path is some whole number of wave lengths, then the waves will reach the eye in phase. If the difference in path is equal to some whole number of wave lengths plus one-half wave length, the waves in the two trains will be in opposite phase, so that destructive interference takes place. The conditions for interference are realized when light from an extended source *S*, Fig. 1, falls on a thin transparent film. Part of the light is reflected from the first surface, *ABCD*, and the remainder is transmitted to the second surface, *ABGF*, where partial reflection again takes place. Since the wave trains reaching *E* from these two reflections have traveled over different distances, reinforcement or destructive interference can, therefore, occur. When white light is used and the film is sufficiently thin, a few brightly colored bands are seen across its surface. If monochromatic light—that is, light of one color or of very limited spectral extent—be employed, alternate light and dark bands or interference fringes may be observed to cross the film.

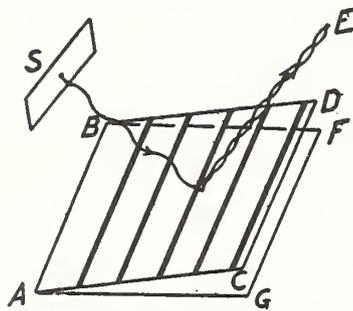


FIG. 1.—Interference of light reflected from the surfaces of a thin film

Part of the light is reflected from the first surface, *ABCD*, and the remainder is transmitted to the second surface, *ABGF*, where partial reflection again takes place. Since the wave trains reaching *E* from these two reflections have traveled over different distances, reinforcement or destructive interference can, therefore, occur. When white light is used and the film is sufficiently thin, a few brightly colored bands are seen across its surface. If monochromatic light—that is, light of one color

or of very limited spectral extent—be employed, alternate light and dark bands or interference fringes may be observed to cross the film.

If one of the surfaces of the film be plane, the shape of the other surface can be obtained from measurements of the film thickness at several points. To derive the relation between the film thickness *t* and the shape of the interference fringes, consider the thin film formed by the two plane surfaces which are represented by the traces *AC* and *AG*, Fig. 2, inclined at a slight angle ϕ . Observing the film at an angle θ with the normal, rays of light originating at the point *S* in the source and reflected by the two surfaces *AC* and *AG* will appear to come from the two sources *S*₁ and *S*₂. The difference in path of two rays reaching *E* is $N\lambda$, where *N* is the "order of interference" or number of waves in the

distance S_2K and λ is the wave length of the monochromatic light. Let the distance between the two images of the source $S_1S_2 = h$.

$$\begin{aligned}
 N\lambda &= h \cos (\theta + \alpha) \\
 &= h \cos \theta \cos \alpha - h \sin \theta \sin \alpha \\
 &= 2(a+b) \sin \phi \cos \theta - 2d \sin \phi \sin \theta \\
 &= 2(a+b) \sin \phi \cos \theta - 2b \sin \phi \cos \theta \\
 &= 2a \sin \phi \cos \theta \\
 &= 2t \cos \theta.
 \end{aligned}
 \tag{1}$$

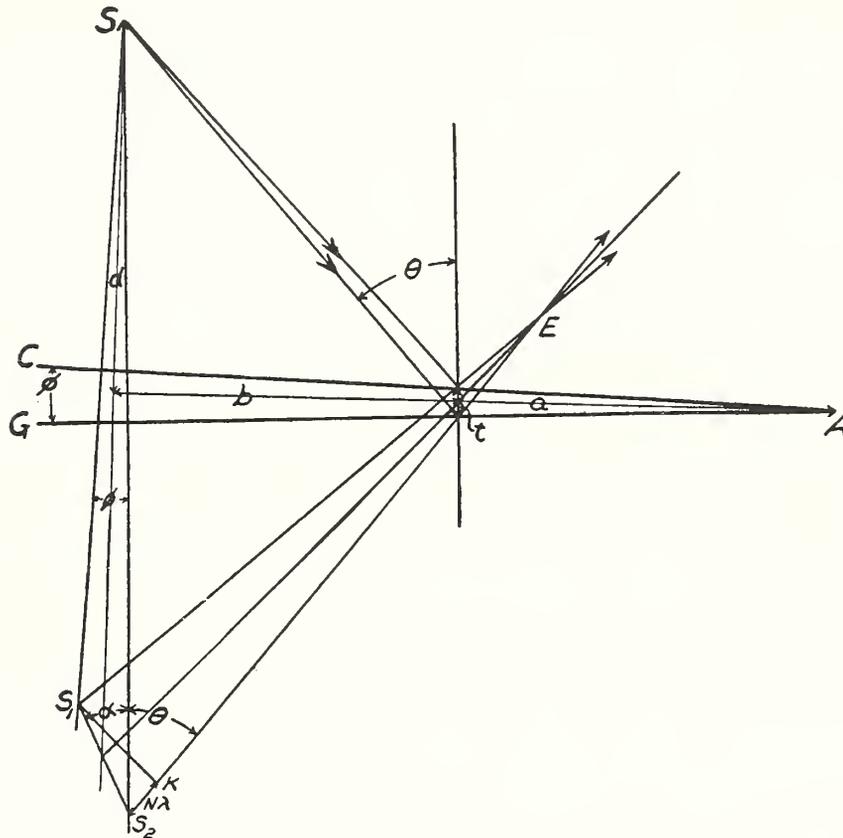


FIG. 2.—Relation between the film thickness and the order of interference

If N is an integer, the two wave trains will be opposite in phase, due to the phase change $\left(\frac{\lambda}{2}\right)$ by reflection at the denser medium,⁷ and the point observed will in that case appear dark. The locus of the points for which N is a given integer is the interference fringe of the N th order and appears as a dark band across the film.

⁷ Only under special cases is this phase change exactly a half wave length; but it is not necessary in the present case to determine its exact value.

Equation (1) may also be expressed in the form

$$t = N \frac{\lambda}{2} \frac{l}{P}, \quad (2)$$

where P represents the perpendicular distance from E to the film and l the distance from E to the point at which t is taken.

IV. TEST FOR PLANENESS OF SURFACES

1. PERPENDICULAR INCIDENCE

In testing the planeness of polished surfaces, such as are produced on prisms, surface plates, precision gages, or micrometer anvils, a test plate is placed in close contact with and slightly inclined to the surface to be tested, thus forming a thin wedge-shaped film discussed in the previous section. This test plate is of glass, one surface of which has been polished accurately true plane and tested against a master true plane or liquid surface of large extent. The accuracy of the test is, of course, limited by the irregularities of the test-plate surface. It is very difficult to make glass surfaces 50 to 75 mm (2 to 3 inches) in diameter plane within 0.25 micron (0.000010 inch), and to reduce this error requires exceptional skill. For ordinary work, however, test plates plane within 0.25 micron (0.00001 inch) are sufficient, but when it is necessary to determine irregularities of a few hundredths of a micron (millionths of an inch) in the surface under test, great care must be exercised in selecting and testing the test plate.

In order to give a definite value to the wave length λ , the thin wedge-shaped film of air formed between the plane surface of the test plate and the surface under test is illuminated with monochromatic light. A convenient source of monochromatic light is a Bunsen flame, in which is inserted a piece of asbestos soaked in a salt solution, or a ground glass plate illuminated either by a helium lamp operated on a 5000-volt ac circuit, or by a mercury vapor lamp covered with a green glass screen. The wave lengths of the most effective visible radiation from these sources are approximately:

Sodium (yellow) = 0.589 micron (0.0000232 inch).

Helium (yellow) = 0.588 micron (0.0000231 inch).

Mercury (green) = 0.546 micron (0.0000215 inch).

A colored glass screen illuminated by an incandescent lamp or ordinary daylight may be used as a source if high precision is not

desired, but the light will not be sufficiently monochromatic to allow assignment of a definite value to the most effective wave length.

A very convenient instrument for illuminating the film and at the same time viewing the interference fringes on the film is one designed by Pulfrich⁸ and shown in Fig. 3. The light from a helium lamp *H* is focused upon a small total reflection prism *p*. After being collimated by the lens *O*₁ it is reflected by the prism *R* down to the interferometer *ABD*, which is in the focal plane of the lens *O*₁. The rays reflected by the film surfaces are brought to a focus by the lenses *O*₁ and *O*₂ upon the slit *S*, and images of the interference pattern and the reference marks on the test plate are viewed with the eyepiece *C*. The direct-vision prism *K* separates the fringe patterns due to the helium light of different wave lengths. A pair of cross wires located at *S* and operated

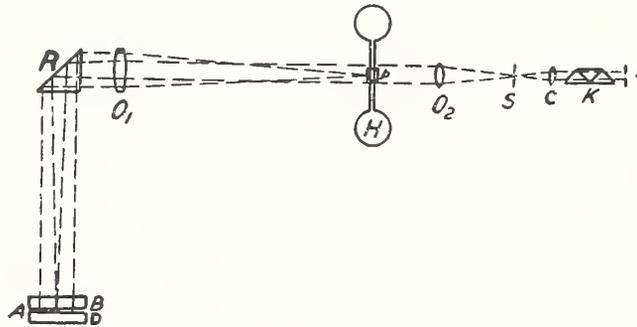


FIG. 3.—Optical system of the Pulfrich instrument

by a micrometer head make it possible to measure small displacements of the fringes from the straight reference lines ruled on the test plate *AB*. With this instrument and an exceedingly true test plate the planeness of an unknown surface can be measured with an accuracy of 0.025 micron (0.000001 inch). The only objection to the instrument is that the field is limited to about 2.5 cm (1 inch), so in testing a large surface only a small portion can be seen at one time. Instruments of similar optical design described by Schultz⁹, Schönrock¹⁰, and Laurent¹¹ eliminate this objection by having fields of view as large as 25 cm (10 inches). With these four instruments the rays of light coming from the source to the film surfaces are made parallel by the lens systems and when reflected back to the eye pass along the perpendicular

⁸ Pulfrich, *Zeits. f. Inst.*, 18, p. 261; 1898.

⁹ Schultz, *Zeits. f. Inst.*, 36, p. 252; 1914.

¹⁰ Brodhun and Schönrock, *Zeits. f. Inst.*, 22, p. 355; 1902.

¹¹ Laurent, *C. R.*, 96, p. 1035; 1883.

to, say, the first surface, and this condition holds over the whole field of view. Under such conditions, in equation (1) $\cos \theta = 1$, and

$$2t = N\lambda, \tag{3}$$

which states that the difference in path of the two interfering trains is simply equal to the double thickness of the film (the double distance signifying that the light travels down and back through the film).

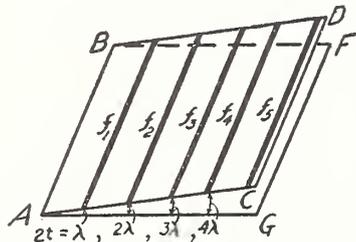


FIG. 4.—Interference fringes, plane surface

From this equation it is evident that where N is constant—that is along any one fringe— t is also constant. Hence, the fringes trace lines of equal separation of the two surfaces.¹² Starting from the line of contact AB of the two

plane surfaces, Fig. 4, and moving to a wider part of the film, when $2t = \lambda$ interference occurs causing the first dark fringe f_1 which is a straight line parallel to AB . When $2t = \frac{3}{2}\lambda$, the wave trains reinforce each other and a bright fringe is produced. Moving to a still thicker part of the wedge, where $2t = 2\lambda$, a second dark fringe f_2 will occur, etc. From this it is evident that if the surfaces are plane the fringes will be straight lines, equally spaced, and parallel to the line of intersection of the surfaces. The next dark fringe always occurs on passing to where the double separation increases by λ . Hence, the distance between fringes depends on the inclination of the surfaces.

If a plane surface be brought in contact with a convex spherical surface, Fig. 5, then at C the point of contact $2t$ is equal to zero. Radially from this point the separation of the surfaces increases uniformly in all directions, so the fringes, and hence the lines on which $2t = N\lambda$, are concentric circles around the point of contact as a center: On any ring the distance of the spherical surface

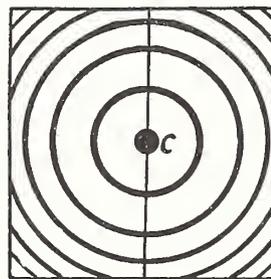


FIG. 5.—Interference fringes, convex spherical surface

¹² The fact should be stressed here, however, that the fringe marks the line of constant thickness of the film only when the direction of view is perpendicular to the film over the whole film. Observed obliquely, straight fringes do not indicate that the tested surface is plane, as shown below.

from the plane is equal to the number of the ring, counting from the point of contact, times $\frac{\lambda}{2}$. By pressing down at *A* the plane surface can be made to roll on the spherical surface, shifting the point of contact and with it the center of the ring system in the direction of *A* or toward the point of application of the pressure. With a convex surface, therefore, the center of the ring system lies at the point of minimum separation.

If one of the surfaces be concave and spherical, Fig. 6, a similar system of concentric circular fringes is produced, but in this case the center of the system lies at the point of maximum separation. Pressing down on *A* causes the center of the ring system to shift toward *B*, the direction of increasing separation, away from the point of application of the pressure. Thus, a slight pressure on one edge of the plane surface *AB* serves to indicate whether the curved surface is convex or concave.

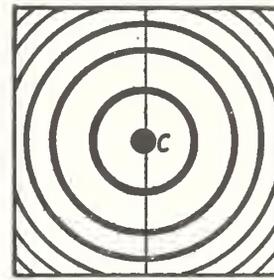


FIG. 6.—Interference fringes, concave spherical surface

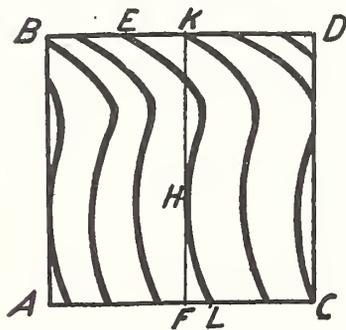


FIG. 7.—Interference fringes, irregular surface

With one surface plane and the other irregular the fringes are irregular, each of which follows the line of equal separation of the surfaces. Whether the irregularity is a projection or depression can be determined by applying a slight pressure to one edge of the upper surface and noting the direction of shift of the fringes.

The amount a curved surface deviates from a true plane can be readily estimated as follows: Draw a straight line *FK*, Fig. 7, across the center of the true plane surface, parallel to the line of contact *AB* of the surfaces. Bring this line tangent to one of the fringes at, say, the point *H*. It is evident that this line represents the direction a fringe through *H* would take if the irregular surface could be converted into a plane tangent to the irregular surface at *H*. The fractional

part of the distance between two fringes by which the fringe *EL* deviates from the straight line *FK* gives the fractional part of a half wave length by which the irregular surface deviates along *FK* from true plane. With *AB* as the line of contact, the point *F* is estimated to be one-fourth wave length above and *K* one-half wave length below the plane surface tangent at *H*.

2. ERRORS OF INTERPRETATION

Although the theory is quite explicit, it is a very common mistake to assume that straight fringes always indicate planeness when a surface under test is placed on a true plane, and conversely curved fringes indicate that the surface is not a true plane. Both of these interpretations may be wrong. For example, the operator

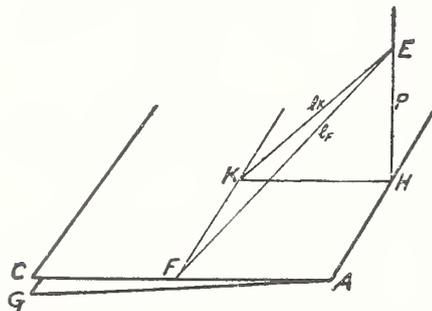


FIG. 8.—Errors of interpretation caused by viewing the film obliquely

may view the light reflected obliquely by the film, and to make sure that the fringes are straight he may lower his eye to sight along them. Or, the fringes from one position appearing curved, he may shift to another and find them quite straight, then draw his conclusions from what he observes under these, to him, best conditions.

It is important to know something about the magnitude of the errors which may be introduced by the assumption that straight fringes always indicate planeness and curved ones indicate curvature of the surface tested. The treatment of this falls under the general case shown in Fig. 2, where the thickness of the film at any point is given by equation (2)

$$t = \frac{N\lambda}{2} \frac{l}{P}, \tag{2}$$

which goes, for normal incidence, into equation (3)

$$t = \frac{N\lambda}{2}. \tag{3}$$

Suppose from the point *E*, Fig. 8, a straight fringe is observed along *FK* which is drawn parallel to *AH*. With one of the film surfaces plane, how much does the other diverge from plane

between F and K ? Using (3) instead of (2) in determining the thickness of the film at any point gives

$$t' = \frac{N\lambda}{2}. \quad (4)$$

The error introduced is

$$t - t' = t' \left(\frac{l}{P} - 1 \right). \quad (5)$$

The actual deviation from planeness between F and K is

$$\Delta t = t_F - t_K. \quad (6)$$

The observed deviation from (3) is

$$\Delta t' = t'_F - t'_K. \quad (7)$$

The error introduced by using (3) instead of (2) is

$$S = \Delta t - \Delta t' = \left(\frac{l_F}{P} - 1 \right) t'_F - \left(\frac{l_K}{P} - 1 \right) t'_K = \left(\frac{l_F}{P} - 1 \right) \Delta t' + \frac{t'_K}{P} \Delta l \quad (8)$$

Where $\Delta l = l_F - l_K$.

This equation is seen to be consistent in that S becomes zero for normal incidence at both F and K , in which both $\left(\frac{l_F}{P} - 1 \right)$ and Δl are zero.

Sighting along a line from a normal position over K , $l_K = P$;

$$S = t'_F \left(\frac{l_F}{P} - 1 \right),$$

which states that the error in determining how much the tested surface deviates from the plane is simply equal to that in determining the thickness of the film at F , equation (5).

If one observes the fringe obliquely at both F and K , l_F and l_K increasing and at the same time approaching equality, we have for a given length FK the error represented by the term that contains $\Delta t'$, increasing with θ , while that represented by the term containing t'_K is decreasing. Take some numerical examples:

(a) With the eye 10 inches above the center of a 6-inch surface being tested on a true plane and viewing a diametrical fringe, $P = 10$, $l_F = 10.445$, $l_K = P$, applying (8)

$$S = 0.044 \Delta t' + 0.044 t'_K.$$

(b) With the eye moved back 10 inches in a direction at right angles to the fringe, P remaining constant, and viewing the center K and the farthest edge F , $l_F = 14.46$ $l_K = 14.14$ we have from (8)

$$S = 0.446 \Delta t' + 0.032 t'_K.$$

(c) With the eye moved back 20 inches instead of 10, $l_F = 22.56$, $l_K = 22.36$,

$$S = 1.256 \Delta t' + 0.020 t'_K.$$

A comparison of (a), (b), and (c) shows that the error arising from the actual deviation in planeness increases rapidly with oblique inspection, while the error rising from the thickness of the film decreases.

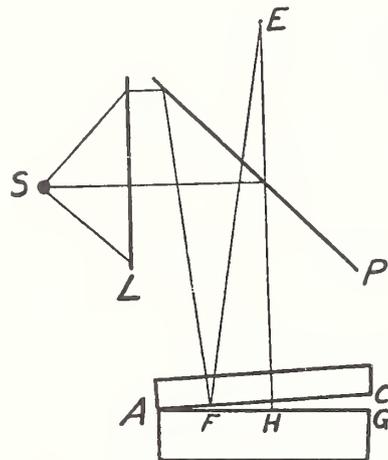


FIG. 9.—Optical system for testing surfaces

Both these effects cause the fringes to straighten with increasing θ . Therefore, using oblique incidence slightly reduces the error of measurement if the surface under test is plane and greatly increases the error if the surface deviates from plane. An improvement on the customary apparatus is made by the addition of a thin plate of glass P , set at an angle of 45 degrees to the perpendicular EH from the eye to the surface, Fig. 9. With this arrangement the error due to deviation of the surface from plane is eliminated and a correction can be applied for error due to the film thickness.

V. ADHERENCE OF SURFACES

When the plane surfaces of two gages, or of a gage and a glass plate, are brought into intimate contact, they adhere to each other, necessitating considerable force to separate them. To cause this adherence the surfaces are first washed with benzol, then with alcohol, and finally wiped with clean cotton to remove all traces of grease and dust. The surface of the plate is brought in contact with the gage surface and pressed on tightly to force out the film of air. A drop of alcohol when placed on the plate against the gage will spread out around the gage and pull the two surfaces into very close contact. Any excess of liquid can be forced out by sliding the surfaces on each other. When they

come into close contact the adhesive force causes them to grip each other and resist a large separating force.

A large number of measurements we have made show that when two very plane surfaces are brought into contact in this manner the separating film is not more than 0.025 micron (0.000001 inch) thick. Doubtless the surfaces come into intimate contact at the high points, the liquid filling the fine furrows or scratches left by the finishing laps. Our tests show that two gages with the ordinary lapped finish when brought into contact as described require to separate them a pull in the direction perpendicular to the surfaces of from 2.5 to 2.8 kg, per square centimeter (35 to 40 pounds per square inch).

With gages possessing a high optical polish more intimate contact is possible—that is, the capillary film is much thinner—and the required separating force ranges between 6.7 to 7 kg. per square centimeter (95 and 100 pounds per square inch). Considering the extreme thinness of the separating film when good contact exists, the need of exceedingly plane surfaces is apparent. A nick or burr on the edge or a small surface elevation which holds the two surfaces 0.25 micron (0.00001 inch) apart makes adherence almost impossible. Two surfaces will also adhere when covered with a film of grease or with moisture from the hand. The thickness of these films, however, is a rather indefinite quantity, in most cases about 0.07 micron (0.000003 inch), and while considerable force is required to slide the gages on each other they can be pulled apart by a force of 5 to 10 pounds per square inch. With gages having slight surface imperfections the oil film assists somewhat in holding them together and for the ordinary uses of gages the existence of the oil or grease film introduces no appreciable error, but in making accurate calibration of the gages themselves it should be eliminated.

The results obtained with two different combinations of gages are given in Table 1 to illustrate the effect produced by the film between the gages. The five gages in the first set were brought into contact as described above. The measured length of the combination differed from the sum of lengths of the individual gages by four millionths of an inch. The nine gages in the second set were first brought into contact as described above. In this case the measured length of the combination A differs from the sum of the lengths of the individual gages by six millionths of an

inch. The gages were then separated, rubbed across the wrist, and brought into contact again. The measured length B shows that the length of the combination increased by thirty-four millionths of an inch. This indicates that a film of moisture about three to four millionths of an inch in thickness had been introduced between each pair of gages.

TABLE 1.—Comparison of the Measured Lengths of Gage Combinations

Designation of length	Inches
Gages 0.30+0.25+0.20+0.125+0.120 inch:	
Sum of individual lengths.....	0.995011
Measured length of combination.....	.995015
Gages 0.15+0.25+0.30+0.35+0.40+0.45+0.55+0.60+0.90 inch:	
Sum of individual lengths.....	4.000079
Measured length of combination A.....	4.000073
Measured length of combination B.....	4.000107

VI. TEST FOR PARALLELISM OF SURFACES

The arrangement of the apparatus used to test the parallelism of the surfaces of a standard gage is shown in Fig. 10. Two

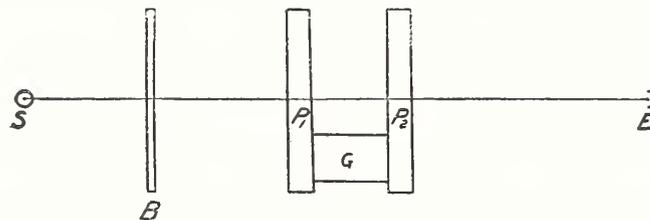


FIG. 10.—Optical system for testing parallelism of gage surfaces

accurately plane interferometer plates P_1 and P_2 have a semi-transparent film of platinum on their inner faces. Near one edge of each a strip of the platinum about one-half inch wide is removed. This clear area on P_1 is brought in contact with one end of the gage, G , and that of P_2 with the other end of the gage. This combination of plates and gage constitutes a Fabry and Perot interferometer. When this interferometer is placed in front of the ground glass screen B illuminated with monochromatic light from the source S , and viewed from E along a line SE perpendicular to the platinized surfaces, concentric interference rings known as "Haidinger rings" are seen. When the eye E is moved in a direction perpendicular to the line of sight SE , the system of rings moves across the plates in the same direction, the center of the system always remaining on the perpendicular from the eye

to the surface. If, now, the interferometer surfaces are parallel to each other, which means that the two gage surfaces which are in contact with them must also be parallel, each ring will retain its original diameter when the eye is moved as designated. If the plates are not parallel, the rings expand on moving opposite points of greater separation and contract as the eye is moved in the direction of smaller separation. Suppose that when the eye is shifted, so that the center of the ring system moves across the plates from one edge to the other, the first central ring expands and takes the place originally occupied by the second (a new ring forming within); then the distance between the two interferometer plates has increased by one-half wavelength, which for yellow helium light is about 0.29 micron (0.000011 inch). If the width of the gage is one-fourth that of the platinized space, this would mean a difference of about 0.07 micron (0.000003 inch) between the gage lengths along opposite sides. Since an expansion of

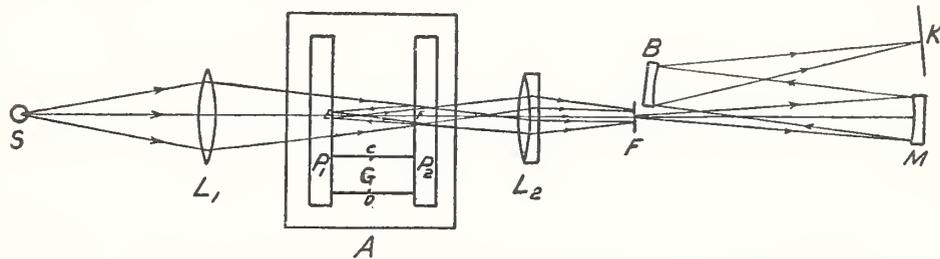


FIG. 11.—Optical system for determining the length of a gage by means of light waves

the rings can be estimated to one-fourth of the diameter of the first ring, an error of 0.025 micron (0.000001 inch) in the parallelism of the gage surfaces can be detected. To realize this precision, however, special care must be taken to have the surfaces of the gage in very close contact with the plates.

VII. CALIBRATION OF END STANDARDS WITH THE FABRY AND PEROT INTERFEROMETER

1. GENERAL PLAN OF APPARATUS

The arrangement of the apparatus used to measure gages in wave lengths is shown in Fig. 11. The interferometer formed by the gage and plates of Fig. 10 is placed within a constant temperature chamber *A* in front of the slit of a grating or prism spectrograph. Light from the neon lamp *S* is focussed by the lens *L*₁ upon the interferometer *P*₁*P*₂. Part of the light is transmitted directly through the interferometer, part is reflected by the

platinized surface of P_2 to the platinized surface of P_1 , where it is again partially reflected, and then a part of it passes on through P_2 , and so on through a large number of multiple reflections and transmissions. The reflected and directly transmitted parts when combined produce a system of interference rings which is focussed by the lens L_2 upon the slit F of the spectrograph. The images of the slit corresponding to the different radiations from neon are separated by the grating B and recorded on the photographic plate K .

2. SOURCE OF LIGHT

The source of light used in this work was an electric discharge tube filled with neon gas and operated on a 10 000 volt ac circuit. Wave lengths between 0.58 and 0.70 micron (0.000023 and 0.000028 inch) were used in making the determinations. The radiations from neon gas are very homogeneous and show good interference with large path differences, making it possible to measure gages 5 cm (2 inches) in length without difficulty. Longer gages can be measured if a telescope is used in making the interferometer adjustments. A limit on the maximum length of gage which can be compared directly with light waves by this interference method is imposed by the fact that no spectral lines are ideally homogeneous or monochromatic. Under ordinary conditions the neon lines fail to show interference when the retardation exceeds 300 000 waves. This permits direct comparison with 10 cm gages. The spectral lines of krypton are sharper and would allow 20 cm gages to be measured by this method.

3. INTERFEROMETER

The interferometer plates were glass disks 42 mm (1.7 inches) in diameter and 8 mm (0.3 inch) in thickness, or glass plates 5 cm (2 inches) square and 1 cm (0.4 inch) thick. These plates were covered with thin platinum films by the method of sputtering from a platinum electrode in a vacuum. Bringing a gage in contact with the platinum film causes the film to scale off, making adherence impossible. To eliminate this difficulty, a strip of the platinum a little larger than the gage surface was removed from each plate and the gage surface brought into contact with the clear glass surface. The gages which formed the separators or étalons for the interferometer plates varied in length from 1 mm to 5 cm and from 0.05 to 2 inches.

4. CONSTANT-TEMPERATURE CHAMBER

Since the length of a gage varies with the temperature, it is necessary to measure and specify the length at 20° C, which has been chosen as the standard temperature for measuring instruments. This was accomplished by placing the interferometer in a chamber *A* surrounded by a thermostated bath previously described.¹³ The temperature of the bath was brought to the desired point and held for a period of at least one-half hour before beginning the photographic exposures in order to allow the gage to assume a steady state.

5. SPECTROGRAPH

Dispersing apparatus of some kind is required in order that length measurements may be made with various colors or spectral lines such as are emitted by luminous neon gas. For this purpose either a prism spectrograph or a diffraction grating may be used, but the concave grating mounted in parallel light with the aid of a mirror has certain advantages in stability, achromatism, and minimum astigmatism over any other type of spectrograph which might be considered as suitable for these measurements.

For most of the work the grating spectrograph of the spectroscopic laboratory was used. *M* is a speculum concave mirror and *B* a concave grating. The mirror and grating each have a radius of curvature of about 640 cm. (21 feet). The grating was ruled by Dr. Anderson and has 39 800 lines on a distance of 13.2 cm. (5.2 inches). The linear dispersion at *K* with this apparatus is about 10 Å per millimeter in the spectrum of the first order.

6. PHOTOGRAPHIC

The spectrum of neon between the wave length limits 0.58 and 0.70 micron (0.000023 and 0.000028 inch) was recorded on Seed 27 photographic plates after staining with pinacyanol.¹⁴ The exposure times ranged from 5 to 10 minutes. Since the slit was illuminated by radiations, each of which produced a system of interference rings, the photograph of the spectrum shows the images of the slit crossed by arcs of the ring systems. The diameters of the interference rings were measured by means of a micrometer screw with a head graduated to 5 microns. By substituting a micrometer eyepiece for the photographic plate visual observations could be made on the diameters of monochromatic

¹³ Meggers and Peters, B. S. Sci. Papers, No. 327, p. 713; 1918.

¹⁴ Meggers and Stimson, J. Op. Soc. Am., 4, No. 3; 1920.

fringes, but a photographic impression of the interference phenomena is generally to be preferred.

7. DETERMINATION OF THE ORDER OF INTERFERENCE

Neglecting here any difference in phase change by reflection at the surfaces (treated in section 9) the thickness of the air space between the two interferometer plates is given by the equation

$$\begin{aligned} t &= \frac{N\lambda}{2} \\ &= (n + n')\frac{\lambda}{2}, \end{aligned} \quad (9)$$

where N is the order of interference at the center of the ring system, n that of the first ring, and hence n' the fraction difference in order between the first ring and the center of the system. In carrying out the computations n' is calculated as the fraction part of the order by which any given ring differs from N . The formula for calculating this is given by Meggers¹⁵

$$n' = \frac{n\omega^2 d^2}{8r^2}, \quad (10)$$

where d is the diameter of any given ring image, r the corresponding length of the image of a stop placed across the slit, and ω the angle subtended by this stop at the lens, L_2 , which projects the ring system on the slit. It is obvious that the approximate value of n obtained by using the nominal length of the gage which separates the plates is sufficient for calculating the value of n' .

The exact value of n is obtained as follows: If T be the nominal length of the gage, the approximate value of n is, for any given wave length,

$$n_1 = \frac{2T}{\lambda_1}.$$

From this

$$n'_1 = \frac{n_1\omega^2 d^2}{8r^2},$$

which, together with n_1 , gives a tentative value T_1 for the thickness. This value T_1 is then used to calculate the trial value $N_c = n_c + n'_c$ of the order for each wave length used. From these approximate values of n and the measured diameters of the rings, n' for each wave length is obtained by substitution in (10). We

¹⁵ Meggers, B. S. Sci. Papers, No. 251; 1915.

now have two values for the fraction part of the order—one, n' , obtained from the measured diameter of the rings, and the other, n'_c , from the trial thickness of the gage. If these magnitudes coincide for the various wave lengths within the limits of experimental accuracy, it shows that n_1 has been correctly chosen. Otherwise n_1 must be corrected by some whole number, y , which may be determined as follows:

Let $R = \frac{\lambda_1}{\lambda}$ be the ratio of the wave length used for obtaining the trial value to the wave length being used to correct it. Since changing n_1 by the whole number y changes N for a wave length λ by Ry , and hence n' by $(1 - R)y$, we have the difference between the observed and calculated values

$$n' - n'_c = (1 - R)y,$$

from which

$$y = \frac{n' - n'_c}{1 - R}.$$

This serves to calculate for λ_1 a value t_1 for the thickness, which is subject only to the errors of measurements, not to the inaccuracy in the nominal length of the gage.

$$t_1 = \frac{\lambda_1}{2} (n_1 + y + n'_1).$$

From this corrected thickness the order of the first ring is readily computed for each wave length. These values of n and the computed fraction n' are then substituted in (9). Each wave length thus furnishes independently a value for t , from which the mean value is finally obtained.

8. CORRECTION OF λ

The wave lengths which are given for standard conditions of 15° C and 760 mm pressure vary with changes in the density of the air. The wave lengths must therefore be corrected to the existing temperature and pressure conditions. Since this correction and the dispersion of the air are small, it is sufficient to apply the correction factor

$$f = \frac{\mu_{15}}{\mu_t}, \text{ where } \mu_t = 1 + \frac{(\mu_{15} - 1) P}{760 T},$$

to the mean value of the thickness t , given above, to obtain the optical thickness t_p of the space at EF between the two platinum films.

9. CORRECTION FOR THE THICKNESS OF THE PLATINUM FILMS

With unplatinized interferometer plates the distance t_g (at EF , Fig. 11, between the two glass surfaces as computed above from measurements of the rings would be the true distance between the plates, because the phase change of π due to the two air-glass reflections is eliminated in the determination of n . With the platinized surfaces, however, t_g differs from t_p by a small correction $x = t_g - t_p$, which is equal to the thickness of the films plus or minus the change in the optical path due to the difference in phase change for air-glass and air-platinum reflections. To measure x , a glass test plate is placed over each interferometer plate so as to cover both the clear and platinized parts. Each gives a system of fringes arising from the interference between the front and back surfaces of the thin air films inclosed. The system over the platinized area, owing to the combined effect of difference in thickness of the air film and difference in phase change by reflection, is displaced (less than one order) relative to the system from the clear portion. Multiplying by $\frac{\lambda}{2}$ the combined displacement resulting at the two plates, when tested with the light in the same direction as under working conditions, gives the correction x to be applied to the thickness of the gage as calculated in section 8.

10. CORRECTION FOR NONPARALLELISM OF THE GAGE SURFACES

If the gage surfaces are parallel, the interferometer plates will be parallel, and the length L of the gage is

$$L = t_g = t_p + x.$$

If the gage surfaces are not parallel, a correction must be applied to t_g to obtain L . Having the difference $z = L_C - L_D$ in the length of the gage along the two opposite sides C and D , Fig. 11, obtained as explained in section VI, and making the distance from EF to the gage at C equal to the width of the gage, the length along C is obviously

$$L_C = t_g - z,$$

and along D is

$$L_D = t_g - 2z.$$

11. COMPUTATIONS

An illustration of the method for making the computations for a 25.4 mm (1 inch) gage is shown in Table 2. Column 1 gives the wave lengths used; 2, the measured diameter d of the first and second rings, respectively; 3, the fractional part n' of the order at the center of each ring system; 4, the order of interference N or number of waves in the double distance between the plates at the center of the ring $N = n + n'$; and 7, the double optical thickness of the air film or $N\lambda$.

TABLE 2.—Computing Method for a 25.4 mm Gage

1 λ in Angstrom units	2 ^a d	3 d^2	4 $\frac{d^2 \omega^2}{8r^2}$	5 n'	6 N	7 $2t$ in microns
5852.488.....	455 676	207 457	93 205	0.807 1.779	86 802.793	50 801.2304
5944.834.....	341 602	116 362	52 162	.444 1.384	85 454.414	50 801.2306
6096.163.....	551 746	304 557	136 205	.133 1.083	83 333.108	50 801.2210
6143.062.....	495 713	245 508	110 228	.910 1.885	82 693.898	50 801.2254
6334.428.....	409 667	167 445	75 199	.601 1.596	80 199.599	50 801.2251
6506.528.....	295 612	87 375	39 168	.304 1.312	78 077.308	50 801.2191
6678.276.....	344 631	118 398	53 178	.403 1.354	76 069.379	50 801.2308
$f=1.0000102$						
Mean value of $2t$						50 801.2261
Corrected for density of air, $2t \times f = 2t_p$						50 801.7442
Correction for film thickness, x						25 400.8721 .0701
Distance between glass plates, t_g						25 400.9422
Correction for slant of gage surface, $-z$0367
Length of gage at C, L_c						25.4009789 mm
Length of gage at D, L_D						1.0000365 inch 25.4010156 mm 1.0000380 inch

^a In columns 2 to 5, the figures in each group of two refer, respectively, to the first and second rings.

12. ACCURACY AND SOURCES OF ERROR

A comparison of the values obtained for the thickness of the air film, Table 2, column 7, shows that the maximum variation of t for the several wave lengths is about one part in five million when using a 1-inch separator. The correction for the change in λ arising from a change in density of the air is about one part in one hundred thousand. With the accurate values of the refractive index of air¹⁶ available, this correction should not introduce an

¹⁶ Meggers and Peters, B. S. Sci. Papers, No. 327; 1918.

error of one part in ten million in the total length. The combined thickness and phase difference effect of the platinum films is about 0.07 micron (0.000003 inch), and the probable error in measuring this thickness about 0.003 micron (0.000001 inch), which would introduce an error in the value of the distance between the plates of about one part in ten million. Therefore, using a 25 mm (1 inch) étalon and making measurements with several wave lengths, the value of the distance t_g between the glass surfaces at EF should be correct relative to the International meter to about 1 part in four or five million.

Transferring this measured distance to the gage introduces the greatest error. With a perfect gage and perfectly plane interferometer plates the only sources of error are the films between the gage surfaces and the plates. If the surfaces are thoroughly cleaned and carefully brought into contact, the separation is less than 0.025 micron (0.000001 inch). It is probable that slight projections on the surfaces are in intimate contact and that the liquid film exists in the intervening spaces. Therefore, with carefully controlled conditions, the error in the measurement of perfect gages should be less than 0.025 micron (0.000001 inch).

In actual practice, however, most interferometer plates obtainable deviate from true plane by about 0.05 micron (0.000002 inch), and the surfaces of carefully selected gages deviate from true plane and parallelism by about 0.13 micron (0.000005 inch) on the average. Therefore, in transferring t_g to the gage the error introduced depends largely upon imperfections of the gage. With carefully selected gages the errors in the values of the length along the C and D sides range from 0.025 to 0.075 micron (one to three millionths inch).

The measurements made on one of our standards (1-inch, set 7) shown in Table 3 are representative of the values obtained for several hundred other standards. Each value given is the result of a single determination made after a separate assembling of the interferometer and adjustment of the spectrograph. The first determination of January 11, 1921, was made with the grating spectrograph shown in Fig. 11. The second determination of that date was made with a different pair of interferometer plates and a prism spectrograph.

TABLE 3.—Length of 1-Inch Gage of Set 7 at 20° C

Date	Length	Date	Length
	Inches		Inches
Feb. 4, 1918.....	1.0000008	Feb. 7, 1918.....	0.9999992
Feb. 7, 1918.....	.9999996	Mar. 17, 1920.....	.9999983
Do.....	.9999990	Jan. 11, 1921.....	.9999974
Do.....	.9999993	Do.....	.9999968

Another very important source of error that may be overlooked is the thermal expansion of the material. A 25 mm (1-inch) length of steel expands about 0.32 micron (0.000013 inch) per degree Centigrade rise in temperature. The gage must therefore be held at the constant temperature of 20° C within a few hundredths of a degree. If the measurements are made at any other temperature, this must be accurately measured and the expansivity of the material known in order to reduce the length to standard conditions. We have found expansion coefficients ranging from 0.000011 to 0.000013 for gage steels of various compositions, hardness, and previous heat treatment. This shows that it is unsafe to assume a value for the expansivity when measuring the absolute length of a gage or comparing an unknown gage with a standard at a temperature that differs very much from 20° C.

TABLE 4.—Thermal Expansion of Several Precision Gages

Gage	Temperature interval	Coefficient of expansion $\times 10^4$	Gage	Temperature interval	Coefficient of expansion $\times 10^4$
	°C			°C	
Johansson, set 5813, 10 mm..	20 -50	0.129	Bureau of Standards:		
Do.....	20 -50	.129	Steel A, 0.4 inch.....	24.0-76.9	0.132
Johansson, set 5813, 9 mm..	20 -50	.125	Steel B, 0.4 inch.....	33.0-82.8	.129
Do.....	20 -50	.125	Pratt & Whitney, 0.375 inch..	21 -73	.135
Johansson, set 20, 0.4 inch...	19.8-75.5	.124	Schuchardt and Schutte, 0.5		
Do.....	32.8-76.5	.123	inch.....	5.8-46.0	.116
Johansson, set 7, 0.4 inch...	56.3-79.6	.131	Do.....	5.8-46.0	.115
Do.....	19.6-79.6	.132			
Johansson, set 7, 0.35 inch...	21.3-82.4	.128			
Do.....	20.6-82.4	.127			

In Table 4 is shown the thermal expansion of several precision gages. These measurements were made with the interferometer and electrical furnace previously described in our publication ¹⁷

¹⁷ Peters and Cragoe, B. S. Sci. Papers, No. 393; 1920.

on the dilatation of optical glass. Column 1 gives the designation of the gage under investigation; column 2, the temperature interval; and column 3, the coefficient of expansion.

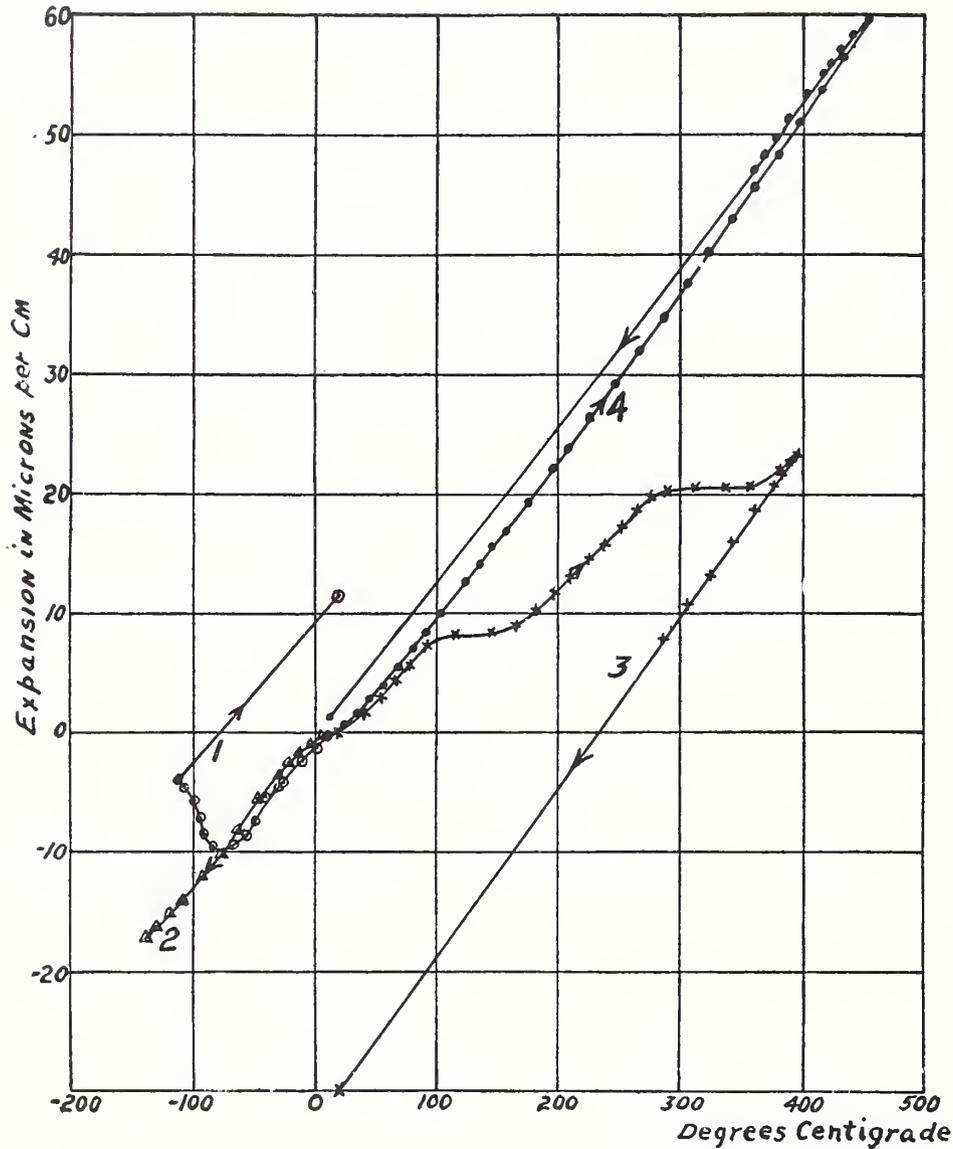


FIG. 12.—Curves showing the thermal expansion of the steel used for precision gages

The composition of steel B which is used for precision gages at the Bureau of Standards is C, 1.00 to 1.10 per cent; Mn, 0.30 to 0.40 per cent; P, 0.025 per cent; S, 0.025 per cent; Si, 0.20 to 0.30 per cent; Cr, 1.30 to 1.50 per cent; balance, Fe. This is almost identical with the compositions that have been published

for the steels used in the Johansson and the Pratt & Whitney gages.

The thermal expansion of a gage about 1 cm long and 2 cm in diameter made from steel B is shown in Fig. 12. It was hardened by heating to 850° C and quenching in oil. The original hardness was 95 as measured with a scleroscope, and length was 11.024 mm. The sample was first cooled to -114° C, the change in length being represented by curve 1. On returning to room temperature the hardness was 94.5 and the length 11.035 mm; that is, the length had increased 12 microns due to this treatment. After cooling again to -135° C, represented by curve 2, and returning to room temperature the hardness was 94 and length 11.035 mm. No further change in length took place. The sample was then heated to 392° C and allowed to cool to 20° C, change in length being represented by curve 3. The final hardness was 76 and length 11.004 mm. This treatment caused a permanent shortening of about 31 microns. The sample was then heated to 450° C and allowed to cool to 20° C. The change in length is represented by curve 4.

TABLE 5.—Changes of Length with Time

Gage	Date	Length	Gage	Date	Length
		Inches.			Inches.
1.000 inch.....	Mar. 1, 1919	1.000000	2.000 inches.....	Mar. 19, 1920	1.999993
	June 3, 1919	.999998		May 22, 1920	1.999992
	July 19, 1919	.999992		3.000 inches.....	Feb. 20, 1919
	Aug. 19, 1919	.999992	Apr. 28, 1919		2.999975
	Oct. 16, 1919	.999993	Aug. 20, 1919		2.999900
	Mar. 20, 1920	.999988	Sept. 19, 1919		2.999877
	May 21, 1920	.999988	Mar. 18, 1920	2.999856	
1.000 inch.....	May 21, 1919	1.000000	May 21, 1920	2.999851	
	May 29, 1919	1.000018	4.00 inches.....	Apr. 29, 1919	4.000000
	June 17, 1919	1.000022		June 2, 1919	3.999998
	Aug. 19, 1919	1.000034		July 19, 1919	3.999992
	Oct. 17, 1919	1.000038		Aug. 19, 1919	3.999980
	Jan. 7, 1920	1.000043		Oct. 16, 1919	3.999980
	Mar. 20, 1920	1.000044		Mar. 18, 1920	3.999971
May 22, 1920	1.000047	May 21, 1920		3.999970	
2.000 inches.....	Mar. 1, 1919	2.000000	4.00 inches.....	May 7, 1919	4.000000
	May 29, 1919	1.999976		June 3, 1919	4.000014
	July 19, 1919	1.999966		Aug. 20, 1919	4.000025
	Aug. 19, 1919	1.999963		Nov. 26, 1919	4.000021
	Oct. 16, 1919	1.999951		May 21, 1920	4.000025
	Mar. 18, 1920	1.999938			
	May 21, 1920	1.999935	4.00 inches.....	Dec. 22, 1919	4.000000
2.000 inches.....	Sept. 24, 1919	2.000000		Mar. 18, 1920	3.999999
	Oct. 16, 1919	2.000002		May 21, 1920	3.999993
	Jan. 9, 1920	2.000000			

Unless the material from which the standards are made is exceedingly stable the high accuracy of the length determination is soon lost due to changes in the length with time. While we have found many steel gages that retain their dimensions remark-

ably well, others have been found to change several microns—or hundred thousandths of an inch—in a few months. A few examples of gages that have undergone changes of length with time are given in Table 5. The fact that these changes may take place requires frequent intercomparisons and redeterminations of the gages used as standards.

VIII. COMPARISON OF GAGES WITH STANDARDS

1. COMPARISON OF LENGTH

The accurate comparison of two gages, *A* and *B*, Fig. 13 (front), of supposedly the same length, is made by the following method:

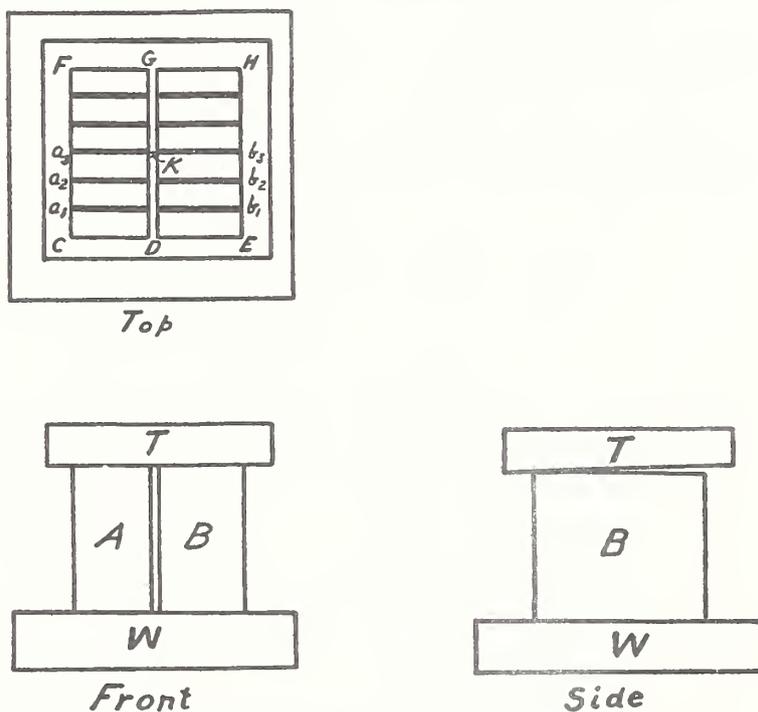


FIG. 13.—Intercomparison of two similar gages of equal length

The two gages are placed side by side in intimate contact, as described in *V* above, with the true plane surface of a glass plate *W*. The lower surfaces of the two gages being in the same plane, the problem is then simply to determine the distance between the planes of the two upper surfaces. This is done by placing a test plate *T* in contact with the gages along the line *CDE*, Fig. 13 (top), and somewhat inclined to these surfaces. This gives side by side two thin wedge-shaped films. When illuminated and viewed as shown in Figs. 3 or 9, two sets of straight fringes parallel

to the edge of the wedge are seen; see top view, Fig. 13. Since it is only necessary then to determine the difference in thickness of these films at some adjacent position, say K , we can assume zero phase changes at the surfaces and calculate the distance at that point between the planes of the two gage surfaces. If the two gages are of the same length, their upper surfaces will lie in the same plane, so when we pass to the thicker part of the wedge from the line of contact CDE , the first fringe a_1 on A will coincide with the first fringe on b_1 on B , the second with the second, etc.

Consider the point K , where there is coincidence between the third fringes on the two gages. For perpendicular view the optical thickness of the film at any point is given by equation (3)

$$t = \frac{N\lambda}{2}$$

In this case the distance t from the test plate to each gage would be $\frac{3\lambda}{2}$ at K , which means that the gages are of equal length.

Suppose the gages are unequal in length, say B is shorter than A . The test plate will then come in contact with A at the point D and with B at the point E and the fringes appear, as in Fig. 14. If at K , we have the second fringe over A coinciding with the fourth over B , the distance at that point between the test plate and A is $\frac{2\lambda}{2}$ and between the test plate and B is $\frac{4\lambda}{2}$. Therefore, the distance between the planes of the two gage surfaces is $\frac{2\lambda}{2}$. If

we are using a helium lamp for a source, $\frac{\lambda}{2}$ is about 0.3 micron (0.000012 inch), hence B is about 0.6 micron (0.000024 inch) shorter than A . If A is a calibrated standard, we immediately have the length of the unknown gage B . By estimating the displacement of the fringes to one or two tenths of the distance between two bands measurements of still greater refinement can be made. In making these measurements it is absolutely essential that the fringes be viewed with the Pulfrich or other instruments previously referred to, or as shown in Fig. 9; that is, normally to

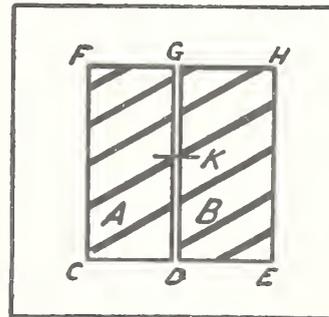


FIG. 14.—Intercomparison of two similar gages of slightly different length.

the gage surfaces. If they are viewed at an angle to the perpendicular, then the thickness is not equal to $\frac{N\lambda}{2}$, but equal to $\frac{N\lambda l}{2P}$, so that an incorrect interpretation of the distances is made.

When observing with the unaided eye, as shown in Fig. 9, it is not advisable to make comparisons between gages that differ in length by more than five or six wave lengths. With the aid of the Pulfrich instrument, however, comparisons between gages that differ in length as much as $\frac{1}{2}$ inch may be made. With this method the fractional orders n' at the point K over each gage are measured for several different radiations of helium gas. The orders of interference N for each wave length are then computed from these fractions and the approximate distance between the test plate and the upper surface of each gage by the method described in Section VII, which holds for straight fringes as well as for Haidinger rings.¹⁸

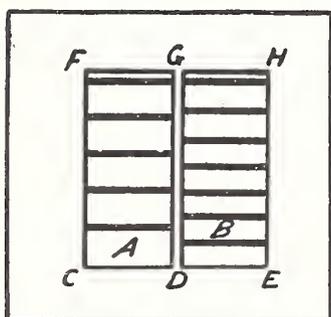


FIG. 15.—Test for parallelism of gage surfaces. B shorter at GH than DE

The absolute length of a gage is found in the same way. The gage is brought in contact with a steel base plate and the thicknesses of the air columns over the gage and base plate, respectively are measured. The distance between the test plate and the base plate minus the distance from the test plate to the gage gives the length of the gage. The steel base plate is used in order to eliminate any difference in phase losses on reflection. This method was used in calibrating most of the end standards less than 5 mm ($\frac{1}{2}$ inch) in length, because the operation of making the measurement and computations requires only about one-third the time consumed when using the circular fringe method.

2. COMPARISON OF PARALLELISM OF SURFACES

The test for parallelism of the two surfaces of an unknown gage B is made along with the length comparison. Assuming that the two surfaces of the standard gage A , Fig. 15, are plane and parallel, the test plate brought in contact with it along CD gives straight fringes over A which are parallel to CD and equally spaced. If the upper surface of the gage B is parallel to the plane of the upper surface of A , the fringes over B will be parallel to those over

¹⁸ Peters, J. of Wash. Acad. of Sci., 9, p. 282; 1919.

A and have the same spacing. If, however, the length of *B* at *GH* is less than at *DE*, then the wedge over *B* will be steeper than over *A* and the fringes closer together. Therefore, if as indicated 7 fringes are observed over *B* and 5 over *A*, *GH* is $\frac{2\lambda}{2}$ below *DE*, or the gage is about 0.000022 inch shorter on the *GH* side than on the *DE* side. If the length of *B* at *GH* is greater than at *DE*, then the wedge over *B* will be thinner than over *A* and the fringes farther apart. If the surfaces of the standard *A* are not parallel, then to determine the true slant or lack of parallelism of the surfaces of *B* along *DG*, the apparent slant is determined as described above. Letting A_{FG} , B_{GH} , etc., also denote the lengths of the gages along those sides, we have $B_{GH} - B_{DE} = s$. If now from other sources it is known that the slant of *A* is $A_{FG} - A_{CD} = a$, then the true slant of *B* is

$$b = a + s, \tag{11}$$

a, *b*, and *s* being positive or negative, as the case may be.

Suppose, as in Fig. 16, that the edge *GD* of *B* is parallel to the plane of the upper surface of *A*, but the surface of *B* slopes slightly, so that *HE* is above *GD*. Since the fringes lie along lines of equal thickness of the air film, they will extend across *B* at an angle to those over *A*, being deflected toward the open end of the film or toward *H*. If *HE* is below *GD*, the fringes on *B* will be deflected toward the thin edge of the film or toward *E*, as in Fig. 17. If we draw a line *KL* parallel to *CE* from the left end of any fringe over *B*, the displacement of the other end of that fringe

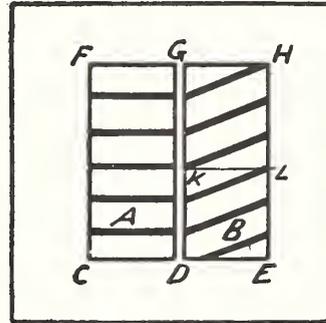


FIG. 16.—Test for parallelism of gage surfaces. *B* longer at *HE* than at *GD*

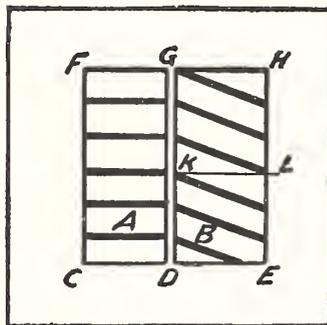


FIG. 17.—Test for parallelism of gage surfaces. *B* shorter at *HE* than at *GD*

from *KL* divided by the distance between two consecutive fringes gives the difference in height between *GD* and *HE* in half wave lengths. This difference gives the slant between the upper surface of *A* and the upper surface of *B*. If the two surfaces of *A* are perfectly parallel, it is also the slant between the two surfaces of *B*. If the

two surfaces of A are not parallel, but the slant $A_{GD} - A_{FC} = c$, using the same expression for length as above, then the true slant of B is equal to the observed apparent slant $B_{DG} - B_{HE} = d$ minus kc , where k is the ratio of the widths of the gages B and A ; that is,

$$f = d - kc. \tag{12}$$

The slant of the two surfaces of B can also be determined by measuring the perpendicular distances between them, say, at the middle points of all four edges of B , by bringing them successively contiguous to A at the point K .

When determining the length of a gage or standard with the Pulfrich instrument, the fringes over the gage should be parallel to and equally spaced with those over the base plate. Any deviation from such a condition signifies that the gage surfaces are not parallel, in which case the amount of nonparallelism may be determined by the method just described on making a and c equal to zero in the formulas (11) and (12).

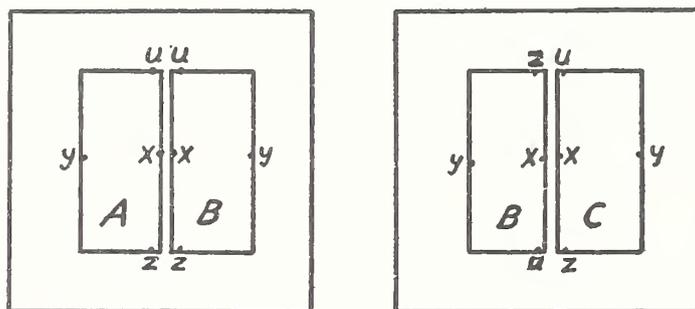


FIG. 18.—Test for relative length of gages and parallelism of the surfaces by the intercomparison of three similar gages

IX. INTERCOMPARISON OF THREE GAGES

The lack of parallelism of the two faces at each of three unknown gages, A , B , and C , can be accurately determined and an intercomparison between their lengths made by bringing them in contact with the base plate two at a time. Let A_x , B_x , C_x , A_y , etc., denote the lengths of the gages at the points x , y , z , u , indicated in Fig. 18, and

$$\begin{aligned} A_K &= A_u - A_z, & A_S &= A_x - A_y, \\ B_K &= B_u - B_z, & B_S &= B_x - B_y, \\ C_K &= C_u - C_z, & C_S &= C_x - C_y, \end{aligned}$$

represent, respectively, the slants of the gages A , B , and C in the two perpendicular directions uz and xy . If the gages A and B are brought into contact with the base plate, so that A_u and B_u ,

A_x and B_x , and A_z and B_z are adjacent, as in Fig. 18 (left) (gage A face up and B face down), then one can make the comparisons between the lengths of the gages:

$$\begin{aligned} A_u - B_u &= r,^{19} \\ A_z - B_z &= s, \\ A_x - B_x &= l, \end{aligned}$$

and observe the apparent slant $B_x - B_y = a$ on B . Gage B can then be replaced by C and a similar comparison made

$$\begin{aligned} A_u - C_u &= t, \\ A_z - C_z &= q, \\ A_x - C_x &= m, \\ C_x - C_y &= b \text{ (apparent slant)}. \end{aligned}$$

Then B replaces A , so that B_z and C_u , B_u and C_z , and B_x and C_x are adjacent (both gages face down), Fig. 18 (right). The final comparisons to be made are:

$$\begin{aligned} B_z - C_u &= v, \\ B_u - C_z &= w, \\ B_x - C_x &= n, \\ C_x - C_y &= c \text{ (apparent slant)}. \end{aligned}$$

Solving these twelve equations:

$$(1) \quad m - l = n$$

showing that this method gives a comparison of the lengths of the gages at the corresponding x -points, and a check on this comparison.

$$\begin{aligned} (2) \quad A_K &= r - q + w, \\ &= t - s - v, \\ B_K &= A_K - r + s, \\ C_K &= A_K - t + q, \end{aligned}$$

giving two determinations of the slant of each gage in the direction uz , the mean of which may be taken as the true value with an error of one-half an algebraic combination of the errors of the six measurements.

¹⁹ The small letters represent the measured quantities.

(3) If B and C are f and h times as wide as A , respectively, then C is $\frac{h}{f}$ times as wide as B and

$$\begin{aligned} B_s &= a - f A_s, \\ C_s &= b - h A_s, \\ C_s &= c - \frac{h}{f} B_s, \\ A_s &= \frac{f(b-c) + ah}{2fh}, \\ B_s &= \frac{f(c-b) + ah}{2h}, \\ C_s &= \frac{h(b+c) - ah}{2f}. \end{aligned}$$

If the gages are all the same width, then $f = h = 1$ and

$$\begin{aligned} A_s &= \frac{b-c+a}{2}, \\ B_s &= \frac{c-b+a}{2}, \\ C_s &= \frac{b+c-a}{2}, \end{aligned}$$

showing that the error of each determination is one-half the algebraic combination of the individual errors. Thus, the method of intercomparing three gages enables one to determine easily and accurately the relative merits of each, their planeness errors having been previously determined, so that the best one can be chosen as a secondary standard of length and the others as working standards for use in the laboratory.

X. DEVELOPMENT OF STANDARD GAGES

Having established the fact that two plane surfaces can be brought into contact, so that the separation is less than two hundredths of a micron (one millionth of an inch), and having the interference method for comparing two gages of nearly equal length, it is possible to calibrate long gages from line standards and make comparisons between these long gages and equal combinations of two or more shorter ones. The arrangements used by Fischer²⁰ and Perard²¹ for comparing a long end standard with a line standard are shown in Fig. 19. Two gages, A and B , are brought into close contact, and two fine lines, C and D , are ruled

²⁰ Phil. Soc. Wash., Bull., 13, p. 241; 1898.

²¹ C. R., 154, p. 1586; 1912.

on them parallel to their plane of contact, EF . The distance X between the lines C and D is determined by comparison with the line standard. A is then brought into contact with one surface of the long gage G which is to be calibrated and B with the opposite surface of G . The distance Y between the two lines C and D is

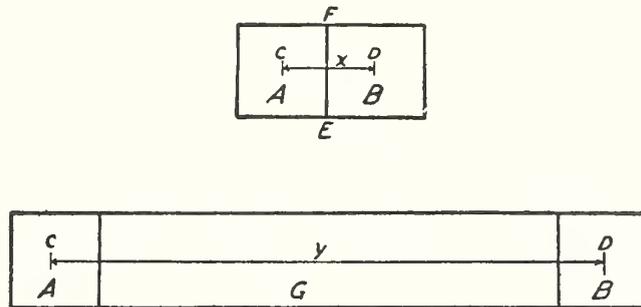


FIG. 19.—Calibration of end standards relative to line standards

again determined by comparison with the line standard. The difference in the two distances $Y - X$ gives the length of the gage G in terms of the line standard.

After the length of G has been accurately determined by comparison with the line standard or by direct measurement with the light waves, combinations of shorter gages can be compared with it by using the interference comparator, as follows: Suppose G is 6 inches long and we have three gages, A , B , and C , each very nearly 3 inches in length. G is brought into contact with the plane base plate W , Fig. 20, B is also placed in contact with W , and A with the upper surface of B . The difference a in the lengths of G and the combination of A and B is obtained from the relative displacement of the interference fringes as described in section VIII. The combined length of A and B is equal to that of G plus a . In the same way the observed difference b between the combined lengths of B and C and that of G is obtained. Likewise c for gages C and A . Letting the designation of the gages also represent their lengths, we have

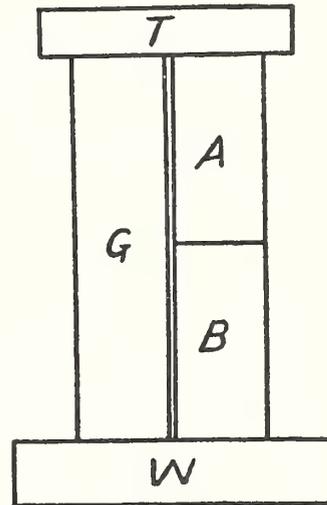


FIG. 20.—Comparison of two short gages with one long gage

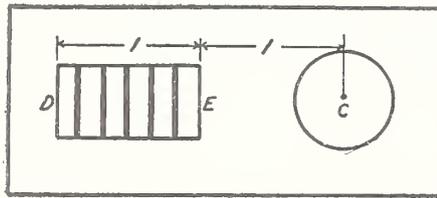
$$\begin{aligned} A + B &= G + a, \\ B + C &= G + b, \\ C + A &= G + c, \end{aligned}$$

where G is the known length of the standard and $a, b,$ and c are measured. These equations can be readily solved for the unknowns, $A, B,$ and C . Similarly, with four gages, $A, B, C,$ and $D,$ each nearly 2 inches long, we would have

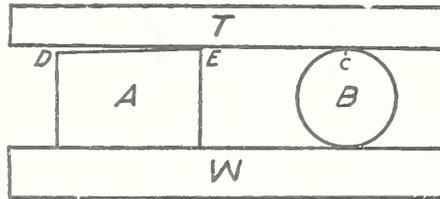
$$\begin{aligned} A + B + C &= G + a, \\ B + C + D &= G + b, \\ C + D + A &= G + c, \\ D + A + B &= G + d. \end{aligned}$$

Since the four independent simultaneous equations contain four unknowns, the length of each unknown gage can be computed.

In general, given $n + 1$ unknown gages of nearly equal length, n of which when combined are nearly equal to the known gage $G,$



there will be $n + 1$ combinations which may be compared with $G.$ Hence, the length of each unknown gage can be obtained by this comparison method. Intermediate sizes may be measured by comparing the combined lengths of a known and unknown gage with a known.



XI. COMPARISON BETWEEN GAGES AND OTHER OBJECTS

FIG. 21.—Comparison of a sphere with a gage block

An accurate determination of the dimensions of any body, say a sphere, can be made by comparison with a gage of nearly the

same size. For this the gage A and the sphere B are placed in contact with the base plate $W,$ Fig. 21, and the test plate T laid over them. If B is slightly smaller than $A,$ the test plate will touch the gage along the edge E and the sphere at the point $C.$ When illuminated and viewed as shown in Fig. 3 or 9, straight fringes parallel to E will be seen to cross the upper surface of the gage. The number of fringes N across the face of the gage from D to E multiplied by $\frac{\lambda}{2}$ gives the distance between the cover plate and the gage surface at $D.$ If the distance CE is equal to $DE,$ then the point C must be $\frac{N\lambda}{2}$ below the plane of the surface of the

gage, and hence the diameter of the sphere $\frac{N\lambda}{2}$ less than the length of the gage. If B is larger than A , the upper plate will touch the gage along the edge D and be $\frac{N\lambda}{2}$ above E . C would then be $\frac{2N\lambda}{2}$ above the plane of the surface of the gage and the diameter of B equal to the length of the gage plus $N\lambda$.

XII. SUMMARY

The extensive use of precision gages as reference standards for precise mechanical work has required more accurately determined end standards and more rapid and precise methods for comparing gages with these standards than have been previously available. Since comparison of end standards with line standards by means of micrometer-microscopes and of precision gages with end standards by means of contact instruments are subject to appreciable errors, methods which make use of the interference of light waves were used in making these measurements. With the interference methods described in this article, the planeness and parallelism errors of precision surfaces can be measured and the length of standard gages determined by direct comparison with the standard light waves with an uncertainty of not more than a few millionths of an inch. The errors of other gages can be determined by comparison with these calibrated standards with equal precision. This process makes the standard light waves which have been determined to one part in four or five million relative to the international meter, the standards of length for this work. Three sets of gages each containing 81 blocks were selected for end standards. These were tested for surface errors and calibrated by comparison with the light waves and have been recalibrated every few months because changes with wear or time are known to occur in material gages. Large numbers of precision gages have been compared with these end standards with sufficient speed and precision to meet all requirements.

WASHINGTON, May 3, 1921.

Assembled Polygon for the Calibration of Angle Blocks

Clyde E. Haven and Arthur G. Strang

A method is described for constructing and calibrating an assembled, multiple-sided, angular standard of exceptional accuracy. Although designed as a master for the 30- and 45-degree angle blocks of a series made in this country, the polygon is equally suitable for the test or calibration of circular dividing equipment. Simpler forms may be easily and economically constructed as masters for a variety of applications in the mechanical and optical industries.

1. Introduction

The advent of World War II produced an acute shortage of precise angular dividing equipment in England and led to the development by the National Physical Laboratory of a series of solid angular standards.¹ These are hardened, ground, and lapped steel blocks about $\frac{5}{8}$ in. wide, 3 in. long, and having specified angles between the two contact surfaces. Like gage blocks, they can be combined with little error by wringing, but since they may be combined to form the sum or difference of a pair, a series of as few as 12 blocks will form any angle up to a little over 80 deg. in increments of 3 sec. By mounting these blocks and a solid square (a rectangular prism having four finished faces), in various positions, any subdivision of a circle to the nearest 3 sec. may be obtained.

The angles of the individual blocks in the NPL set are, in general, based on the mathematical relationship whereby each successively larger block is twice the sum of the preceding blocks plus 1, respectively, in seconds, minutes, and degrees. Exceptions to the general relationship are the omission of a 1-sec. block, and the use of a 41-deg. block instead of an 81-deg. block. A 1-sec. block is unwarranted in a set having permissible errors of 1 sec., and the 41-deg. block provides a more convenient size than the 81-deg. block. The series is thus comprised of blocks having angles of 3, 9, and 27 sec., 1, 3, 9, and 27 min., and 1, 3, 9, 27, and 41 deg.

At about the same time that the English series of blocks was developed, a similar series was patented in this country.² The American series differs from the English in that it is made up of more commonly used sizes and thus requires a few more blocks for the same angular coverage. The series is 1, 3, 5, 20, and 30 sec.; 1, 3, 5, 20, and 30 min.; 1, 3, 5, 15, 30, and 45 deg.

Because the generation of nearly optically plane surfaces and the control of pyramidal error within the required limits are not excessively difficult, it is possible to produce blocks that are susceptible to very precise calibration.

One of the most satisfactory methods of calibrating angle blocks is by comparison with a master set, provided that the angular values of the masters can

be determined with adequate accuracy. Knoyle³ has described a primary determination of the angles of all the blocks in a set without reference to an external standard. Using an interferometric method for the required comparisons, we have found his procedure entirely satisfactory except for measurements on master blocks or combinations of master blocks having angles of 45 deg or larger and particularly on the 90-deg combination, which is used in the primary determination for the entire series. Alinement of the sides of the blocks in a combination becomes increasingly critical as the angle increases, and some difficulty has been experienced in obtaining satisfactory agreement on 90-deg combinations.

Similar types of alinement difficulties arise in the comparison of 45-deg blocks by interferometric methods wherein two blocks are wrung side by side on an optical flat and the difference in angle established by the difference in fringe count over a specific length on the unwrung surfaces of the blocks. If, for example, the adjacent sides of the two 45-deg blocks are more than 0.00005 in./in. out of parallel along the length of the blocks, interference bands on the two blocks form too large an angle with each other to permit an accurate fringe count. Considering the errors that may accrue in the determination of the angle of 45-deg blocks, and possibly 30-deg blocks, an alternative method of determining their angles is clearly desirable.

The success of the National Physical Laboratory in the calibration of angular-dividing equipment by means of multiple-sided solid angular standards⁴ suggested the possibility of using a 24-sided solid polygon having 15-deg exterior angles between adjacent faces. Such a polygon would permit direct calibration of both 30- and 45-deg blocks. The mechanical requirements for a polygon of the requisite accuracy are quite severe; the defining surfaces must be flat over practically their entire area to one or two millionths of an inch, the surface roughness of the faces must be less than 0.5 μ in. rms with practically no visible scratches perpendicular to the side of the polygon, and the variations in the angle between the defining faces and the axis of the polygon not in excess of 15 sec. Convenience dictates a maximum departure of the angular intervals from nominal value of not more than 10 sec. The con-

³ C. H. Knoyle, NPL combination angle gauges, *Machinery* (London) **77**, 1975 (1950).

⁴ C. O. Taylerson, Testing circular division by means of precision polygons, *Machinery* (London) **71**, 1821 (1947).

¹ British patent 569,002.

² U. S. Patent 2,134,062.

struction of a conventional, solid 24-sided polygon to these limits constitutes a challenge to the best mechanical skill and equipment.

2. Construction of the Polygon

As a substitute for such a formidable undertaking, the possibility of assembling a polygon from more easily fabricated units was considered. The availability of satisfactory defining faces in the form of gage blocks suggested the assembly of modified blocks on a suitable base, using one finished surface of each block to define the polygon. Conventional gage blocks could readily be modified by lapping the side faces plane and parallel, with, of course, due control of the angle between adjacent lapped faces and by providing holes for fastening the blocks to the base. With this type of construction, control of any of the required features of the polygon could be readily accomplished, usually with little effect on the other dimensions.

In order to determine the merits of this method of construction, an experimental polygon was made. The base of the polygon was $\frac{3}{4}$ in. thick and 8 in. in diameter. It was made of oil hardened steel and heat treated for maximum dimensional stability. A $1\frac{3}{4}$ -in.-diameter center hole and appropriate tapped holes in the base provided means for attaching a bushing so that the polygon could be used on centers if desired. In order to keep the diameter of the plate within reasonable limits, the gage blocks were placed in two layers of twelve each.

Twenty-four tapped holes were equally spaced on a circle of a radius such that the chord of a 30-deg sector corresponded to the spacing of the holes in the gage blocks. This permitted the use of the same

screws for fastening both the upper and lower layers of blocks. The tapped holes in the base were counter-sunk and then counterbored to a depth of $\frac{1}{4}$ in. to minimize distortion of the base due to tension of the clamping screws.

After hardening, the base was ground on both sides and then hand-lapped until the error in parallelism of the two surfaces was less than 0.0001 in. and the maximum error in flatness of each surface less than 0.00006 in. A relatively high finish was imparted to one surface in the final lapping to permit wringing the special gage blocks in position.

Modified 0.750-in. rectangular gage blocks were obtained from a gage-block manufacturer for use as the defining faces. All four lapped sides were plane to 0.000005 in. or better, and adjacent sides were square to 2 sec of arc. The surface roughness of each of the sides was less than 1 μ in. rms. The thickness ($\frac{3}{8}$ -in. dimension) of the 24 blocks in the group varied less than 0.00003 in. Each block was measured and the bottom layer of blocks so selected that adjacent blocks did not differ in thickness by more than 0.00001 in. This permitted at least partial wringing of the blocks in the upper layer to their supporting blocks and reduced distortion arising from the pressure of the clamping screws.

Commercial gage blocks are frequently used as mirrors for an autocollimator, and as such form reasonably sharp images of the reticle. It was found, however, that the crispness of the image could be further enhanced by a limited amount of polishing on a cloth lap, and each of the defining faces was so finished. Figure 1, illustrating the finish before and after polishing, was made with the Ziess micro-interferometer.

The assembly of the components of the polygon

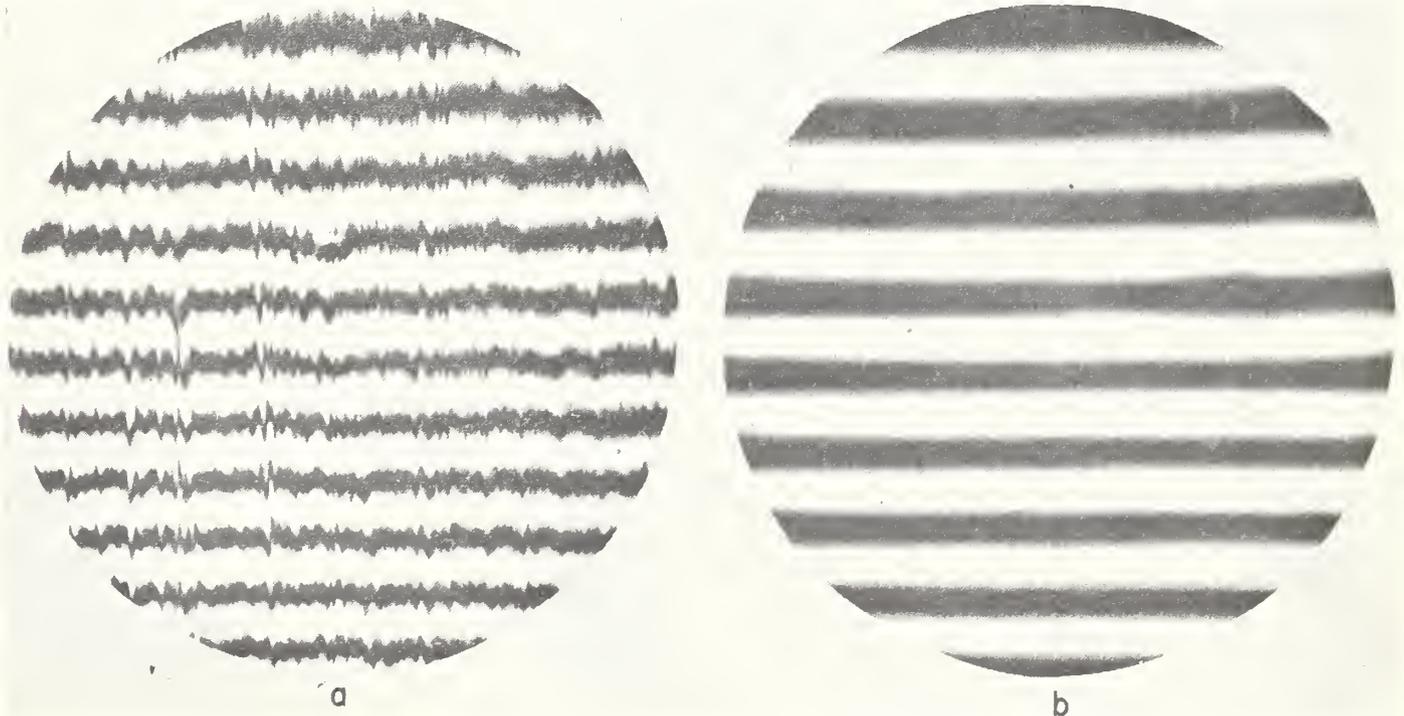


FIGURE 1. *Interference micrographs of a reflecting face (a) before and (b) after cloth polishing, $\times 400$.*

The interference fringes may be considered as contour lines with the distance between adjacent fringes equal to 0.000011 inch. Thus the excursion of a fringe one-half way toward an adjacent fringe indicates a hill or valley 0.0000055 inch high or low with the nature of the irregularity determined by the direction of the fringe displacement.

was made on a rotary table by the use of 15-, 30-, and 45-deg angle blocks, a solid square, and an autocollimator. The rotary table had previously been adjusted so that its surface was closely perpendicular to its axis of rotation and, for convenience in alining the autocollimator, parallel to the surface plate on which it rested. The autocollimator was mounted on a rigid base with its axis parallel to the surface plate at an appropriate height to cover both the polygon faces and the square or angle block.

The 0-, 90-, 180-, and 270-deg faces were positioned by means of the square and the intermediate faces by adding or subtracting 15, 30, and 45 deg from these positions. For example, the 0-deg block was wrung to the base with its two holes approximately concentric with two of the tapped holes; one face of a 30-deg angle block was adjusted parallel to the 0-deg block by means of the autocollimator; the base and the angle block were rotated through 30 deg by means of the rotary table, and the 30-deg polygon block was positioned parallel to the second face of the angle block. Because the 0- and 30-deg blocks could not be fastened to the base until the 15-deg block was in place, this block was positioned next, using the same method as for the 30-deg block. All the other blocks of the polygon were similarly located, working alternately in clockwise and counterclockwise directions from the 0-deg block.

The use of the same screws for holding both layers of blocks to the base made the assembling somewhat tedious, as the top block frequently moved when the screws were tightened, even though well lubricated, cupped, brass washers were used between the screw heads and the blocks. At least a part of this difficulty arose from the fact that some of the tapped holes were not accurately square with the base. The angular position of the top block was observed as the screws were tightened and, when necessary, the positioning repeated until the block was located within the required limits. The blocks in the lower layer had little tendency to move as they wrung quite firmly to the base, but their position was checked, as a precautionary measure, after the fastening screws were tightened.

It was found that quite accurate settings could be made by gently tapping a block to its final position, and, if only a single layer of blocks had been involved, it is believed that the angular intervals could easily have been set to an accuracy of 2 sec.

On this particular polygon no further adjustment was attempted when a block was once located to an accuracy of ± 10 sec, as moderate random departures from nominal angle may, in some applications, be used to good advantage to confirm the direction of corrections.

3. Calibration of Equipment

The autocollimators used in the calibration have a total range of 10 min and a least reading of 0.5 sec.

In order to reduce autocollimator errors to a minimum, a number of precautions were observed. Rotation of the reticle from its true position in a

plane perpendicular to the optical axis introduces an error. The true position of the reticle is parallel to the setting hairlines of the micrometer eyepiece and perpendicular to their direction of travel. When the autocollimator is properly alined, the setting hairlines are parallel to the axis of rotation of the work. The relationship can be expressed by the formula $\theta = \beta \tan \alpha$, where θ is the error in seconds of the reading, β the departure in seconds of the reflecting surface from the plane of measurement of the micrometer eyepiece, and α the deviation of the reticle line from its true position. For a 30-sec change in vertical angle of the polygon reflecting surface and for a 2-deg rotation of the reticle line, the error is 1.05 sec. Because visual alinement of a reticle line is usually limited to an accuracy of about 2 deg, a more accurate method of alinement was required. Accordingly, we removed all the lenses of the autocollimator, projected an image of the reticle on a screen, and, by adjustment of the autocollimator rotational stops, brought the image into coincidence with the image of a suitably disposed machinist square. By this method the reticle line could be set perpendicular to the base of the instrument to within less than 15 min.

After adjustment of the reticle line, the autocollimator microscope was carefully focused on the reticle and the autocollimator objective adjusted to form a crisp image with a representative polygon face.

The two lines of the micrometer eyepiece so closely bracketed the reticle image that traces of color along each edge of the image affected the precision of setting. After some experimentation, a blue-green Wratten filter (B2-58) was found that transmitted an adequate amount of light but largely suppressed the objectionable colors. The remaining secondary chromatic aberration appeared to the operator to be equal on both sides of the reflected line.

Both autocollimators were calibrated over the central 9 min of range in 3-min intervals and over the central 5 min in 1-min intervals by means of master angle blocks. No error in excess of 0.3 sec/min was found in the central part of the range on either autocollimator. Because the differences in the angles to be measured were not expected to exceed 10 sec, it was considered that the progressive errors of the autocollimators could be neglected. As a check on the possibility of periodic errors in the micrometer eyepiece screws, comparisons were made of the angular value of the micrometer eyepiece screws of the two autocollimators in 5-sec intervals over a 1-min range by directing the autocollimators at separate faces of an angle block and rotating it in 5-sec increments between readings. The angular values of the screws on both instruments were found to be uniform within 0.3 sec, which is about the limit to which two settings can be repeated.

4. Calibration of Polygon

All the equipment for the calibration of the polygon was then assembled on a large surface plate. The

polygon was mounted on a rotary table and each autocollimator on a substantial iron base with its optical axis in line with the junction of the upper and lower layers of blocks. Each autocollimator was centered on the appropriate polygon face by viewing with a low-power microscope the real image of the face that is formed in the exit pupil of the autocollimator eyepiece. At the same time, the position of the autocollimator light source was adjusted to provide uniform illumination over the entire polygon face.

Preliminary observations soon indicated that, after handling of the equipment, the entire setup required a fairly long stabilizing period in a constant-temperature room and that temperature gradients in the surface plate due to the presence of observers changed the angle between the autocollimators enough to introduce intolerable errors when the observations extended over a period of 1 or 2 hr. These effects were largely overcome by erecting an insulating barrier completely around and over the top of the setup and scheduling the observations so that at least 4 hr intervened between the readjustment of units and a series of measurements. Small openings in the insulating wall at each autocollimator position permitted reading of the autocollimator. As a check on angular change in autocollimator positions, the initial interval was remeasured after six (or fewer) consecutive intervals were observed.

Measurements were made by directing each autocollimator toward the center of the appropriate polygon face at such an angle that the reflected image of the reticle came within the selected range of the autocollimator. Figure 2 shows the disposition of polygon and autocollimators for the measurement of the 30-deg intervals. After recording the reading of the two autocollimators, the rotary table on which the polygon was mounted was then turned to present the faces of the adjacent angular interval. This process was repeated until every interval of the same nominal size had been measured and the circuit closed. Because some of the intervals are aliquot parts of 360 deg and others are aliquots of a multiple of 360 deg, a closure consisted of from 2 to 24 read-

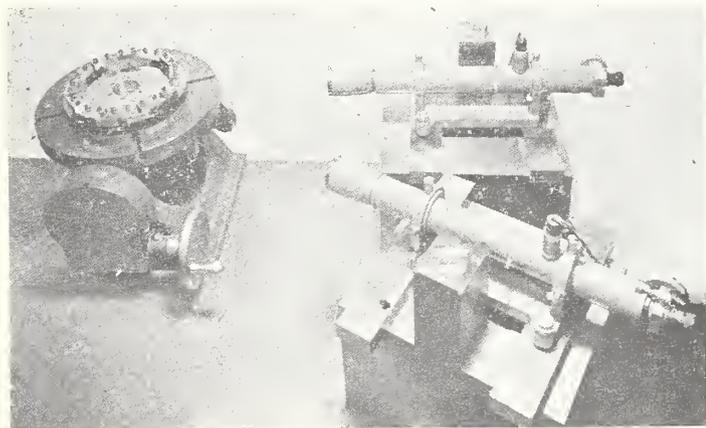


FIGURE 2. Arrangement of polygon and autocollimators for the measurement of 30-degree intervals.

One autocollimator is directed at a selected face and the other at a face 30 degrees away.

ings. Where a closure consisted of less than 24 readings, additional closures were required to cover all the intervals. For example, 12 closures of two readings each were required to cover the 180-deg intervals, whereas one closure consisting of 24 readings over 3,960-deg (11 revolutions) was required for the 165-deg intervals.

The difference between the readings of the two autocollimators is an estimate of the difference in angle defined by the two autocollimators and the angle between the two polygon faces. Because the angle between the collimators was only approximately equal to the nominal polygon interval, each difference, d_i , in any particular closure is an estimate of the error of the polygon interval, γ_i , plus a constant error, Δ , due to the deviation from nominal of the angle between the autocollimators. Thus $d_i = \gamma_i + \Delta$.

For a complete closure

$$\sum_{i=1}^n d_i = \sum_{i=1}^n \gamma_i + n\Delta,$$

where n is the number of readings required for a closure. But since $\sum_{i=1}^n \gamma_i$ for a circle or any multiple thereof equals 0,

$$\Delta = \frac{1}{n} \sum_{i=1}^n d_i.$$

Therefore,

$$\gamma_i = d_i - \frac{1}{n} \sum_{i=1}^n d_i,$$

from which values for each polygon interval of a closure can be obtained. However, each difference is subject to a random error e , so that we have an e_i associated with each d_i . It is reasonable to assume that all errors, e_i , arise independently and randomly from the same error distribution, and that the distribution is characterized by an average value of zero and a dispersion of the magnitude indicated by a standard deviation, σ . This standard deviation is a measure of the precision of an individual difference by this process of measurement; an average of n individual values will have the precision indicated by a standard deviation of σ/\sqrt{n} and the sum of k individual values will have a standard deviation of $\sigma\sqrt{k}$.

If only the closure for the 15-deg interval is made, the precision with which the corrections to each of the 24 basic angles is known would be the same for all angles, but that of any angle comprised of the sum of two or more 15-deg angles would vary with the number of 15-deg angles involved.

In order to provide corrections of equal precision for all intervals, 552 measurements involving 76 closures were made to obtain all possible sums of consecutive angles. From these measurements corrections were derived for each interval. For example, in the determination of the correction for the 0- to 15-deg interval, 48 values were obtained from the following two series of differences:

[(0- to 15-deg correction)—(15- to 15-deg correction)],
 [(0- to 30-deg correction)—(15- to 30-deg correction)], . . . ,
 [(0- to 360-deg correction)—(15- to 360-deg correction)], and
 [(360- to 15-deg correction)—(360- to 0-deg correction)],
 [(345- to 15-deg correction)—(345- to 0-deg correction)], . . . ,
 [(15- to 15-deg correction)—(15- to 0-deg correction)].

The least-squares estimate for each angular interval involves the same number of measurements, and the precision with which the correction to any angle is known is $\sigma/\sqrt{24}$. An estimate of the standard deviation, σ , was determined from the least-squares solution. For any single measurement, the standard deviation, σ , was found to be 0.22 sec. Hence, the final value for each of the intervals had a standard deviation of $0.22/\sqrt{24}$, or 0.045 sec.

In addition to random errors, any calibration process commonly has systematic errors associated with it. In this case, however, no error existed in the primary standard, a circle, and any possible small errors in the autocollimator screws were so distributed over the entire series of measurements that their effect largely assumed a random characteristic. Accordingly, it is believed that the systematic errors may be considered negligible and, therefore, disregarded. Knowledge of the true corrections is, therefore, obscured solely by the random errors of measurement, and the accuracy of the process involves only some upper limit to the magnitude of the random errors.

A reasonable upper limit for the magnitude of the random errors can be set by selecting the value from the error distribution for which there is a chance of only one in one hundred of a greater discrepancy occurring. For this definition (assuming a normal distribution of errors), the accuracy of the corrections is $\pm 2.58(.045) = \pm .12$ sec.

The actual values of the intervals are of no particular interest to a reader of this paper, but it may be noted that the maximum deviation from nominal size of any 15-deg interval was 8.2 sec and for any interval, 9.0 sec.

5. Application of the Polygon

The polygon was used to calibrate a master 45-deg angle block belonging to the Bureau. The calibration could be made either by comparing the angle block with one or more intervals of the polygon or by comparing the angle block with a sufficient number of consecutive intervals to encompass an integral number of polygon rotations or circuits. In the case of a 45-deg block, whose external angle is 135 deg, eight consecutive readings covering 1,080 deg are required for a closure. Two additional series of eight readings each are required to provide comparisons with every 135-deg interval of the polygon.

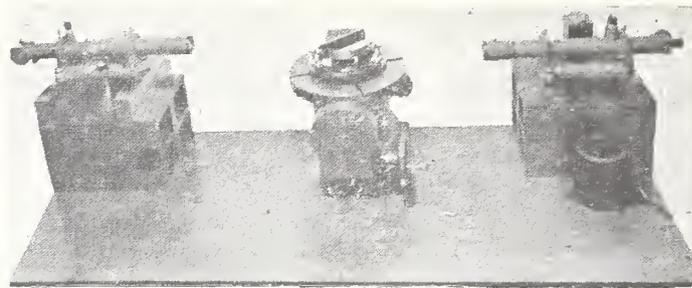


FIGURE 3. Arrangement of polygon, angle block, and autocollimators for calibration of 45-degree angle block.

One autocollimator is directed at the polygon and the other at the angle block.

The disposition of the equipment for the calibration of a 45-deg angle block is shown in figure 3. The polygon and angle block were mounted on a rotary table with the angle block elevated just above the polygon faces by means of a plane parallel block interposed between the polygon base and the block. Two autocollimators were positioned 180 deg apart, with one directed at a polygon face and the other at an angle-block face. The same care was observed in alining the equipment as for the calibration of the polygon, but temperature effects were largely eliminated by the 180-deg disposition of the autocollimators.

With the autocollimators appropriately alined, the readings of the two instruments were recorded. The assembly of polygon and angle block was then rotated 135 deg by means of the rotary table, and the autocollimator readings were again noted. The angle block was next rotated 135 deg with respect to the polygon, and comparison of the angle block with the adjacent 135-deg polygon interval was obtained by observing readings of both autocollimators at that position and after another 135-deg rotation of both polygon and angle block. Similar comparisons with the next six consecutive intervals of the polygon completed a closure and permitted computation of the angle of the angle block. In order to obtain a more accurate value, additional closures were made, relating the angle block to other 135-deg-polygon intervals.

Table 1 is a tabulation of two closures on a 45-deg angle block. Column 1 is the polygon interval; column 2, the difference in autocollimator readings on the polygon; column 3, the difference in autocollimator readings on the angle block; column 4, the difference between columns 2 and 3; column 5, the error in the polygon interval by the previous calibration; column 6, the error of the angle block by direct comparison with the polygon interval; and column 7, the error of the angle block by comparison with all the polygon intervals in each of the separate closures. The latter is wholly independent of any errors in the values for the polygon intervals and therefore somewhat more accurate than the average of eight values not constituting a closure.

The angular rotation of the block and polygon between readings was $135 \text{ deg} + e_p + d_1$ in terms of polygon values and $135 \text{ deg} + e_b + d_2$ in terms of angle block values, where e_p is the error in the polygon interval, d_1 is the difference in autocollimator

readings on the polygon, e_b is the error in the angle of the block, and d_2 is the difference in autocollimator readings on the angle block.

Equating the two expressions, we have $e_b = d_1 - d_2 + e_p$. The values of e_b are given in column 6 of table 1. Since the sum of the e_p 's for a closure is zero, e_b is estimated by $\frac{1}{8}\Sigma(d_1 - d_2)$, where 8 is the number of readings in a closure. This estimate is given in column 7 of table 1, and it is independent of the errors in the polygon.

TABLE 1. Measurements of a 45-deg angle block by comparison with 135-deg intervals of polygon

1	2	3	4	5	6	7
Polygon interval	Difference in polygon readings, d_1	Difference in block readings, d_2	$d_1 - d_2$	Error of polygon intervals	Error of angle block	$\frac{\Sigma(d_1 - d_2)}{8}$
deg	sec	sec	sec	sec	sec	sec
0 to 135	12.13	10.10	2.03	-1.76	0.27	-----
135 to 270	1.77	0.63	1.14	-1.19	-.05	-----
270 to 45	-5.57	-3.20	-2.37	2.34	.03	-----
45 to 180	12.86	12.03	0.83	-0.45	.38	-----
180 to 315	13.60	14.70	-1.10	0.87	-.23	-----
315 to 90	4.13	1.47	2.66	-2.41	.25	-----
90 to 225	6.53	7.43	-0.90	0.48	-.42	-----
225 to 0	-5.14	-2.76	-2.38	2.12	-.26	-0.01
15 to 150	-7.34	-8.60	1.26	-1.17	.09	-----
150 to 285	6.97	2.00	4.97	-4.96	.01	-----
285 to 60	-3.17	-1.20	-1.97	1.82	-.15	-----
60 to 195	-11.13	-5.93	-5.20	4.81	-.39	-----
195 to 330	5.44	4.07	1.37	-0.76	.61	-----
330 to 105	1.47	-1.57	3.04	-3.02	.02	-----
105 to 240	12.70	10.94	1.76	-1.43	.33	-----
240 to 15	1.46	6.67	-5.21	4.71	-.50	0.00

Four additional measurements gave values of +0.01, -0.01, -0.12, and +0.03 sec for $\Sigma(d_1 - d_2)/8$.

The standard deviation, σ , for the six values of $\Sigma(d_1 - d_2)/8$ is 0.02 sec. If we use a 99 percent confidence limit, as in the case of the polygon, the average value of -0.01 sec may be considered accurate to ± 0.09 sec.

6. Other Possible Uses

Although designed primarily as a master for angle blocks, the polygon is equally suitable for the test or calibration of angular dividing equipment. Used alone, it permits the calibration of 15-deg. intervals and, in conjunction with other polygons, of intervals as small as 1 deg by the method described by Taylerson (see footnote 3).

Simple arrangements of a few blocks can be easily assembled to serve as masters or gages in the optical or mechanical industry.

A further extension of the application of assembled polygons to the calibration of circular dividing equipment is being considered.

Taylerson's method for calibrating 1-deg intervals requires the use of a number of polygons, each having a different number of sides. The complete calibration, using one suggested group of polygons, requires 49 circuits of three polygons, with a total of 408 readings.

The ease and precision with which defining faces can be positioned on an assembled polygon suggests the possibility of using a single polygon having in addition to the usual large intervals, one or more blocks offset at such angles as to subdivide the principal intervals. If, for example, a 12-sided polygon having 30-deg intervals is used, the addition of a single block offset from any one of the faces by 1 deg permits the calibration of test equipment in 1-deg increments. The 1-deg interval would fix the relationship between successive series of comparisons with the equal intervals of the polygon and necessarily enter the complete calibration as many as 15 times. If, however, two additional blocks are offset 10 and 20 deg, respectively, no more than five 1-deg intervals need be added or subtracted to establish the relationship between circuits. The addition of three more blocks offset 5, 15, and 25 deg, respectively, further reduce the number of 1-deg intervals entering a calibration and also provides very convenient method of calibrating equipment in 5, 10, or 15-deg increments.

The procedure for calibrating a rotary table in increments of 1 deg with a 12-sided polygon having three additional blocks offset 1, 10, and 20 deg, respectively, is as follows: Every 30-deg interval of the polygon is compared with rotary-table intervals starting at zero, then at 1, 2, 3, etc. up to 29 deg, inclusive. Additional comparisons are made with the 1-, 10-, and 20-deg intervals starting at zero and with the 1-deg interval on succeeding circuits. From the values of the polygon intervals, the errors of the rotary table can be derived in 1-deg increments.

For simplicity of explanation, the additional 1-, 10-, and 20-deg blocks have been indicated as offset from the zero polygon face, whereas space limitations would require that they be distributed around the polygon and offset from other polygon faces. The only effect of such a distribution is to change the computational procedure.

The use of a single polygon having faces offset in fractions of the main intervals has two advantages over Taylerson's method:

1. Thirty circuits with 392 readings are required, as compared with 49 circuits with 408 readings. The minor reduction in the number of readings is of little importance, but the large reduction in the number of circuits and the concomitant manipulation of the polygon should effect an appreciable saving in time.

2. A major reduction in the cost of equipment is achieved by the substitution of a single polygon for a group of three.

The authors express their appreciation to J. M. Cameron and his associates of the Bureau's Statistical Engineering Laboratory for guidance in selecting the most suitable series of observations, development of formulas for the best utilization of the data, and computation of results.

WASHINGTON, September 16, 1952.

MEASUREMENT OF SURFACE ROUGHNESS OF ELECTRO- DEPOSITED AND ELECTROPOLISHED SURFACES BY MEANS OF THE MICROINTERFEROMETER

ARTHUR G. STRANG* AND FIELDING OGBURN†

I. INTRODUCTION

As indicated by the papers that are being given at this symposium, electroplaters have an increasing interest in leveling phenomena and finishing operations. In order to evaluate these processes, it is necessary to have methods or tools for examining and measuring surface smoothness or brightness. One such tool is the microinterferometer, or interference microscope^{1, 2}. This instrument was developed and built during World War II at the Carl Zeiss Works, Jena, Germany, and has not yet become available commercially. Probably less than three dozen instruments exist. Because it is a potentially valuable tool for studying the surfaces of electrodeposits, a brief discussion of it may be of interest.

II. DESCRIPTION OF THE MICROINTERFEROMETER

The Zeiss Microinterferometer (Fig. 1) gives absolute measurements of surface irregularities ranging from 2 to 80 μ in (0.05–2 μ) on highly reflective samples and from 2 to 10 μ in (0.05–0.25 μ) on the more reflective matte surfaces. This instrument gives a visual magnification of 664 \times of a circular area, 0.007 inch (175 μ) in diameter, with interference fringes superimposed upon the surface. When an optically flat surface is viewed, these interference fringes consist of a number of equally spaced parallel black bands separated by green bands (Fig. 3). If an imperfection is present on the surface, the band or bands will follow around or across the imperfections, moving to one side for a valley and to the other side for a hill. The interference micrograph is similar to a topographical map, on which the contour lines represent positions of equal heights.

Black-band interference is produced when one part of the light waves from a single monochromatic source is 180° out of phase with respect to the other part of the light waves. If a wedge-shaped space with an angle of 20'' is formed between two transparent, optically flat surfaces which are in contact on one edge, ten black interference fringes per inch are formed under monochromatic blue-green light with a wave-length of 5080 Å \ddagger . At the point of

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‡ One Ångstrom unit, Å, is equal to 1×10^{-8} cm, or 0.004 μ in.

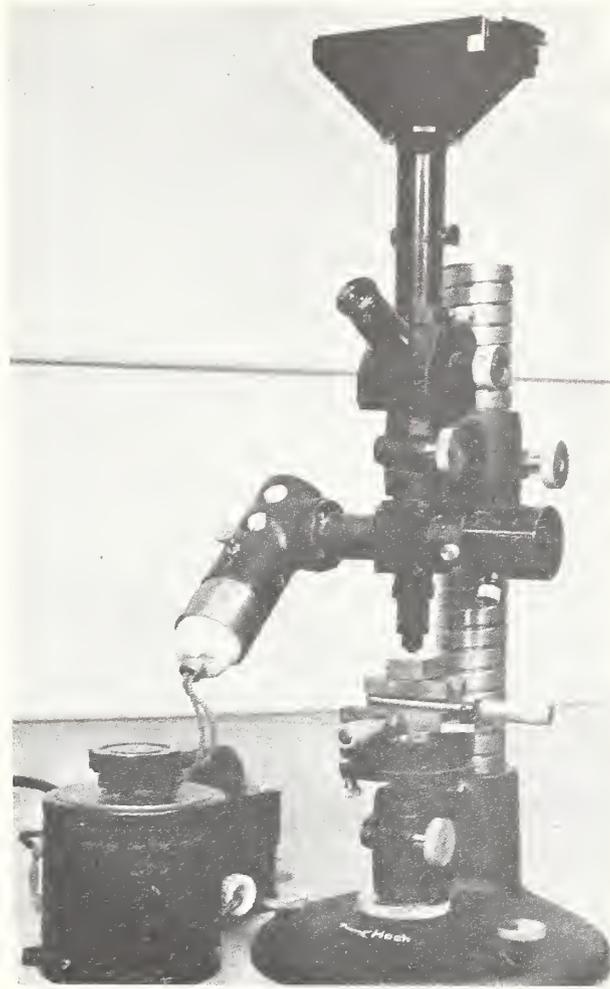


FIG. 1.—Zeiss Microinterferometer

contact, a black fringe can be seen. Part of the light is reflected from the bottom surface of the top optical flat and part from the top surface of the bottom optical flat, because a thin air film, the thickness of which is a very small fraction of a wave-length, exists at the point of contact. Interference occurs because light reflected from a surface of higher refractivity than the transmitting medium undergoes a 180° phase shift*. Whenever the distance between the two reflecting surfaces is a multiple of $10 \mu\text{in}$ (0.25μ), *i.e.*, a whole number of half wave-lengths apart, interference occurs. Between the

* If the lower surface is metal, the phase shift is less than 180° ; consequently there is no black fringe at the point of contact, but the fringe system is displaced towards the point of contact by about $\frac{1}{4}$ of the distance between fringes.

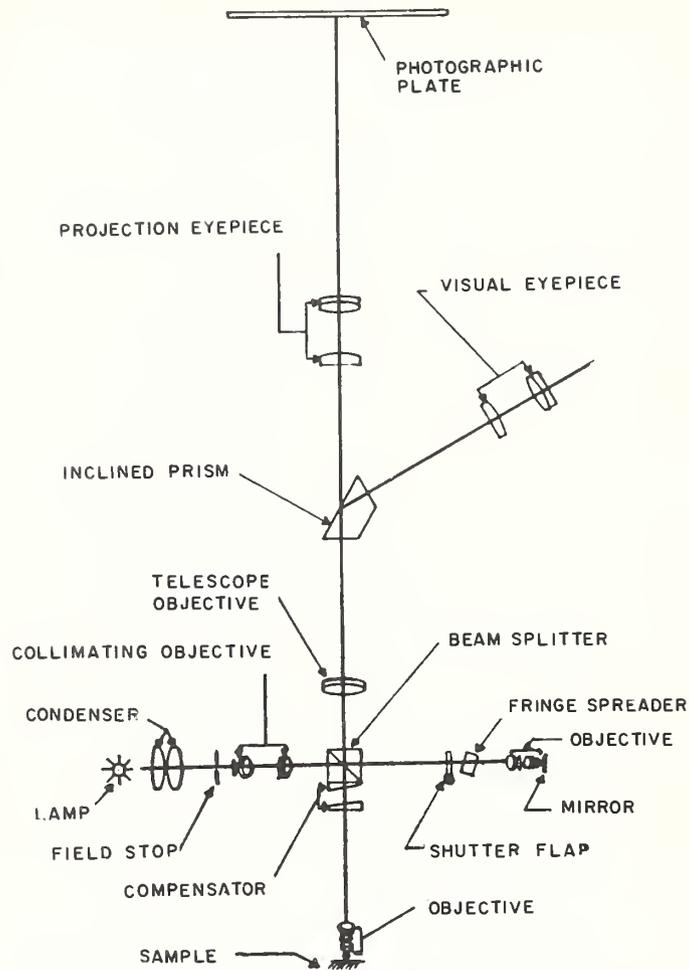


FIG. 2.—Optical diagram of Zeiss Microinterferometer

black fringes, there are bright blue-green fringes caused by the light waves reinforcing each other.

Fig. 2 shows the optical diagram of the Zeiss microinterferometer. The Michelson Interferometer functioning at zero path difference is built into the microscope. The entering light is focused horizontally on a pin-hole stop by a condensing lens system and then passes through a collimator objective. This parallel light is split in half at the beam splitter, which is a biprism made by cementing two reflecting prisms together with a metal film of 50 per cent transmittance between them. Half of the collimated light goes straight through the metal film of the beam splitter, past a shutter flap, through a fringe spreader, and then through a 4-mm microscope objective which is focused on an optically flat mirror located perpendicular to the optical axis. This beam is reflected back through the objective, fringe spreader, and shutter

flap to the beam splitter, where its path is directed 90° up towards the eyepiece. The other half of the collimated light is reflected 90° down towards the base of the instrument by the metal film on the prism, through a compensator and through another 4-mm microscope objective which is focused on the surface of the specimen. Any highly reflective sample returns the light through the objective, compensator, and biprism. Here the two beams meet and interference fringes of equal inclination are formed. Then the combined beams pass vertically upwards through a telescope objective, which takes these parallel rays and converges them through an inclined prism to the focal plane of the visual eyepiece or through the projection eyepiece to the photographic plate.

While viewing a test piece, the operator can examine the microgeometrical surface with or without interference fringes by turning a shutter which cuts off the mirror of the interferometer. The fringe count can be varied from one to 25 fringes over the entire surface by simply rotating a fringe spreader, which is a plane parallel glass plate located between the beam splitter and the microscope objective which is focused on the mirror. If smaller fringe spacing is required, the sample may be tilted slightly. Also, the fringe direction can be shifted to any desired position by rotating the objective which is focused on the mirror.

Sharp fringes, being desired a monochromatic light source is essential. The optics in the Zeiss microinterferometer were corrected for a wave-length of approximately 5400\AA . A special thallium light source is used because its radiation produces a green light that is monochromatic for all practical purposes without the use of filters, although there are faint red and blue fringes present. Other light sources can be used, but then monochromatic filters are necessary. If a polychromatic source is employed, the black fringes are replaced by colored fringes which cannot be interpreted accurately. Even with monochromatic radiations the black fringes have diffuse borders which do not permit the accurate evaluation of peak-to-valley distances of less than $2\ \mu\text{in}$ ($0.05\ \mu$) on highly polished flat surfaces.

The test surface should have the same reflectance as the mirror in order that maximum fringe contrast with the microinterferometer be obtained. This instrument has a mirror with 60 per cent reflectivity, which is a close match for polished steel surfaces commonly found on gage blocks and for nickel and chromium plated surfaces. Surfaces with reflectivities of 20 per cent specular reflectance can be photographed by using a mirror of low reflectivity. Reflective, flat matte surfaces are difficult to analyze because the entire surface is made up of pits and nodules. If these imperfections are less than $10\ \mu\text{in}$ ($0.25\ \mu$) above or below the nominal surface, the fringes will be covered with fine irregularly shaped spots. Poorly reflective matte finishes, having imperfections larger than $10\ \mu\text{in}$ ($0.25\ \mu$), produce several small concentric fringe enclosures which are packed very close together, thus concealing the identity of the fringes.

Curved surfaces can be inspected, but only that portion within the objective field which lies within $50\ \mu\text{in}$ ($1.25\ \mu$) of either side of the focal plane

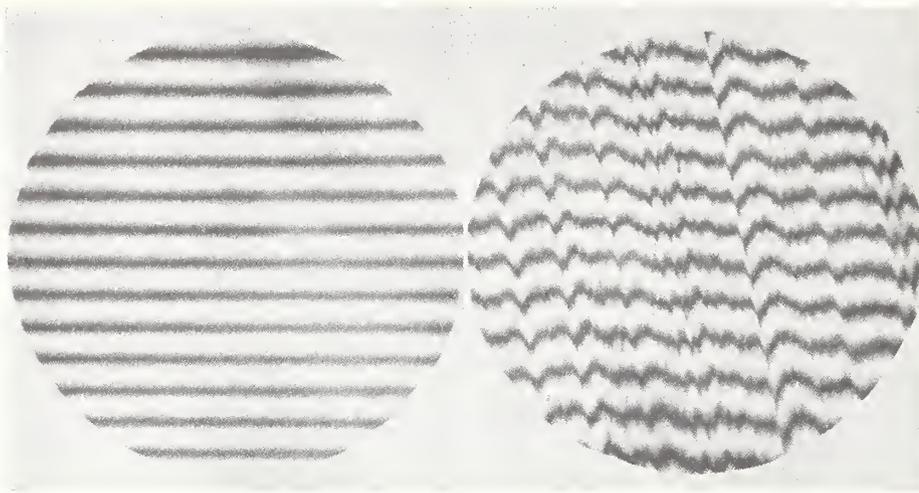


FIG. 3 (left).—Outer surface of 0.0085-inch thick bright-nickel deposit. Profilometer reading $1.5 \mu\text{in}$ RMS. $400\times$

FIG. 4 (right).—Underside of bright nickel deposit in Fig. 3, which was stripped from the stainless-steel basis metal, is a reproduction of the stainless-steel surface. Profilometer reading, $4 \mu\text{in}$ RMS. $400\times$

can be seen. Surfaces on spheres and cylinders with diameters as small as 0.030 inch (0.76 mm) have been successfully examined with this limitation.

III. INTERPRETATION OF INTERFERENCE MICROGRAPHS

An understanding of the interference patterns may be simplified by visualizing parallel and equally spaced layers, or band levels, located perpendicular to the optical axis between the microscope objective and the test surface. The test piece is on an incline with respect to the band levels, and therefore each band level intersects the surface under observation to produce contour bands or altitude levels. The bands represent altitude intervals of $11 \mu\text{in}$ (0.28μ). This value is the half wave-length for the predominate green spectrum line of thallium, which is 5350 \AA . On flat surfaces, these bands are straight, parallel, and equally spaced as shown in Fig. 3. On cylinders they are straight and parallel, unequally but symmetrically spaced. On a ball or socket, they are concentric circles of varying diameters. If a V groove $16 \mu\text{in}$ (0.4μ) deep cuts across a flat surface perpendicular to the bands, the bands will all follow the shape of the scratch, and the points of the fringe arrows will extend halfway beyond the adjacent fringe. If there is a burred edge extending $5 \mu\text{in}$ (0.13μ) above the surface adjacent to this scratch, another series of arrows will point in the opposite direction and stop halfway between the fringes. If the scratches are oblique to the fringes, the straight-line fringes will deviate obliquely. Since most mechanically formed surfaces contain unidirectional or random-directional scratches of varying magnitude, the sur-

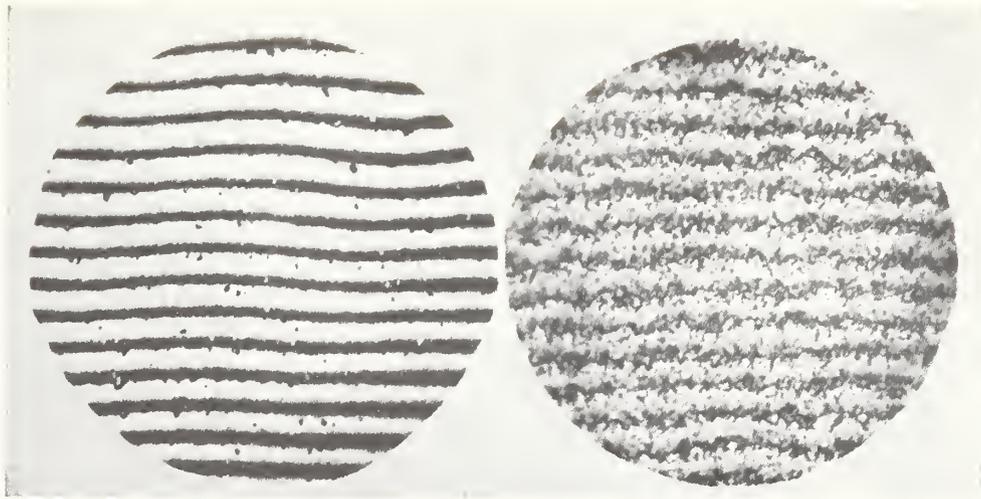


FIG. 5 (left).—A bright-nickel deposit 0.006-inch thick deposited on stainless steel. Surface of basis metal is similar to that shown in Fig. 3. Profilometer reading, $4 \mu\text{in}$ RMS. $400\times$

FIG. 6.—Outer surface of 0.008-inch semibright nickel deposited on stainless-steel surface similar to that shown in Fig. 3. Profilometer reading, $2.5 \mu\text{in}$ RMS along and $4 \mu\text{in}$ RMS across the rolling scratches. $400\times$

face finish may be evaluated by ascertaining the fringe deviations. Certain types of electrodeposited surfaces can be evaluated, but a suitable modification of the procedure must still be devised to measure matte surfaces adequately.

The microinterferometer has a number of advantages and some disadvantages when compared with surface analyzers of the tracer type. An exact peak-to-valley measurement can be made over a small area with the microinterferometer. Further, it is possible to make a fairly accurate estimate of an RMS (root mean square) value from the image. Measurements on a given area are reproducible. The character and texture of the surface can be observed and compared with those of other surfaces. It is possible to evaluate the finish on soft materials or surfaces without marring them, because there is no physical contact between the measured surface and the microscope. Measurements of internal surfaces cannot be made except by destroying the sample.

Surface analyzers of the tracer type have a much wider range, e.g., from 2 to 500 μin (0.05 to 12.5 μ). They give an RMS value, but they do not indicate the exact depth and shape of the scratches. Matte surfaces can be measured. Frequently it is not possible to reproduce RMS values on a given sample with different makes of tracer units, due to many such variables as radii of tracer points, complicated amplifiers, crystal pickups, as well as mechanical and personal errors.

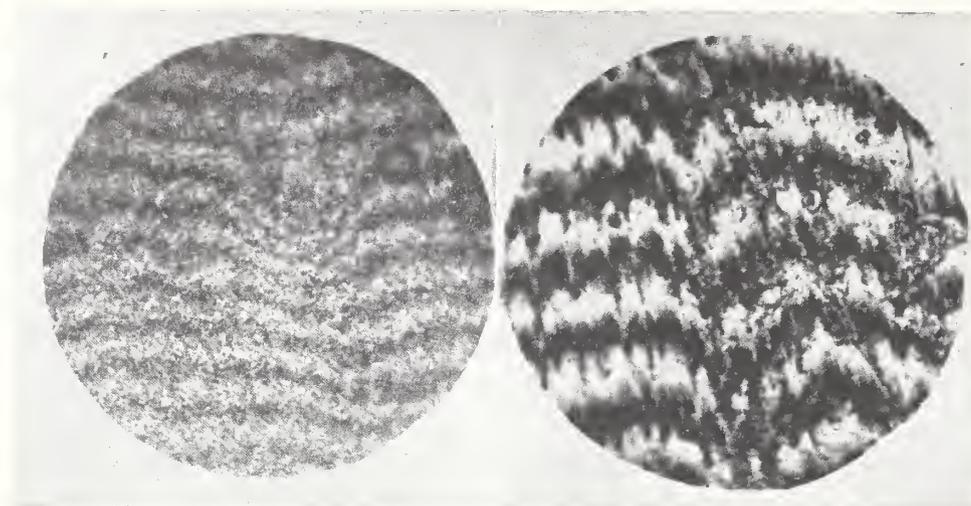


FIG. 7 (left).—Outer surface of matte deposit of nickel from an all-chloride bath. Profilometer reading, $11 \mu\text{in RMS}$. $400\times$

FIG. 8 (right).—Underside of deposit shown in Fig. 7. Profilometer reading, $11 \mu\text{in RMS}$. $400\times$

IV. EXAMPLES OF INTERFERENCE MICROGRAPHS

Figs. 3-14 were taken on "contact-process ortho sheet film". Exposures were less than 80 seconds, because temperature changes and vibration alter the optical paths in the interferometer and cause the fringes to shift. A thallium-vapor light source was used for some of the exposures, and a mercury lamp (G. E. AH-4) for the others. Filters were used to eliminate all mercury spectrum lines except the green line, which is very close to the thallium green line.

Each interference micrograph is accompanied with an RMS value for the same surface, obtained with a profilometer. These values are $1/3$ - $1/5$ of the average peak-to-valley distance³ and may not seem to be in agreement with some of the interference micrographs. This is to be expected because the field of the micrograph is much smaller than that of the profilometer and may be too small for detection of some of the imperfections which are picked up by the profilometer.

These interference micrographs of various metal surfaces were chosen merely as illustrations to indicate the potentialities of the instrument. The photographs are mounted so that displacement of the interference lines towards the bottom of the photograph indicates a depression in the surface of the specimen, and displacement of the fringes towards the top indicates a raised area. The interference lines are $11 \mu\text{in}$ (0.28μ) apart.

Several electrodeposits were made on stainless steel and stripped off. Fig. 4 is an interference micrograph of the underside of a bright-nickel deposit which should be an accurate reproduction of the stainless steel surface. The

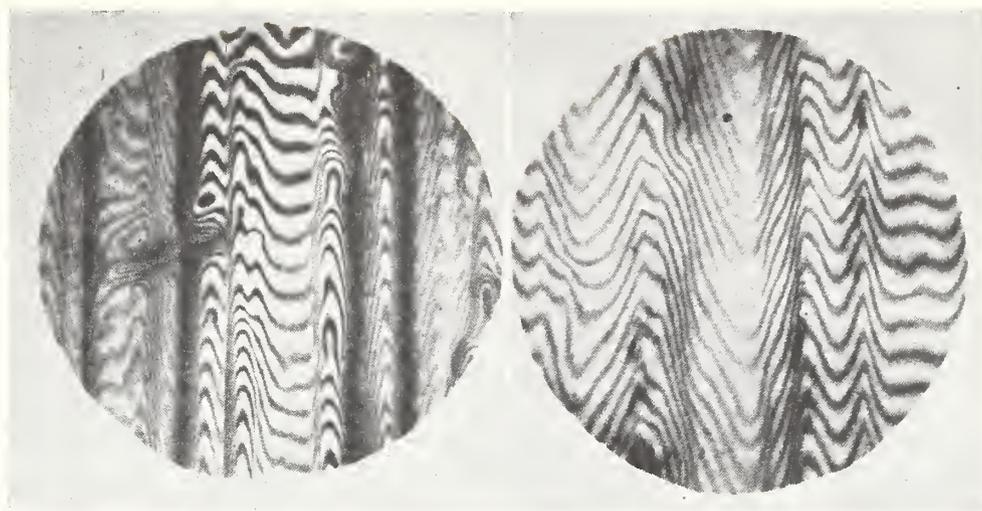


FIG. 9 (left).—Dull nickel and chromium successively deposited on steel surface polished with 90-grain abrasive. Profilometer reading, $62\ \mu\text{in}$ RMS. $400\times$

FIG. 10 (right).—Copper, bright nickel, and chromium successively deposited on steel surface polished with 150-grain abrasive. Profilometer reading, $30\ \mu\text{in}$ RMS. $400\times$

scratches indicated were visible to the eye, the deepest one shown being about $6\ \mu\text{in}$ ($0.15\ \mu$) deep. The RMS value for this surface, in a direction perpendicular to the scratches, is $4\ \mu\text{in}$ ($0.1\ \mu$).

Fig. 3 shows the top surface of the same deposit shown in Fig. 4. This bright-nickel deposit was about 0.0085 inch ($216\ \mu$) thick. The regularity and parallelism of the interference lines indicate, in this local area, a very smooth bright surface with peak-to-valley irregularities of 2 microinches or less. An observational estimation of RMS would be $0.5\ \mu\text{in}$ ($0.013\ \mu$). Since the sample has a wavy surface, the profilometer indicates a higher RMS value of $1.5\ \mu\text{in}$ ($0.038\ \mu$).

Fig. 5 shows another bright-nickel surface. This deposit was 0.006 inch ($150\ \mu$) thick and was plated on a surface similar to that shown in Fig. 3. This surface has many pits and nodules which cannot be seen with the ordinary light microscope. When the interference fringes are superimposed upon the surface, the imperfections show up as black spots between fringes, as white spots inside of the fringes, and as concentric black circles with a dark or light center. The greatest pit depth is $13\ \mu\text{in}$ ($0.33\ \mu$). On this particular sample, it is not known whether the nickel as deposited had a flat surface, because the nickel deposit may have become distorted during stripping from the basis metal. The fringes indicate a waviness of $3\ \mu\text{in}$ ($0.075\ \mu$), which could be caused by such distortion.

Fig. 6 shows the outer surface of a semibright-nickel deposit, plated to approximately the same thickness and on the same type of stainless-steel surface as the deposit shown in Figs. 3 and 4. This flat nickel surface is

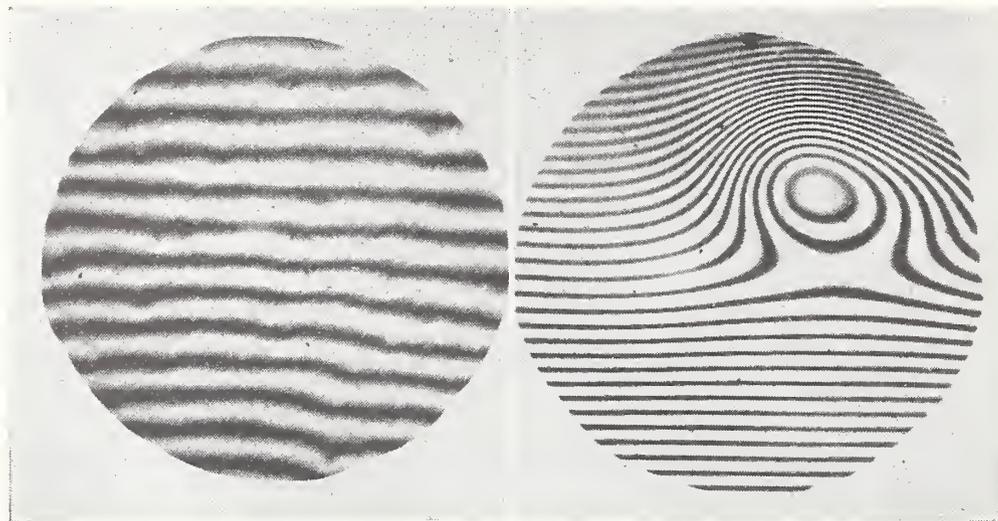


FIG. 11 (left).—Commercial "chromium" finish on plumbing fixture (chromium and nickel on brass). Profilometer reading, $2 \mu\text{in}$ RMS. $400\times$

FIG. 12 (right).—Surface of nickel-chromium finish on bumper bar with a small nodule 0.0001-inch high. Profilometer reading, $6 \mu\text{in}$ RMS. $400\times$

covered with many minute, irregularly shaped pits and nodules which obscure any pattern or scratches attributable to the basis metal. These imperfections average around $5 \mu\text{in}$ (0.13μ) above or below the nominal surface, the maximum being $20 \mu\text{in}$ (0.5μ).

The appearance of the outer surface of a nickel deposit about 0.001 inch (25μ) thick obtained from an all-chloride bath is shown in Fig. 7. The underside of this deposit is shown in Fig. 8. The under surface and top surface both have a profilometer RMS value of $11 \mu\text{in}$ (0.28μ). Matte finishes of the kind shown in Fig. 7 are difficult to measure with the microinterferometer, because the interference bands become extremely diffuse. In some cases the dimensions of the irregularities can be estimated, but not with the accuracy with which scratches on otherwise bright surfaces are measured. The imperfection in the center is approximately 2 bands or $22 \mu\text{in}$ (0.56μ) deep. The isolated dark circle to the right in Fig. 7 is a pit $15 \mu\text{in}$ (0.38μ) deep.

Figs. 9 and 10 are photographs of two panels prepared for an earlier study⁴. The steel panels were polished to a definite finish and then plated successively with nickel and chromium.

The steel surface of Fig. 9, which was polished with a 90-grain abrasive, plated with 0.00075 (19μ) inch of dull nickel, color buffed, and plated with 0.00001 inch (0.25μ) of chromium, produced a surface having a profilometer RMS value of $62 \mu\text{in}$ (1.57μ). This surface is entirely too rough to measure by microinterferometry because it is impossible to trace the fringe path. The black bands which follow the deep scratches are made up of closely packed fringes which cannot be separated. On the basis of the profilometer reading,

it is expected that some of the scratches are $240 \mu\text{in}$ (6.1μ) deep. The center third of Fig. 9 is in sharp focus, but the outer parts of the photograph are beyond the focal plane of the microscope objective. If the sample is elevated, it is possible to see fringes on the bottom of these deep scratches which have reflecting surfaces. It can also be seen that if the slope on the side of the groove or pit is steep, the lines tend to merge and become difficult to follow.

The steel surface in Fig. 10 was polished with 150-grain abrasive and plated successively with 0.00025 inch (6.3μ) of copper, 0.001 inch (25μ) of bright nickel, and 0.00001 inch (0.25μ) of chromium. This surface has an RMS value of $30 \mu\text{in}$ (0.76μ) and is smoother than the surface shown in Fig. 9. The peak-to-valley distance is about 8 fringes, or $88 \mu\text{in}$ (2.2μ). This photograph has to be out of focus to permit the large fringe displacement to be traced.

Fig. 11 shows a commercially prepared "chromium" finish of a cover panel for kitchen-sink valves and faucets. This surface is bright and has a number of faintly visible scratches. These scratches, which can be seen in the interference micrograph, are around 3 or $4 \mu\text{in}$ ($0.075\text{--}0.1 \mu$) deep.

The chromium-nickel finish on a bumper bar is shown in Fig. 12. No scratches are detected on the bright surface, but a nodule about 0.0001 inch (2.5μ) high is clearly shown. This surface is made up of small waves or ripples, which are much broader than the field of the micrograph, but which are picked up by the profilometer. The RMS value is $6 \mu\text{in}$ (0.15μ) when measured perpendicular to the ripples and $2 \mu\text{in}$ (0.05μ) when measured parallel to the ripples.

Fig. 13 shows the surface of a chromium plated knob of a microscope. The scattered spots are small pits or nodules. Several fine scratches and one small ridge are visible. The RMS value is $2 \mu\text{in}$ (0.05μ).

A piece of low-carbon steel was ground to give a very rough finish. Part of this surface was then electropolished in a phosphoric-chromic acid solution

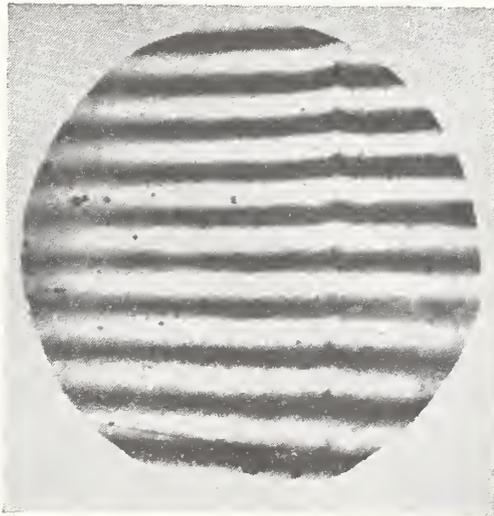


FIG. 13.—Surface of a chromium plated knob of a microscope. Profilometer reading, $2 \mu\text{in}$ RMS. $400\times$

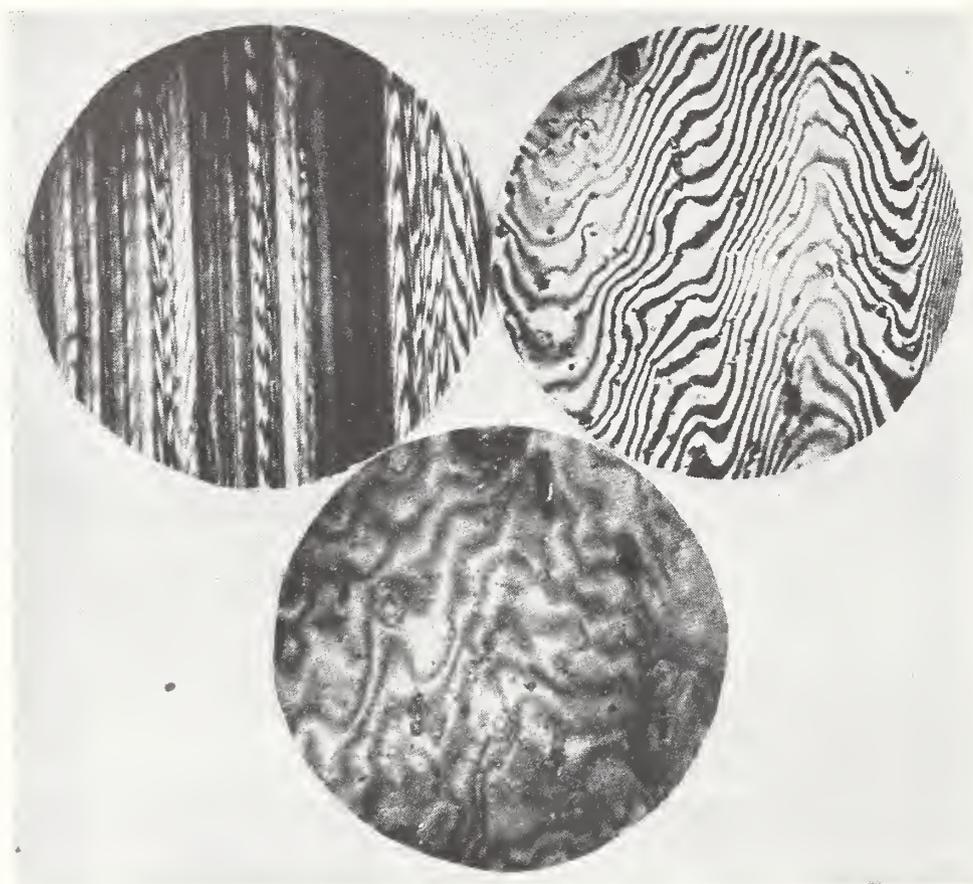


FIG. 14.—Ground low carbon steel surface before and after electropolishing. Top left, original surface; profilometer reading, $50\ \mu\text{in}$ RMS. Top right, after 20-minute electropolish; profilometer reading, $40\ \mu\text{in}$ RMS. Bottom, after 40-minute electropolish; profilometer reading, $30\ \mu\text{in}$ RMS. $400\times$

for 20 minutes, and a second portion was electropolished for 40 minutes. These surfaces, shown in Fig. 14, have RMS values of 50 , 40 , and $30\ \mu\text{in}$ (1.25 , 1.0 , and $0.75\ \mu$), respectively. The original surface, at left, is too rough to measure on the interference micrograph, because the interference lines merge on the steep sides of the numerous scratches. The center micrograph indicates a considerably smoother surface after a 20-minute electropolish. The lines do not merge and hence indicate more gentle slopes. It will be noticed that neither the high points nor the low points are in focus. After 40 minutes of electropolishing, at right, most of the shallow scratches disappeared and the deep scratches were reduced in depth. The surface is wavy and covered with pits and nodules. Since the field of the microscope is limited, this picture shows only part of one large wave.

V. APPLICATIONS OF THE MICROINTERFEROMETER

As compared with most other instruments now in use, the microinterferometer has the advantage that it will readily yield measurements in three dimensions instead of only in two. The distribution and dimensions of imperfections can be obtained. This Zeiss instrument is already being used to study the finish on various materials produced by grinding wheels; to check the depth of scratches on polished limit gages; as an aid in finding the most efficient grinding, lapping, and polishing techniques to produce finished gage blocks; and to determine whether stoning has completely removed nicks and scratches. It has proved very useful in determining the amount and type of wear on contact surfaces. The pits, flats, raised spots, and out-of-roundness on steel balls and cylinders can be seen readily. Metallurgists are finding it useful in studying the relative levels of crystals and microconstituents of metallographically prepared surfaces.

The interference micrographs shown in this paper indicate possible uses of the microinterferometer for electroplating and related processes. For example, the instrument might be used to study the smoothing or brightening action of electrodeposition, electropolishing, or mechanical finishing, such as buffing and polishing. It might also be useful in specifications for surface finishes.

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Determination of Planeness and Bending of Optical Flats

Walter B. Emerson

The true contours, undistorted by gravitational bending, were determined for four 10 $\frac{5}{8}$ -inch-diameter standard optical flats of fused quartz. The bending deflections of these flats were determined by a method based upon the differential bending with thickness of the flats. Bending deflection curves of a flat supported at three points equidistant from the center of the flat and equidistant from each other were obtained. The locus of the bending deflections at the center of a flat, similarly supported but with supports at different distances from the center, approximates a straight line. This paper describes the method used to obtain the true contours and the bending deflection curves of the flats, and compares the bending values so determined with theoretically derived values.

1. Introduction

One activity of the National Bureau of Standards is to determine the deviation from a plane of surfaces of optical flats submitted for calibration. Usually these are disks of fused quartz used as working standards by makers of optical elements. Disks 10 in. in diameter, with specified maximum deviation from a plane of 0.1 fringe (approximately 0.000,001 in.) are frequently submitted. These are tested by interferometric comparison with the Bureau's 10 $\frac{5}{8}$ -in.-diameter fused quartz standard planes, the contour of which should be known to a few hundredths of a fringe to make the tests valid to the required tolerance.

It is commonly assumed that by intercomparison of three surfaces, the deviation of each surface from a plane may be determined. This is true if the flats are similarly supported and are identical except for differences in surface contour. Otherwise, consideration must be given to the deflection of each flat when supported in its position of test. Even in the case of support of the lower flat in mercury, some bending attributable to nonuniform distribution of forces by the weight of the upper flat and by surface-tension effects at the edge of the lower flat is to be expected. The theory of the bending of circular plates has been developed by Nádai [1, 2, 3]¹ and Timoshenko [4], and from these theoretical considerations an equation was formulated for circular plates supported at the vertices of an inscribed equilateral triangle whereby the gravitational bending deflection along a diametric line parallel to two of the supports may be determined with respect to a plane passing through the points of support. The results, based on a value $\nu=0.14$ for Poisson's ratio, are plotted as a dimensionless function from which the bending along a given diameter of a quartz plate may be readily determined.

In addition to deflections caused by bending, deflections are present because of shearing stresses and stress normal to the face of the plate. When these stresses are all taken into account for the case of a plate simply supported at the edges, Timoshenko [4] gives the following equation for the lateral deflection:

$$w = \frac{q}{64D} (a^2 - r^2) \left(\frac{5 + \nu}{1 + \nu} a^2 - r^2 \right) + \frac{qh^2}{8D} \frac{3 + \nu}{6(1 - \nu^2)} (a^2 - r^2), \quad (1)$$

where

$$\begin{aligned} w &= \text{lateral deflection,} \\ q &= \text{lateral pressure,} \\ \bar{D} &= \text{flexural rigidity} = Eh^3/12(1 - \nu^2), \\ E &= \text{Young's modulus,} \\ a &= \text{plate radius,} \\ r &= \text{distance from center,} \\ h &= \text{plate thickness,} \\ \nu &= \text{Poisson's ratio.} \end{aligned}$$

For plates acted on by gravitational forces, the lateral pressure is given by

$$q = \rho gh, \quad (2)$$

where ρ is the density and g the acceleration of gravity. Substituting (2) into (1) and replacing \bar{D} by $Eh^3/12(1 - \nu^2)$ gives

$$w = \left(\frac{1}{h^2} \right) \frac{\rho g}{16E} 3(1 - \nu^2)(a^2 - r^2) \left(\frac{5 + \nu}{1 + \nu} a^2 - r^2 \right) + \frac{\rho g}{4E} (3 + \nu)(a^2 - r^2). \quad (3)$$

It is seen that the first term in this equation varies inversely as the thickness squared and the second term is independent of thickness. Relations similar to (3), but depending differently on r , apply when the plate is supported by other means than simple support. One can express the deflection of the plate as

$$w = \frac{1}{h^2} f_b(r) + f_s(r), \quad (4)$$

where the functions f_b , for bending, and f_s , for shear and lateral pressure depend only on the means of support.

It should be pointed out that eq (3) does not include the effect of local deformation near points of concentrated load. These will be shown to be small, by measurement, for the plates tested.

Relationship (4) furnishes a basis for determining the true contour, undistorted by bending, shear, and lateral pressure effects, of the surfaces of optical flats.

A method for determining the *true contour* and the *bending* (the first term in relation (4)) of optical flats is presented and this method is applied to determinations of contour and to evaluation of the bending of four optical flats, and compares the bending values so determined with the values obtained according to theory.

¹ Figures in brackets indicate the literature references at the end of this paper.

2. Methods

2.1 Determination of True Contours

If a circular plate, resting on supports at the vertices of an inscribed equilateral triangle, supports a similar plate on supports directly above those for the lower plate, the surfaces of the plates tend to sag. If the surfaces were originally plane, the upper surface of the lower plate will now be concave, and the adjacent surface of the upper plate will be convex by an equal amount if the plates are of equal thickness, and by a greater amount if the upper plate is thinner. With three transparent plates of like material, properties, and diameter the *true contour* of their surfaces (that is, if the plates were supported uniformly and did not bend) may be determined by intercomparison of the plates, but no knowledge of the amount the plates bend will be obtainable. However, intercomparison of a series of plates that differ only in thickness will yield not only the true contour of the surfaces but also an evaluation of the change in contour (bending deflection) of the surfaces caused by the weight of the plates.

The following fused-quartz optical flats having a nominal diameter of 10 5/8 in. were available for intercomparison in this investigation:

Flat number	Diameter	Thickness (<i>t</i>)	ρ
	<i>Inches</i>	<i>Inches</i>	
1-----	10.66	1.428	2.039
2-----	10.61	1.626	2.644
3-----	10.59	2.50	6.250
4-----	10.58	2.50	6.250

Flats 1 and 2, which have served as standard planes at the Bureau for the past 30 years, contain numerous strias and inclusions, and the quality of their materials is inferior to that of flat 3, which was recently purchased, and also of flat 4, which was loaned for this investigation by the maker of flat 3. Flats 3 and 4 are slightly wedge-shaped, and the listed thickness is the average of measurements at several places around the circumference. For the purpose of this investigation, flats 3 and 4 are considered identical, except for difference in contour.

Based on the relationship that the bending deflection varies inversely as the square of the thickness, the deflection of flat 1 will be 1.30 times that of flat 2 and 3.07 times that of 3 or 4; the deflection of flat 2 will be 2.36 times that of 3 or 4.

By intercomparing flats 1, 2, and 3 in the relative positions shown in figure 1 (series 1) the true contour of flat 1 may be obtained directly from interferometric measurements of the sum of the contours of adjacent surfaces of the supported flats.

Let:

Each flat be supported at three points equidistant from the center and equidistant from each other; the supports for the upper flat be directly above those

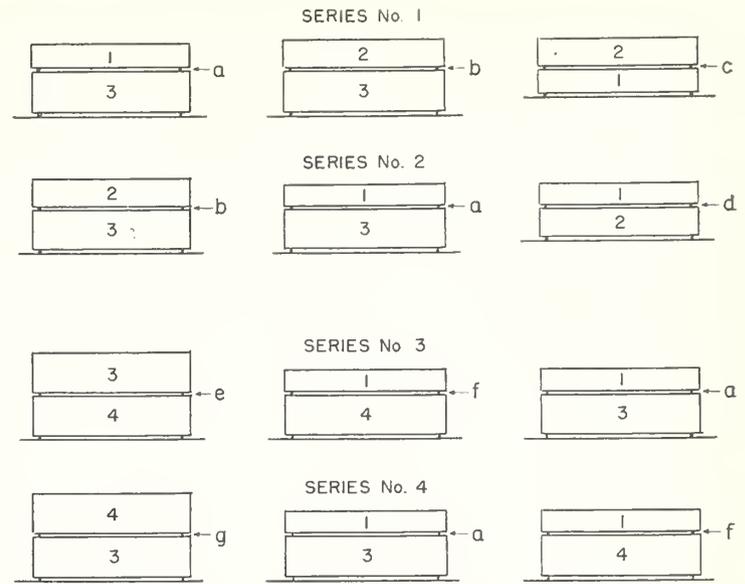


Figure 1. Series 1—Combinations of flats to determine the true contour of Numbers 1, 2, 3, and 4.

for the lower flat; the algebraic sum of the contours of adjacent surfaces be measured along a diametric line parallel to two of the supports of each flat;

$C_1; C_2; C_3$ =true contours, undistorted by bending, of flats 1, 2, and 3;

$a; b; c$ =measured sum of the contours of 1 on 3; 2 on 3; and 2 on 1, respectively;

$\pm D$ =bending deflection of flat 3 (also of 4);

$\pm D_1$ =bending deflection of flat 1= $\pm 3.07 D$;

$\pm D_2$ =bending deflection of flat 2= $\pm 2.36 D$;

S =shearing deflection of each plate;

+value=downward deflection;

-value=upward deflection.

Then:

$$C_1 + D_1 + S + C_3 - D - S = a$$

$$C_2 + D_2 + S + C_3 - D - S = b$$

$$C_2 + D_2 + S + C_1 - D_1 - S = c$$

and:

$$C_1 = \frac{a - b + c}{2}$$

$$C_2 - 0.71D = \frac{-a + b + c}{2}$$

$$C_3 + 2.07D = \frac{a + b - c}{2}$$

Whereas C_1 is obtained directly in terms of measurable quantities a, b, c , values for C_2 and C_3 cannot be determined from these measurements unless the bending deflection D is known. Determinations that require correction for D will be referred to as *indirect determinations*.

Similarly, series 2, 3, and 4, figure 1, yield direct values of C_2, C_3 , and C_4 , respectively, and yield also indirect values of C_1, C_3 , and C_4 .

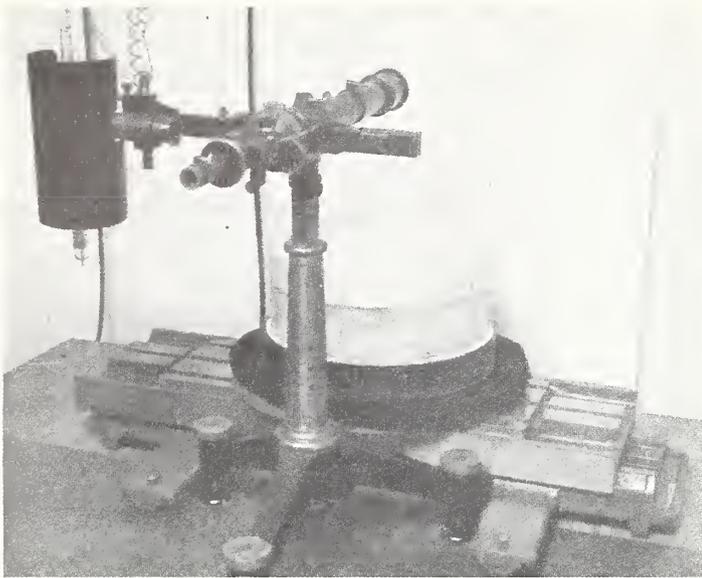


FIGURE 2. Setup for the determinations of contour and bending.

2.2. Determination of Bending Deflection

Since the bending deflection (given by the first term of eq (4) varies inversely as the thickness squared the bending deflections D_1 and D of flats 1 and 3, respectively, may be determined from measurements of the sum of the contours with flat 1 above and beneath flat 3. If the measured values are a with flat 1 above and a' with flat 3 above:

$$C_1 + 3.07D + S + C_3 - D - S = a; \quad C_1 + C_3 + 2.07D = a; \quad (5)$$

and

$$C_3 + D + S + C_1 - 3.07D - S = a'; \quad C_1 + C_3 - 2.07D = a'. \quad (6)$$

From eq (5) and (6),

$$4.14D = a - a'; \quad D = 0.242 (a - a'); \quad D_1 = 0.743 (a - a').$$

Had a thinner flat, 0.75 in. thick, been available for comparison with flat 3, the value $(a - a')$, from the measurements would equal 20.2 times the deflection of flat 3, thus increasing appreciably the accuracy of the determinations of bending.

3. Procedure for Comparing the Flats

One flat was supported, with its optical surface up, by three 1/8-in.-diameter paper disks spaced equidistant from the center of the flat and equidistant from each other on the glass plate shown in figure 2. A second flat, with its optical surface down, was supported on the first flat by three similar disks directly above the supports of the lower flat. The combination was placed on a movable carriage beneath a Pulfrich viewing instrument [5]. By means of a hand crank operating through reduction gears, the combination could be moved slowly and smoothly across the field of the viewing instrument, which

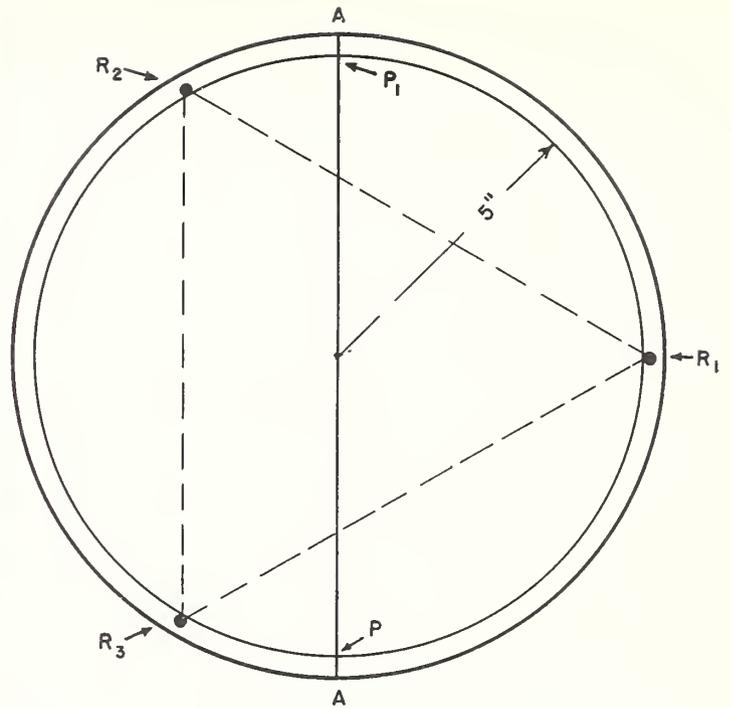


FIGURE 3. Position of diametric line A-A relative to the points of support, and of points P and P₁ between which the measurements were made.

served both as a source of monochromatic light and as a means for measuring deviations of the resulting fringes from a straight line, that is, the algebraic sum of the contours of adjacent surfaces. Desired fringe widths were obtained by selecting paper disks of proper relative thickness. Widths corresponding to 300 divisions of the micrometer head of the viewing instrument proved satisfactory in this work.

Measurements for each combination of flats were made between points P and P₁, figure 3, which are 5 in. from the center and lie on a definite diametric line AA that is parallel to supports R₂ and R₃. The sum of the contours was determined for the inner 10-in. diameter surface of the flats rather than for the entire surface because of irregularities in the outlying area that might affect the accuracy of measurements.

The deviation of a fringe from a straight line passing through the intersections of that fringe with the circumference of the 10-in. surface was measured in terms of divisions of the micrometer head of the viewing instrument. The number of divisions thus measured (given the proper sign to indicate the direction of curvature) divided by the divisions between successive fringes is the algebraic sum of the contours in terms of fringes. Determinations were made at eleven points along the diameter.

Comparisons were made in a temperature-controlled room with the flats enclosed in an insulated container (not shown in fig. 2) covered inside and outside with aluminum-coated paper. By uncovering a small opening in the top of the container, fringes were observed and measured. The usual procedure was to set up the flats during the forenoon, measure the combination several times in the afternoon when thermal conditions were stabilized, and

repeat the observations the following morning, thus completing one set of observations. The flats were then reset and the procedure repeated. The final values for the contours are based on an average of five sets of observations for each combination of flats. The maximum deviation of the individual sets from the mean for a given combination in no case exceeded 0.017 fringe for measurements at the center; and the average deviation from the mean was 0.005 fringe.

4. Measurements and Results

4.1 True Contours

a. Direct Determinations

To test the validity of the methods described in section 2.1 for determining the true contours of the four standard flats, the differences a, b, c, d, e, f, g (fig. 1, series 1, 2, 3, and 4) were determined by interferometric comparison when the flats were supported at three points located as follows:

5.06 in. ($0.95 \times$ radius r) from the center

3.5 in. ($0.66 \times$ radius r) from the center.

From these measurements the true contours of the flats were obtained from equations

$$C_1 = \frac{a-b+c}{2}, \quad C_2 = \frac{b-a+d}{2},$$

$$C_3 = \frac{e-f+a}{2}, \quad C_4 = \frac{-a+f+g}{2}.$$

Additional values were obtained with the flats supported at $0.95r$ by substituting flat 2 for flat 1 in series 3 and 4, and flat 4 for flat 3 in series 1 and 2.

The resulting determinations of the true contour of flat 1 are given in table 1. The two determinations at $0.95r$, columns 2 and 3, differ by only a few units in the third decimal place. Column 4 gives the mean of the determinations at $0.95r$. The contours determined with supports at $0.66r$ are given in column 5. Column 6 gives the difference between the contours determined for the two conditions of support, and indicates that true contours obtained by this method are nearly the same whether determined with supports at $0.95r$ or at $0.66r$. Column 7 gives the mean of the direct determinations of true contour for these two supports.

The magnitude of the differences given in columns 4 and 6 for this flat are representative of those similarly obtained for flats 2, 3, and 4.

b. Indirect determinations

Intercomparison of the various combinations in section 2.1 for different positions of the supports yields some equations that include terms $0.71D$, $1.36D$, or $2.07D$, where D is the bending deflection of 2.5-in.-thick flats 3 or 4 at points between P and P_1 , figure 3, relative to the bending deflection at points P, P_1 . Values for D were obtained from determinations described in section 4.2. The magnitude of D will depend upon the position of the supports and the place of measurement. By substituting the values thus determined for D in the equations that include a term for the bending deflection, additional values of the true contours of the four flats were obtained.

TABLE 1. True contour (in fringes) of the central 10-in.-diameter surface of optical flat 1 by direct and indirect methods

+value=convexity; -value=concavity

Distance from P	Direct determinations						Indirect determinations (corrected for bending term)								Difference 7-16	Contour (mean 7 and 16)	
	Position of supports			Difference 4-5	Mean 4 and 5	Position of supports				Difference 11-14	Mean 11 and 14						
	0.95r	Mean 2 and 3	0.66r			0.95r		0.66r									
				For corresponding bending terms—		Mean 8, 9 and 10	For corresponding bending terms—		Mean 12 and 13								
	(2.07D)	(0.71D)	(0.71D)	(2.07D)	(0.71D)												
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
<i>Inches</i>																	
0.0	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
.5	-.021	-.019	-.020	-.021	+.001	-.021	-.023	-.027	-.025	-.025	-.024	-.027	-.026	+.001	-.026	-.005	-.024
1.5	-.069	-.065	-.067	-.061	-.006	-.064	-.062	-.068	-.064	-.065	-.067	-.072	-.070	+.005	-.068	+.004	-.066
2.5	-.084	-.081	-.083	-.079	-.004	-.081	-.077	-.080	-.077	-.078	-.082	-.085	-.084	+.005	-.081	.000	-.081
3.5	-.111	-.112	-.112	-.104	-.008	-.108	-.107	-.108	-.109	-.108	-.110	-.114	-.112	+.004	-.110	+.002	-.109
4.5	-.137	-.135	-.136	-.133	-.003	-.135	-.136	-.135	-.133	-.135	-.135	-.136	-.136	+.001	-.136	+.001	-.136
5.0	-.132	-.128	-.130	-.129	-.001	-.130	-.128	-.129	-.125	-.128	-.130	-.129	-.130	+.002	-.129	-.001	-.130
5.5	-.127	-.127	-.127	-.125	-.002	-.126	-.124	-.124	-.125	-.124	-.127	-.127	-.127	+.003	-.126	.000	-.126
6.5	-.108	-.110	-.109	-.108	-.001	-.109	-.109	-.102	-.105	-.105	-.110	-.110	-.110	+.005	-.108	-.001	-.109
7.5	-.079	-.077	-.078	-.072	-.006	-.075	-.074	-.075	-.073	-.074	-.074	-.074	-.074	.000	-.074	-.001	-.075
8.5	-.053	-.053	-.053	-.055	+.002	-.054	-.053	-.050	-.049	-.051	-.054	-.054	-.054	+.003	-.053	-.000	-.054
9.5	-.022	-.021	-.022	-.024	+.002	-.023	-.024	-.022	-.021	-.022	-.026	-.026	-.026	+.004	-.024	+.001	-.024
10.0	.000	.000	.000	.000	.000	.000	.000	.000	.000	.000	.000	.000	.000	.000	.000	.000	.000

The indirect determinations for flat 1 with supports at $0.95r$ are given in table 1, columns 8, 9, 10, and with supports at $0.66r$, in columns 12, and 13. Columns 11 and 14 give the means of determinations at $0.95r$ and $0.66r$, respectively. Column 15 gives the difference between the mean values at $0.95r$ and $0.66r$. As with the direct determinations, the individual indirect determinations differ from their respective mean values in the third decimal place, and likewise the differences in their mean values are small notwithstanding the large differences in bending term values applied in the equations. Column 16 gives the mean of the indirect determinations for the flats supported at $0.95r$ and at $0.66r$. The small differences between the direct values (col. 7) and the indirect values (col. 16) shown in column 17 are attributed to experimental errors. Equally small differences between direct and indirect values were obtained for flats 2, 3, and 4. Column 18 gives the true contour of flat 1, and is taken as the average of the direct and indirect determinations. The true contours of the four standard flats are shown in figure 4.

4.2 Bending of Flats

The bending deflection was determined from comparisons of flats 1 and 3 by the method described in section 2.2.

These flats were supported as previously on 1/8-in.-diameter paper disks at three points equidistant from the center of the flat and equidistant from each other. The deviation of a fringe from a straight line was measured for the inner 10-in.-diameter surfaces along a diametric line parallel to two of the supports of each flat: first with flat 1 above; and second with flat 3 above. The measured deviations were a and a' , respectively.

To determine the effect on bending when the flats were supported at different distances from the center, tests were made with supports at the following distances: 1.0 in. ($0.19r$); 1.5 in. ($0.28r$); 2.5 in. ($0.47r$); 3.5 in. ($0.66r$); 4.25 in. ($0.80r$); 5.06 in. ($0.95r$)

The bending deflection of flat 1 was calculated from equation $D_1 = 0.743(a - a')$, except for the $0.19r$ support, for which no value of a' was determined.² The deflection for this support was calculated from $D_1 = 1.483[a - (C_1 + C_3)]$, using the values previously determined for C_1 and C_3 .

Figure 5 gives the bending curves for the inner 10-in. surface of flat 1 supported at different distances from the center. The bending deflection at the center changed from a downward (+) deflection of 0.143 fringe for supports at $0.95r$ to an upward

² Support near the center appears to be a severe condition that might cause permanent change in the flat, and therefore should, in general, be avoided. Previous tests of flats supported 0.5 in. from the center gave a pronounced peak distortion at the center. In the present instance, flats 1 and 3 were supported for 1 day at $0.19r$. A month later, when the flats were supported at $0.95r$ and compared, the measured values for a and a' differed from previous values by approximately -0.03 fringe, thus indicating either a permanent or temporary differential change in the flats. During the following month, the flats were supported at $0.95r$, that is, in a position in which the deflection is in a direction opposite to that with supports near the center. At the end of the month measured values of a and a' approached closely the original values, indicating that the change may have been caused wholly or in part by plastic flow of the fused quartz. As a subject for investigation, it appears important to determine, with flats other than the Bureau's standard planes, the nature of the changes in contour that occur when flats are supported in extreme positions.

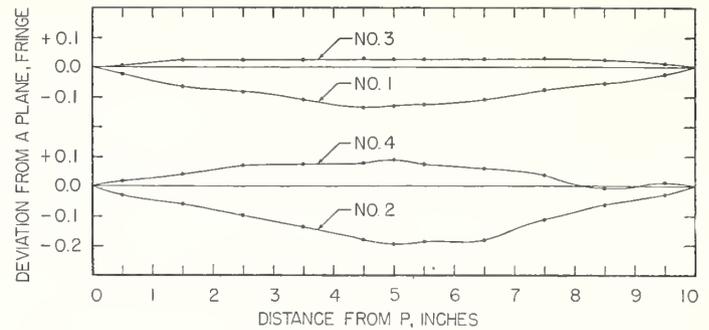


FIGURE 4. True contours of the four standard optical flats. (+ Value indicates convexity; - value indicates concavity).

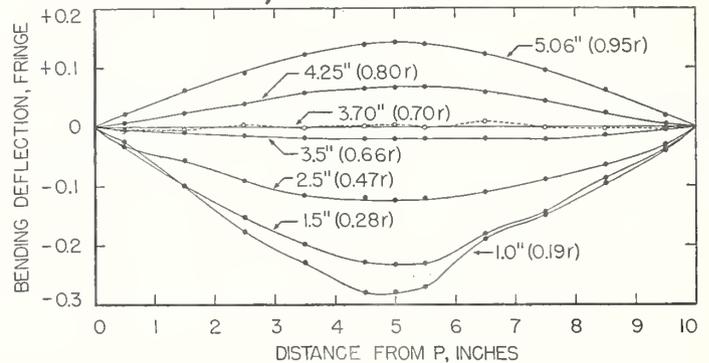


FIGURE 5.—Bending curves for flat Number 1 supported at different distances from its center.

(+ Value = downward deflection; - value = upward deflection)

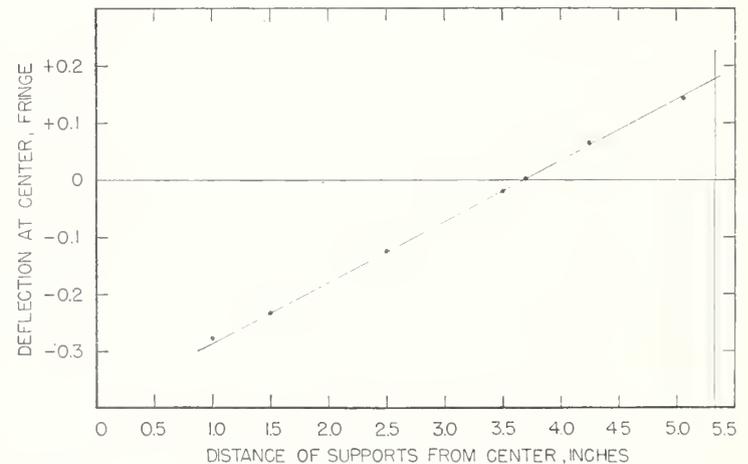


FIGURE 6.—Bending deflection at the center of Number 1 relative to points P, P₁ 5 in. from the center, for supports at different distances from the center.

(-) deflection of 0.278 fringe for supports at $0.19r$. Inspection of the curves indicated that the bending deflection of a flat supported at $0.70r$ would be quite small. Determinations were then made for this condition. The results, which are shown with the other curves in figure 5, indicate that the bending along the diametric line does not exceed 0.01 fringe for flat 1. The bending of thicker flats 3 and 4 when supported at $0.70r$ could be considered negligible.

The bending deflections at the center of the surface of flat 1 when plotted for the different supports appear to fall approximately on a straight line, figure 6. By projecting the line to intersect the circumfer-

ence of this 10.66-in.-diameter flat, the bending deflection that would result, if the flat were supported at its edge, is obtained. The best value for the bending deflection, W_E , at the center of flat 1 with respect to points on the diametric line 5 in. from the center (that is, for the inner 10-in. surface) when the flat is supported at its edge appears to be

$$W_E = 0.177 \text{ fringe} = 2.05 \times 10^{-6} \text{ in.}$$

The data given in figures 5 and 6 may have application when very accurate comparisons of a standard flat and a test flat of less diameter are required. The test flat in this case may rest on supports at 0.70 of its radius to give minimum bending, and the supports for the standard may be placed in vertical alignment with these. The true contour of the standard may then be corrected by the bending data to give its contour in the position of test.

5. Comparison of Theoretical and Experimental Values for Bending Deflection

Figure 7 is the theoretical curve for the bending deflection at the center of a 10-in. diameter inner surface of flat 1 along AA with respect to a plane defined by the three symmetrically spaced points of support BBB of a uniformly loaded fused-quartz plate.

Constants for flat 1:³ $a = 5.33$ in.; $h = 1.428$ in.; $P = 10.18$ lb; $E = 10.15 \times 10^6$ psi; $\nu = 0.14$.

From figure 7, $w(Eh^3/Pa^2)$ at the center of flat 1 ($r/a = 0$) with respect to a plane through the points of support is 0.424; or

$$w = 4.15 \times 10^{-6} \text{ in.} \quad (7)$$

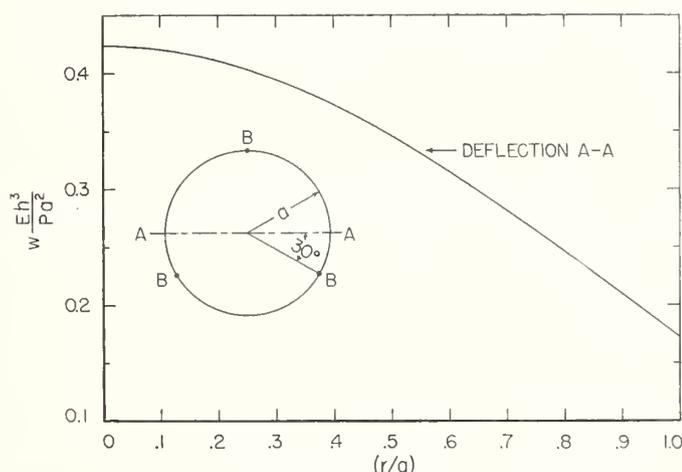


FIGURE 7. Bending deflection of uniformly loaded plate supported at three equally spaced points on circumference BBB.

E , Modulus of elasticity; P , weight of plate; a , radius of plate; h , thickness of plate; r , distance from center; ν , 0.14, Poisson's ratio for fused quartz; w , deflection along A-A relative to BBB.

³ Values for E and ν are those considered by Sosman [6] as the best values for used quartz.

Similarly, $w(Eh^3/Pa^2)$ at a point along AA 5 in. from the center ($r/a = 0.94$) is 0.195; or

$$w_1 = 1.91 \times 10^{-6} \text{ in.} \quad (8)$$

The difference between (7) and (8), or w_T is the bending deflection at the center of flat 1 with respect to points P and P₁ on AA that are 5 in. from the center; or $w_T = 2.24 \times 10^{-6}$ in. The corresponding deflection w_E determined experimentally (section 4.2) is $w_E = 2.05 \times 10^{-6}$ in.; that is, the theoretical value is between 9 and 10 percent greater than that determined experimentally.

In order to determine the relative shapes of theoretical and experimental bending curves for comparable conditions of support, the deflections given in figure 5 for the bending curve of flat 1 supported at $0.95r$ were increased by the ratio 177/143 (see section 4.2) to approximate the condition of edge support for which the theory applies.

Figure 8 shows that the theoretical and the experimental bending curves are similar in shape but differ in magnitude of the deflections. The values derived by theory average about 11 percent greater than those obtained by the method used in this investigation.

This agreement between the theoretical and the experimental values for bending appears satisfactory in view of the many factors that might influence either result. The experimental values would be affected by small differences in setting the flats, errors of measurement, insufficient number of measurements taken to integrate small local variations in optical surfaces as noted by Saunders [7], and the use of supports of finite size rather than point supports. Likewise, the theoretical values would be different were other values used for E and ν . The values determined for these elastic constants of fused quartz by different observers vary appreciably as shown in a summary by Sosman [6]. He states, "A study of the assemblage as a whole will show there

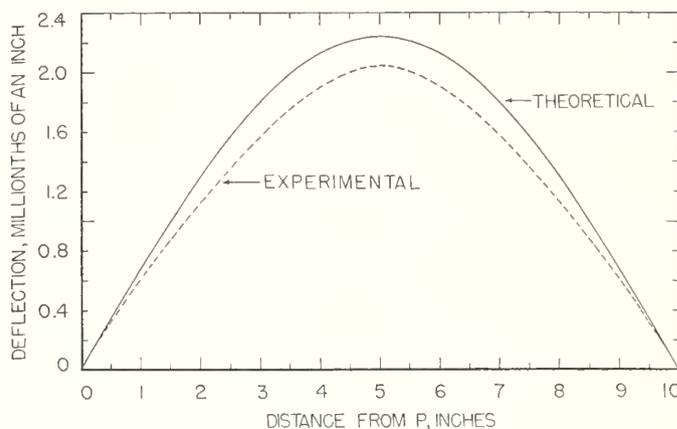


FIGURE 8.—Theoretical and experimental bending curves of flat Number 1 supported at three equally spaced points on its circumference. The curves give the difference in the bending deflection at points along a diametric line that is parallel to two of the supports and the deflection at points on the same line that are 5 in. from the center of the flat. Bending deflections relative to the points of support may be obtained by increasing the theoretical values 1.91×10^{-6} in. and by increasing the experimental values 1.7×10^{-6} in.

is not much consistency, either in the theory or experiment among the various values. Part of the reason, I feel sure, is that the usual specimen of vitreous silica is not structurally isotropic, as we have been assuming, but is to a greater or less degree anisotropic." From this consideration alone, the values of ν and E used to calculate the theoretical bending may not apply to the standard flats.

Substitution of the theoretical value for bending for the experimental value would change the true contour at the center of flat 1, as given in table 1 for supports at $0.95r$, by 0.01 fringe in those cases where corrections of $2.07D$ are applied, and would vitiate to some extent the excellent agreement of direct and indirect determinations of contour. The determined bending deflection values may, therefore, be more applicable to the present flats and their conditions of test than the theoretical ones.

6. Effect of Supports of Finite Size

When flats supported on balls are compared, a series of interference fringes concentric about the point of contact of support and surface is visible, indicating an appreciable deformation of the surface concentrated at the point of contact. The reason for using 1/8-in.-diameter paper supports in the present work was to distribute the load over a larger area and thereby avoid damage to the flats. Local distortion of the surface surrounding the disks for about 3/32 in. beyond the supports was evident when viewing the fringes. At the edge of a disk, the surface of the flat was depressed about 0.3 fringe. It is quite reasonable that the deflection may be somewhat less when flats are supported on the disks rather than points, thus giving a lower value than that required by theory. To test this, from consideration of contour, bending, shear, local deformation at the supports, and thickness of the flats, the following equation was derived, in which contour, shear, and the effect of the supports are eliminated.

$$\frac{D_{in.}}{t_x^2} = \frac{1}{2} \frac{t_z - t_x}{t_x + t_y} \frac{b' - b}{t_y^2} - \frac{a' - a}{t_x + t_z} \frac{t_y - t_x}{t_z^2}$$

in which $D_{in.}$ is the bending deflection of a plate 1 in. thick and t_x, t_y, t_z = thickness of plates X, Y, Z, and $D_{in.}/t_x^2$ is the bending deflection of X. From comparisons of X and Y: a is the deviation of a fringe from a straight line, with X above, and a' is the deviation of a fringe from a straight line, with Y above. From comparisons of X and Z: b is the measured deviation, with X above, and b' is the measured deviation, with Z above.

The difference between the values for bending previously determined and those by this method was

less than the experimental error of this method. No effect of local deformation on the bending deflection was therefore indicated by these experiments.

The excellent agreement between contours obtained with support at $0.95r$ and at $0.66r$, table 1, indicates that local deformation near the supports had negligible effect on the contour measurements.

7. Conclusion

Precise calibration of optical flats requires that account be taken of the surface distortion that results when the flats are supported in their position of test. For a 10 5/8-in.-diameter, 1 7/16-in.-thick fused-quartz optical flat supported at three equally spaced points at its edge, the gravitational bending deflection at the center of the flat with respect to its points of support will be approximately 4×10^{-6} in. To reduce this to 1×10^{-6} in. (0.1 fringe) would require a thickness of 2.9 in. on the basis of theoretical computations, which agree quite closely with experimental determinations. The bending deflection will increase appreciably for larger flats because it varies directly as the fourth power of the radius. For three-point support, bending is small and at a minimum when the supports lie on the circumference of a circle having a radius 0.7 that of the flat.

Whenever two flats of the same material and diameter but of different thicknesses are available and can be similarly supported during comparison, the bending deflection of each flat may be determined by the described methods. Likewise, if three flats of the same material and diameter are available, very precise determinations of the true contours, undistorted by bending of their surfaces, may be determined.

The author expresses his deep appreciation to Samuel Levy of the Engineering Mechanics Section of the Bureau for his cooperation in the theoretical determinations of the deflection of plates that were used in this investigation and for his many helpful suggestions.

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WASHINGTON, May 27, 1952.

Testing of Large Optical Surfaces With Small Test Plates

James B. Saunders

A procedure for testing large optical surfaces with relatively small optical standards is described. Simplified formulas are used to apply a statistical method for obtaining increased precision. A practical example is used to illustrate the procedure for testing surfaces that may be assumed to have revolution symmetry.

1. Introduction

With the increased use of massive optical parts, there is need for a practical method of testing large optical surfaces with relatively small standards, such as are available in the average optical shop. The conventional test of observing fringes between the standard and unknown at various positions on the surface enables the optician to form a rough estimate of the nature of the surface. For more precise values either extensions of the present conventional tests or new methods must be used. Present shop methods of testing optical surfaces are not practical for massive optical elements as standard test plates of the sizes required for conventional test methods are seldom available.

A test for shape of very large optical surfaces, by means of interference of light, may be made with a standard test plate (flat or spherical) that is much smaller than the surface to be tested. The principles involved are not new and may be considered elementary, but those who perform the tests on optical surfaces during the polishing are not as a rule sufficiently versed in mathematics to apply the principles outlined in the present paper to the best advantage. They can, however, apply observed data to a set of simple formulas and compute the ensuing results.

A procedure is described in which an ordinary-sized standard test plate can be used to measure the shape of surfaces that are much larger than the standard. Use is made of statistical methods for improving the computed results. Simplified formulas will be developed that may be applied by opticians, after a short training period, even though they may not be able to follow the mathematical derivations and theory. The application of the general formulas to a typical set of data will then be made to illustrate the testing of a surface by means of a smaller standard optical flat. The method is not limited to plane surfaces. The sphericity of very large spherical or aspherical surfaces may be measured with standard test plates of approximately the same radius, if the viewing system permits the use of normal incident light. Figure 1, (A, B, C) shows simple optical arrangements for testing plane, convex, and concave surfaces, respectively.

The basic principle may also be applied to the testing of off-axis curved, or nonsymmetrical, surfaces, with modifications of the formulas. However, as these are encountered only rarely, the present paper will be limited to surfaces of revolution.

We will consider a Fizeau viewer (fig. 1) that has an aperture equal to or greater than the area of the standard surface. Spherical surfaces are referred to the spherical master with which they are tested, and plane or approximately plane surfaces are referred to planes. The equations and measurements are identical for plane and spherical surfaces. This discussion will deal with the testing of an approximately plane surface against a standard optical flat. The unknown surface is assumed to be a figure of revolution about an axis normal to and passing through a known point on it. In general, this point will be at or near the center, if the surface is circular.

2. Experimental Procedures

If the standard flat is adjusted normally to the collimated beam of light and the unknown is placed close to and approximately parallel to the standard, interference fringes may be observed with monochromatic light of known wavelength. Measurements on the fringe pattern permit a computation of the shape of the unknown relative to a plane (sphere for spherical surfaces) over the visible region of interference. If adjustments permitted, the entire area of the unknown could be covered by

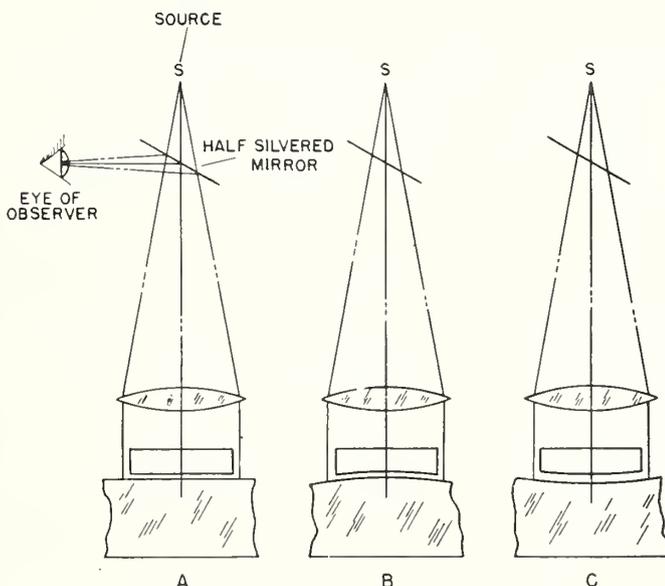


FIGURE 1. Optical arrangements for testing (A) flat surfaces, (B) convex surfaces, and (C) concave surfaces.

moving the standard step by step without rotation, in a plane parallel to the standard plane. The aggregate of the resultant fringe patterns, when properly assembled to form a composite pattern of the whole surface, would be quite similar to what would have been obtained with a standard surface that covered the whole of the unknown.

As it is virtually impossible to make the above assumed adjustments, corrections for rotation and changes in separation of the unknown, relative to the original surface of the standard, must be made by measuring these changes and applying the required corrections. The corrections are always subject to errors of observation. If several successive positionings of the standard, relative to the unknown, are required to measure the shape along a diameter, these errors accumulate. Consequently, it is desirable to reduce these errors. The "Method of Averages,"¹ which is easy to apply and yields simple working formulas, is used since it provides adequate precision.

In general, large optical surfaces are ground and polished by machines that produce figures of revolution about a known point, usually located at the center of the surface. If the surface is one of revolution,² its departure from a straight line that is tangent to it at the center of revolution is a measure of its departure from its tangent plane and consequently determines the shape of the entire surface relative to any other chosen plane. The axis of abscissas (see fig. 2) is chosen as the intersection of a plane through the axis of rotation of the surface with the plane that is tangent to the surface at its center. The unit of abscissas is chosen as the separation of equally spaced reference marks along a chosen diameter of the surface. The chosen diameter is the axis of abscissas. The axis of ordinates is the axis of revolution of the surface. The unit of ordinates is one-half the wavelength of the light used.

The following steps are the chosen procedure for acquiring the data necessary to compute the shape of the surface. The master flat is placed on, and concentric with, the surface (positions *A* and *A'*, fig. 2). The magnitude of the air wedge between the two surfaces is adjusted to produce a satisfactory number of fringes in the field (see fig. 3). The direction of this wedge is adjusted to make the fringes approximately normal to the line along which the chosen reference points lie. Except for plane surfaces this wedge varies from one reference point to the next. Consequently, if linear interpolation is used in estimating fractions of fringes at the reference points, the error in estimation varies with the fraction observed. For most observers this error is a minimum when the fraction is 0.0 or 0.5, that is, when the reference point falls on the center of a dark or a bright fringe. Best accuracy is obtained by reading abscissas corresponding to the center of all fringes—both dark and bright—and from these data, by nonlinear interpolation, compute the relative

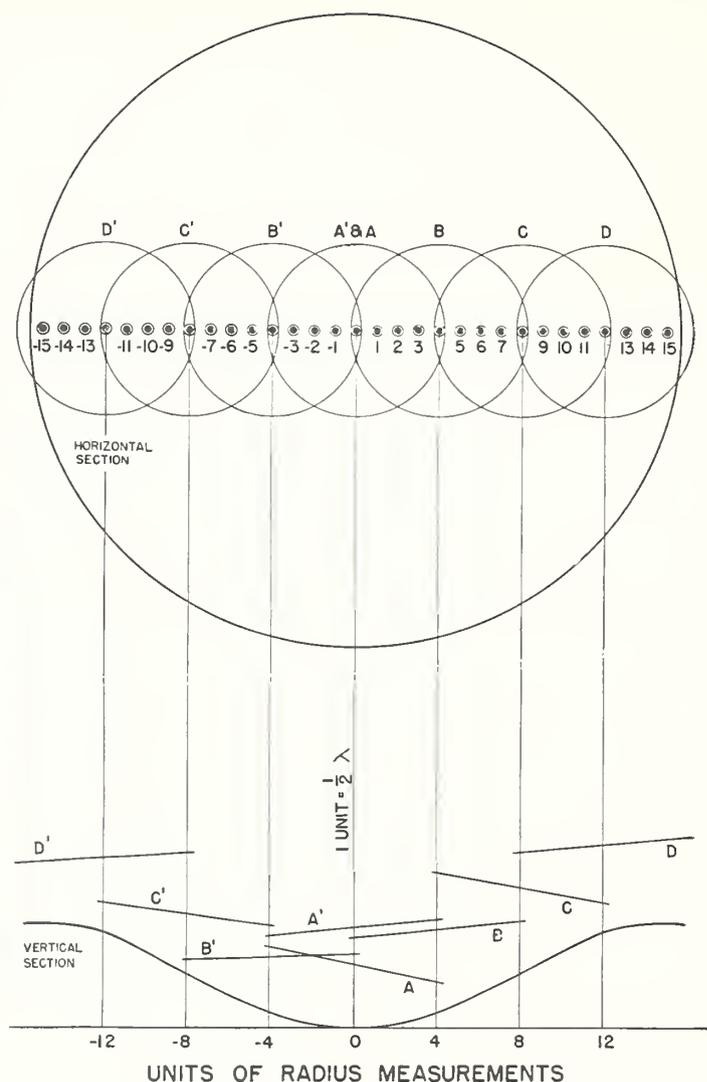


FIGURE 2. Coordinate system of reference.

The vertical section (below) cuts the horizontal section (above) along the line of reference points—dots concentric with small circles. Circles *A*, *A'*, *B*, *B'*, etc. represent the several positions of the standard test plate. The corresponding straight lines below represent sections through the standard surface of reference.

orders at the chosen reference points. The procedure will be explained in greater detail with the help of the fringe configuration shown in figure 3.

The fringes (relative orders of interference) in position *A* are evaluated at the several equally spaced reference points by the method indicated above. The standard is then moved to position *B*, which overlaps an appropriate amount of the area covered in its first position, *A*. Again the fringe readings at all reference points, covered by the standard in this new position, are evaluated. The standard is then moved to position *C* and the corresponding fringe readings noted. This procedure is repeated until the surface covered by the master extends to the edge of the unknown surface of revolution. The positions represented by primed letters are a second independent set, useful in checking the precision of the method.

In general, the fringe values at the reference points will not be integral. Consequently, the fractional parts must be obtained by interpolation and, in marginal cases, by extrapolation. Linear interpolation

¹ J. B. Scarborough, Numerical mathematical analysis, p. 446 (Johns Hopkins Press, Baltimore, Md., 1950).

² W. A. Granville, Elements of differential and integral calculus, p. 264 (Ginn & Co., New York, N. Y., 1911).

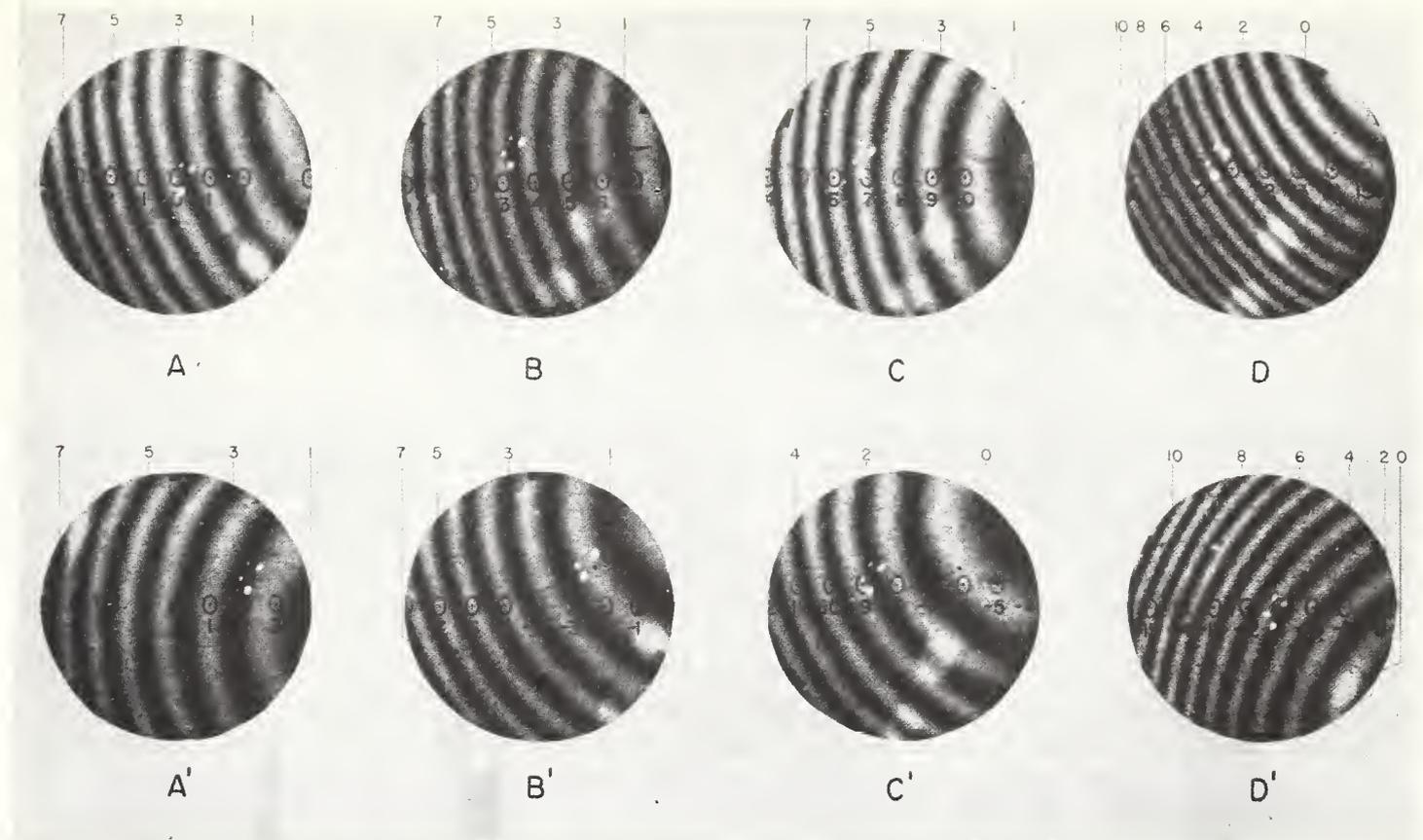


FIGURE 3. Fringes for the several positions of the standard test plate.

The numbers above each picture represent relative orders of interference.

or extrapolation is accurate only when the fringes are equally spaced along the straight line on which the reference points are chosen. If they are equally spaced, the surface is plane and no further test is necessary. In general, the fringes will be curved and nonlinear interpolation should be used if highest accuracy is to be obtained. Of the several methods of performing nonlinear interpolation, the graphical method is simplest and, for these purposes, adequate. The details of this are best shown by a description of its application to actual data. This will be given in a later section, where the shape of the surface of a glass disk will be computed from photographs of interference fringes that are obtained when the glass disk is tested interferometrically against a standard flat.

3. Derivation of Formulas

A set of formulas are now derived for use in the above-mentioned computation. In figure 4 the curved line represents the surface to be measured. The chosen reference plane is represented by the axis of abscissas. The chosen reference points are indicated by circles on the curved line. The several positions of the master plane relative to the coordinate system are represented by straight lines at various angles. The coordinates of all chosen reference points, which are marked on the surface of the unknown, are (R, Y_R) . The observed fringe (a relative order of interference) at (R, Y_R) is designated

A_R for the first position of the standard, B_R for the second position of the standard, C_R for the third position, and so forth. The slopes and intersections of the straight lines with the axis of ordinates are represented by M_A and A_0 , respectively, for the A position, M_B and B_0 for the B position, M_C and C_0 for the C position, and so forth.

The slope-intercept form of the equation for the straight line representing the standard in the X position ($X=A, B, C$, etc.) is

$$Y_R + (X_R - X_0) = M_X R. \quad (1)$$

In position A , for example, the ordinates of the line are $(A_R + Y_R) = A_0 + M_A R$, where A_R is the separation of the two optical surfaces. The absolute values for X_R are unknown, but differences in X_R , for any given position of the standard, are directly observable. Consequently, the integral part of the smallest X_R will be subtracted from all X_R 's for purpose of computation. The quantity $(X_R - X_0)$ is unaffected by this operation.

We will evaluate M_X and X_0 by applying the method of averages (footnote 1) to the data. The grouping of observations will be made in such manner that there is no overlapping of the two groups of points except when an odd number of reference points are to be applied to an evaluation. When the number of reference points in the two groups is odd, two observations are considered to be made at each of the points, and the two observations made at the central

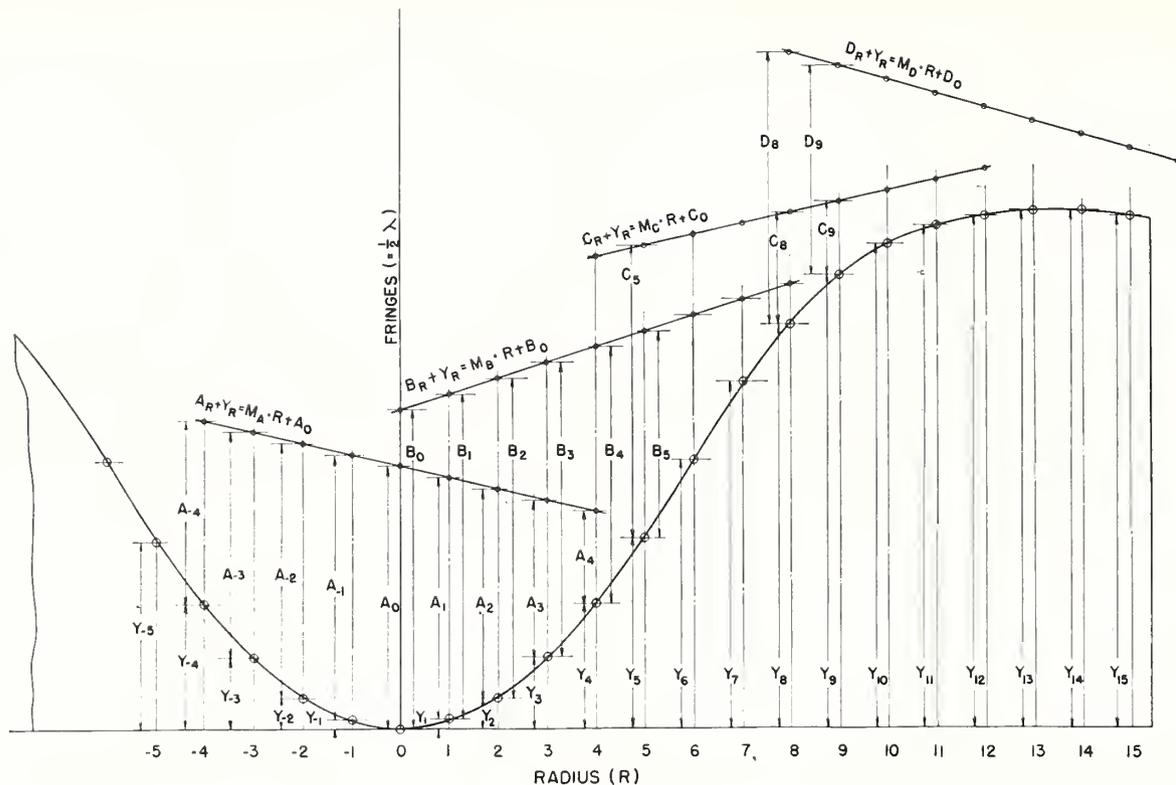


FIGURE 4. Analytical representation of the surface and standard for mathematical analysis.

The slanting straight lines represent the several positions of the standard and the adjacent formulas the equations of its corresponding loci.

point are divided between the two groups. The two groups then have an equal number of observations, and all observations are thereby assigned equal weights. When the total number of reference points is even, there is no overlapping of groups for an equal number of points to be allocated to each of the two groups for equal weighting of datum.

In applying the method of averages (footnote 1), we require that the algebraic sum of errors be zero for each of the two groups of observations. This requirement is represented by the following equations:

$$\left. \begin{aligned} \sum_{R_1}^{R_2} (Y_R + X_R) &= M_X \sum_{R_1}^{R_2} R + \frac{1}{2} N_X X_0, \\ \sum_{R_3}^{R_4} (Y_R + X_R) &= M_X \sum_{R_3}^{R_4} R + \frac{1}{2} N_X X_0, \end{aligned} \right\} \quad (2)$$

where summations in the first of this pair of equations are from the lowest value of $R (= R_1)$ to the largest value of $R (= R_2)$ in this group and summations in the second equation are from the lowest value of $R (= R_3)$ in the second group to the largest value of $R (= R_4)$ in the second group. Equality of R_2 and R_3 will result when the number of reference points is odd, whereas they will differ by unity when the total number of reference points is even. The number of reference points used in each evaluation is N_X .

Solving for M_X and X_0 from the pair of equations (2), we get

$$M_X = \frac{\sum_{R_3}^{R_4} (Y_R + X_R) - \sum_{R_1}^{R_2} (Y_R + X_R)}{\sum_{R_3}^{R_4} R - \sum_{R_1}^{R_2} R} \quad (3)$$

$$X_0 = \frac{2 \left[\sum_{R_3}^{R_4} R \sum_{R_1}^{R_2} (Y_R + X_R) - \sum_{R_1}^{R_2} R \sum_{R_3}^{R_4} (Y_R + X_R) \right]}{N_X \left[\sum_{R_3}^{R_4} R - \sum_{R_1}^{R_2} R \right]} \quad (4)$$

Equations (1), (3), and (4), together with the assumption that the surface to be measured has revolution symmetry, permit a computation of Y_R for all chosen reference points and consequently the shape of the surface.

The foregoing analysis permits a statistical evaluation of M_X and X_0 for all values of X (i. e., A, B, C, \dots) except the quantity A_0 . An error in A_0 represents an error in the position of the reference plane and since this has no effect on the computed shape of the surface, the observed A_0 is assumed to be free from error.

As the surface is assumed to have revolution symmetry,

$$Y_R = Y_{-R}. \quad (5)$$

From eq (1) $Y_R = M_A R + (A_0 - A_R)$, and $Y_{-R} = -M_A R + (A_0 - A_{-R})$. On eliminating M_A and Y_R from these three equations, we have

$$Y_R = A_0 - \frac{1}{2} (A_R + A_{-R}). \quad (6)$$

As M_A does not enter into the computation, it need not be evaluated.

4. Illustrative Example

Equations (1), (3), (4), and (6) form a set of fundamental equations from which all values for Y_R may be computed.

To clarify any possible lack of understanding in the development and application of these formulas, we will apply them to a set of observations and will compute the resultant shape of a surface. In this case the large circle in figure 2 represents the periphery of a 31.1-cm-diameter disk that is to be measured by means of a standard whose diameter is 8.2 cm. The seven circles, centered on one diameter of the large disk, represent the several successive positions of the standard. The eight circular fringe patterns shown in figure 3 (forming two independent sets, primed and unprimed) were obtained by photographing the fringe patterns formed by light reflected normally from the top surface of the large disk and the standard flat when in the several positions indicated in figure 2. The differences in values of the ordinates ($Y_R - Y_{-R}$), obtained from the two sets of photographs, is a measure of the accuracy obtainable. These differences are due to errors of observation and to an error in the choice of the center point. If the unknown is ground and polished on a spindle that is not concentric with it, the axis of rotation of the unknown surface will not be centered at the chosen origin of coordinates. However, the average value of Y_R and Y_{-R} from the two sets of data, primed and unprimed, respectively, will be almost free from the error of centering.

In order to obtain more precise values for A_R , the following procedure is followed: Reference marks with their associated R -values, or abscissas, are placed on the glass surface along the chosen reference line. These reference points are indicated by black dots centered in the small circles shown in figure 2. The standard is placed in the desired position and the air wedge adjusted so that a desirable number (5 to 10) of fringes cross the reference line. The present author prefers to have the fringes approximately perpendicular to the reference line. Considerable departure from this, however, is usually tolerated. The fringe pattern is photographed.³ This is repeated for all desired positions of the standard. A fine straight line, using a needle point and a straight edge, is drawn across the photograph through the nine reference points on it. The positions of the centers of the reference points are marked by pricking the photograph with a needle. This is to permit accurate readings on their positions. If no distortion is introduced in the photographic reproduction, the separation of all adjacent points will be equal. The intersections of all fringes, both dark (i. e., integral orders of interference) and light (integral plus half orders), with the reference line are marked also by pricking the photograph. The direction of increasing orders of interference is ascertained from tests and notations made when the fringes are photographed. The relative orders of interference, beginning with 1, are indicated by inserting numbers on the dark fringes. In figure 3 these numbers appear above each of the fringe patterns. The lesser visible numbers, adjacent to the reference points, represent the abscissas (or

R -values) of the several reference points that were marked on the unknown surface.

In general, the scale of the photographic reproduction will differ from that of the chosen abscissa scale. For interpolation purposes any convenient scale may be used to measure the relative positions of points on the photograph. A centimeter scale with millimeter marks was used for this compilation. We will define the scale used as the P -scale and the readings from it as P -values. The P -values corresponding to all reference points and fringes are read off the centimeter scale and inserted in column 1 of table 1, which represents a typical data sheet. The corresponding R -values (numbers associated with the fiduciary reference points and appearing in the photographs) are inserted in column 2; the A_R -values, corresponding to integral and half-integral orders of interference, are inserted in their respective places in column 3. If an observed P -value, corresponding to an R -value, falls precisely on a fringe (reference point centered on a fringe), two identical P -values are inserted as for $R = -2$ and $A_R = 5.5$ in the case given here.

The object of this listing and the following computations is to obtain more precise A_R -values (orders of interference) than can be obtained by direct interpolation or reading from the photographs.

A direct plot of the P -values versus A_R -values first entered in column 3, table 1 (the blank spaces to be filled later), shown as small black dots in figure 5, may be made. A smooth curve is then drawn through these points. The values for A_R in column 3, which correspond to R -values in column 2, are located on the curve. The corresponding ordinates represent the interpolated A_R -values desired. The values with asterisks, shown in table 1, are more precise values obtained by an interpolation process described in the following paragraphs.

The values for A_R , determined above from the direct plot, are not as precise as the directly observed values from which the plot was made. More precise

TABLE 1. Arrangement of data for computation

Values for A_R in column 3 that are followed by an asterisk are interpolated values from the curve of figure 5.

P	R	A_R	$0.7 P$	$(0.7 P + A_R)$
0.21	-4	8.23*	0.15	8.38*
.44	-----	8	.31	8.31
.95	-----	7.5	.65	8.15
1.39	-----	7	.97	7.97
1.54	-3	6.80*	1.08	7.88*
1.85	-----	6.5	1.29	7.79
2.40	-----	6	1.68	7.68
2.94	-2	5.50*	2.06	7.56*
2.94	-----	5.5	2.06	7.56
3.54	-----	5	2.48	7.48
4.20	-----	4.5	2.94	7.44
4.29	-1	4.42*	3.00	7.42*
4.80	-----	4	3.36	7.36
5.43	-----	3.5	3.80	7.30
5.61	0	3.33*	3.95	7.28*
6.05	-----	3	4.24	7.24
6.85	-----	2.5	4.80	7.30
7.00	1	2.41*	4.90	7.31*
7.70	-----	2	5.39	7.39
8.37	2	1.62*	5.86	7.48*
8.60	-----	1.5	6.02	7.52
9.67	-----	1	6.77	7.77
9.73	3	0.98*	6.81	7.79*
10.80	-----	.5	7.56	8.06
11.08	4	.39*	7.76	8.15*

³ A micrometer eyepiece may be used to read the data directly and thus dispense with photography.

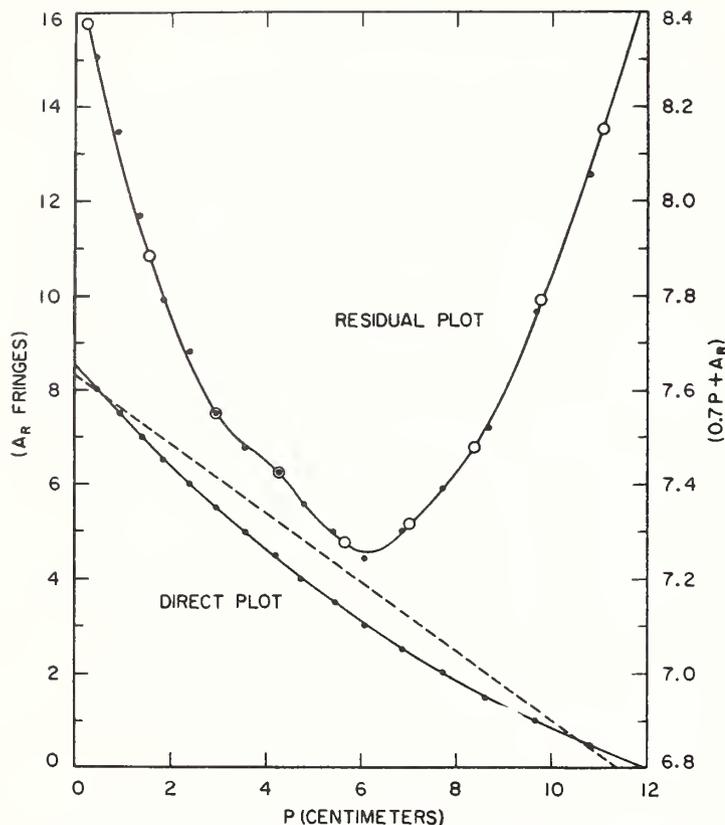


FIGURE 5. Direct and residual plot of data for nonlinear interpolation of fringe orders.

values may be obtained from a residual plot.⁴ To obtain data for the residual plot, a column of values (table 1, column 4), computed with a slide rule, is added to the table of observed quantities. This column is a product of corresponding P -values and an appropriately chosen constant. This constant represents the slope of a straight line that crudely approximates the plotted points. It is indicated in figure 5. The approximate slope of this line is 0.7. A relatively large error in the choice of the slope contributes little or no error in the final results. Values of $0.7P$ are accordingly computed to two decimal places. The sums of corresponding values from columns 3 and 4, except where vacancies exist in column 3, are entered in column 5. These values are plotted, on an appropriately chosen scale, against corresponding P -values, and a smooth curve that best fits the points is drawn in by inspection (residual plot, fig. 5). One may now locate the points, shown as circles, on this curve, whose abscissas are those observed for the chosen reference points. The corresponding ordinates are read off and inserted in the $(0.7P + A_R)$ column. These values are marked with an asterisk for distinction. The corresponding values of $0.7P$ are subtracted from $(A_R + 0.7P)$, and the resultant A_R values, shown also with asterisks, are inserted in the vacant places of the A_R column. These are the desired orders of interference at the several reference points. Values for X_R ($X = B, C, D, \dots$) are obtained from all photographs in this manner.

⁴ H. M. Goodwin, Elements of the precision of measurements and graphical methods, p. 60 (McGraw-Hill Book Co., New York, N. Y., 1920).

TABLE 2. Computation sheet for statistical evaluation of data
The average of corresponding values in columns 3 and 5 represents the most acceptable results

R	A_R	Y_R	B_R	Y_{-R}
-4	8.23	-----	-----	-----
-3	6.90	-----	-----	-----
-2	5.50	-----	-----	-----
-1	4.42	-----	-----	-----
0	3.33	0.00	8.71	0.00
+1	2.41	-.09	7.13	-.09
2	1.62	-.23	5.75	-.27
3	0.98	-.56	4.49	-.56
4	.39	-.98	3.32	-1.04
5	-----	-1.57	2.34	-1.58
6	-----	-2.30	1.49	-2.28
7	-----	-3.16	0.77	-3.13
8	-----	-4.26	.29	-4.20
9	-----	-5.67	-----	-5.39
10	-----	-7.05	-----	-6.74
11	-----	-8.64	-----	-8.28
12	-----	-10.30	-----	-9.96
13	-----	-12.37	-----	-11.86
14	-----	-14.51	-----	-13.93
15	-----	-16.86	-----	-16.21

In order to determine the shape of the surface from the fringe readings and their corresponding abscissas, or R -values, these quantities are now applied to the formulas of eq (3), (4), and (6). Table 2 is a typical computation sheet. From eq (6) one computes $Y_4 = 3.33 - \frac{1}{2}(8.23 + 0.39) = -0.98$, and likewise for all values of Y_R in the range covered by the standard in position A (column 3 of table 2). In order to proceed to the evaluation of Y_R for other values of R , eq (3) and (4) are used. Values for B_R , found in the same manner as described previously for A_R , are inserted in column 4, table 2. This permits a summation to be made of all terms shown in eq (3) and (4) and consequently an evaluation of M_B and B_0 . As the number of reference points covered by the standard in each of the two positions, A and B , is odd (i. e., from $R=0$ through $R=4$, or 5 points), they are each assumed to represent 2 observations, making the total number of observations 10, which may now be divided into 2 equal groups. With this in view, it will be seen that $\sum_{R_1}^{R_2} R = 2 \times 0 + 2 \times 1 + 1 \times 2 = 4$, and likewise for all summations shown in eq (3) and (4). The resultant values for M_B and B_0 are, respectively, -1.580 and $+8.67$. Slide-rule computation is adequate except for final precision. In general, the computed parameter B_0 will differ slightly from the observed value found in table 2. On substituting these values for M_X and X_0 in eq (1), and using values for R and B_R in columns 1 and 4, values for Y_R may be computed from $R=5$ through 8. These operations are now repeated, using consecutively the C and D data, to compute additional values for Y_R to the edge of the surface. These values are inserted in column 3, table 2. A similar treatment of the primed data yields an independent set of value for Y_{-R} shown in column 5, table 2, which should agree approximately with the above set. An average of Y_R and Y_{-R} values for each value of R is the accepted value. This averaging operation tends to decrease the effects of error in choice of the axis of rotation of the unknown surface as well as observational errors.

WASHINGTON, December 3, 1953.

UNITED STATES DEPARTMENT OF COMMERCE • Charles Sawyer, *Secretary*
NATIONAL BUREAU OF STANDARDS • E. U. Condon, *Director*

Thermal Expansion of Solids

by *Peter Hidnert and Wilmer Souder*



National Bureau of Standards Circular 486

Issued March 15, 1950

Preface

Thermal expansion is one of the fundamental properties of materials that is important in science and industry. The problems in which the thermal expansion of materials must be recognized are as varied as our industries.

This Circular is issued to supply a demand for information about various methods for determinations of thermal expansion of solids. Related topics such as relations between thermal expansion and chemical composition of materials are also discussed in the Circular.

Three types of thermostats (differential expansion, bimetallic flexure, and fluid expansion) used in various applications, are discussed.

At various times during the past four decades, this Bureau has published the results of investigations on the thermal expansion of various materials. A list of these publications will be sent free of charge to anyone interested, upon request.

E. U. CONDON, *Director.*

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Thermal Expansion of Solids

by Peter Hidnert and Wilmer Souder

This Circular describes 10 methods for determinations of thermal expansion of solids. The procedures used in determining expansion equations and coefficients of expansion of materials are given. Relations between thermal expansion, temperature, chemical composition, density, compressibility, specific heat, melting point, atomic weight, and other properties of materials are indicated. The problems in which the thermal expansion of materials must be recognized are as varied as our industries. One of the important applications of thermal expansion is in thermostats that are described in the publication.

I. Introduction

One of the frequently measured physical properties of materials is thermal expansion. Practically all materials undergo a change of dimensions when they are heated or cooled. For isotropic bodies, the expansion or contraction is the same in all directions, but in anisotropic bodies the expansion or contraction is dependent on direction.

Determinations of dimensional changes of materials, of coefficients of linear and cubical expansion, and of the temperatures and magnitudes of dimensional changes during transformations at constant temperature and with slow or relatively rapid heating and cooling rates (including quenching) are important in science and industry. Dilatometric methods have an advantage over the thermal method (temperature-time cooling curves) in investigating transformations, for the temperatures may be passed through slowly or quickly or the sample may be held at a given temperature for any length of time in order to attain equilibrium and the heating or cooling then resumed. Various types of thermal-expansion apparatus are used for these determinations.

The change in length that takes place when a solid body is heated depends upon the original length of the body and the temperature range over which it is heated. The observation of this change is meaningless unless it is related to the length of the body and the temperature range. The relation between these factors known as the coefficient of linear thermal expansion has been defined in a number of ways.

The average coefficient of linear expansion is defined as

$${}_1a_{t_2} = \frac{L_2 - L_1}{L_0(t_2 - t_1)} = \frac{\Delta L}{L_0 \cdot \Delta t}, \quad (1)$$

where ${}_1a_{t_2}$ is the average coefficient of expansion between temperatures t_1 and t_2 , L_1 and L_2 are the lengths at t_1 and t_2 , respectively, and L_0 is the length at a reference temperature. This reference temperature may be 0° C, room temperature, or any other convenient temperature. The authors prefer to use 0° C for the reference temperature. If it is not convenient to obtain the length at 0° C, the length at room temperature is used. The difference introduced in the latter case is negligible for the average coefficient of linear expansion.

The instantaneous coefficient of linear expansion or coefficient of expansion at any temperature, t , may be defined as

$$a_t = \lim_{t_1 \rightarrow t_2} \frac{L_2 - L_1}{L_0(t_2 - t_1)} = \frac{dL}{L_0(dt)}, \quad (2)$$

where a_t is the instantaneous coefficient of linear expansion at temperature t .

If L_0 is the length of a solid body at 0° C, then its length at any temperature t° C may be represented by the empirical equation

$$L_t = L_0(1 + at + bt^2 + \dots), \quad (3)$$

where a and b are constants, depending upon the material. In most cases, these constants a and b are positive, for bodies usually expand at a faster rate as the temperature increases. For a short range of temperature, the equation $L_t = L_0(1 + at)$ representing a straight line may be used instead of eq 3 representing a curve. When a molecular change (transformation) occurs on heating or cooling a body, its length may not be accurately represented by a first, second, or third degree

equation. In such cases, it may be desirable to obtain two equations, one for the range below the transformation temperature or point of inflection, and the other equation for the range above the transformation temperature.

The instantaneous coefficient of linear expansion may also be derived by differentiation of eq 3, and represented as follows:

$$\alpha_t = \frac{a + 2bt + \dots}{L_0}, \quad (4)$$

where a and b are the same constants used in eq 3. The instantaneous coefficient of expansion at 0° C is a/L_0 .

A change in volume that takes place when a solid body is heated may be treated in a similar manner. The following equations correspond to the previous equations relating to linear thermal expansion:

$${}_t\alpha_{t_2} = \frac{V_2 - V_1}{V_0(t_2 - t_1)} = \frac{\Delta V}{V_0(\Delta t)} \quad (5)$$

$$\alpha_t = \lim_{t_1 \rightarrow t_2} \frac{V_2 - V_1}{V_0(t_2 - t_1)} = \frac{dV}{V_0(dt)} \quad (6)$$

$$V_t = V_0(1 + \alpha t + \beta t^2 + \dots) \quad (7)$$

$$\alpha_t = \frac{\alpha + 2\beta t + \dots}{V_0} \quad (8)$$

In eq 5 to 8

${}_t\alpha_{t_2}$ = average coefficient of cubical thermal expansion between temperatures t_1 and t_2

V_1 = volume at t_1

V_2 = volume at t_2

V_0 = volume at a reference temperature (In eq 7, volume at 0° C)

α_t = instantaneous coefficient of cubical thermal expansion at any temperature t°

V_t = volume at any temperature t°

α and β = constants depending upon the material.

The average coefficient of cubical thermal expansion of an isotropic body between t_1 and t_2 , is represented by the equation

$${}_t\alpha_{t_2} = 3{}_t\alpha_{t_2} + 3{}_t\alpha_{t_2}^2(t_2 + t_1) + {}_t\alpha_{t_2}^3(t_2^2 + t_2t_1 + t_1^2) \quad (9)$$

When $t_1 = 0^\circ$, eq 9 may be expressed as

$${}_0\alpha_t = 3{}_0\alpha_t + 3{}_0\alpha_t^2 t + {}_0\alpha_t^3 t^2, \quad (10)$$

where ${}_0\alpha_t$ and ${}_0\alpha_t$ represent the average coefficient of cubical expansion and the average coefficient of linear expansion, respectively, between 0° and t° . As the second and the third terms of eq 9 and 10 are usually negligible, they reduce to

$${}_t\alpha_{t_2} = 3{}_t\alpha_{t_2} \quad (11)$$

$${}_0\alpha_t = 3{}_0\alpha_t \quad (12)$$

II. Types of Thermal-Expansion Apparatus

Ten methods for measurement of thermal expansion are described in this Circular.

1. Precision Micrometric Method

The National Bureau of Standards uses micrometric thermal-expansion apparatus for the most precise work on the linear thermal expansion of solids between -150° C and $+1,000^\circ$ C. This apparatus is of two types: (1) The air-type heating chamber, shown at the left in figure 1, in which the sample is surrounded by air or an inert gas, and (2) the stirred-liquid bath, shown at the right in figure 1, in which the sample is surrounded by a suitable liquid.

In both types of apparatus electric heating is used. Thermocouples and a potentiometer are used for measuring temperatures. For determinations at low temperatures, cooling is secured in the stirred-liquid bath by expanding compressed air from a liquid-air interchanger through coils immersed in the bath.

The positions of the ends of the sample in the air chamber are indicated by fine wires (0.001 or 0.002 in. in diam) weighted and hanging vertically from the ends of the sample through slots in the

bottom of the chamber. Two micrometer microscopes, mounted horizontally on a traveling comparator and separated by a lateral distance approximately equal to the length of the sample (10 to 50 cm), are used to measure the length changes, as indicated by the changes in separation of these wires. In the stirred liquid bath, tungsten wires (0.001 in. in diam) in contact with the ends of the sample, extend from a fixed horizontal bar above the sample to hinged fingers, inserted beneath the ends of the sample as indicated in figure 2. Here the length changes of the sample are proportional to the changes in the distances between the wires in the plane of the microscopes.

The sample is in the form of a straight rod or bar of uniform cross section. The usual length is 300 mm, and the diameter (or diagonal if the rod has a rectangular cross section) is 5 to 10 mm. The rod is prepared with cylindrical ends having radii of curvature equal to one-half the length of the rod, so that the measured length will not change for slight vertical rotations about its center. Provision has been made for handling samples of other lengths and diameters. The limits of length are the length of the furnace for long rods (50 cm) and the limit of contact approach of the two

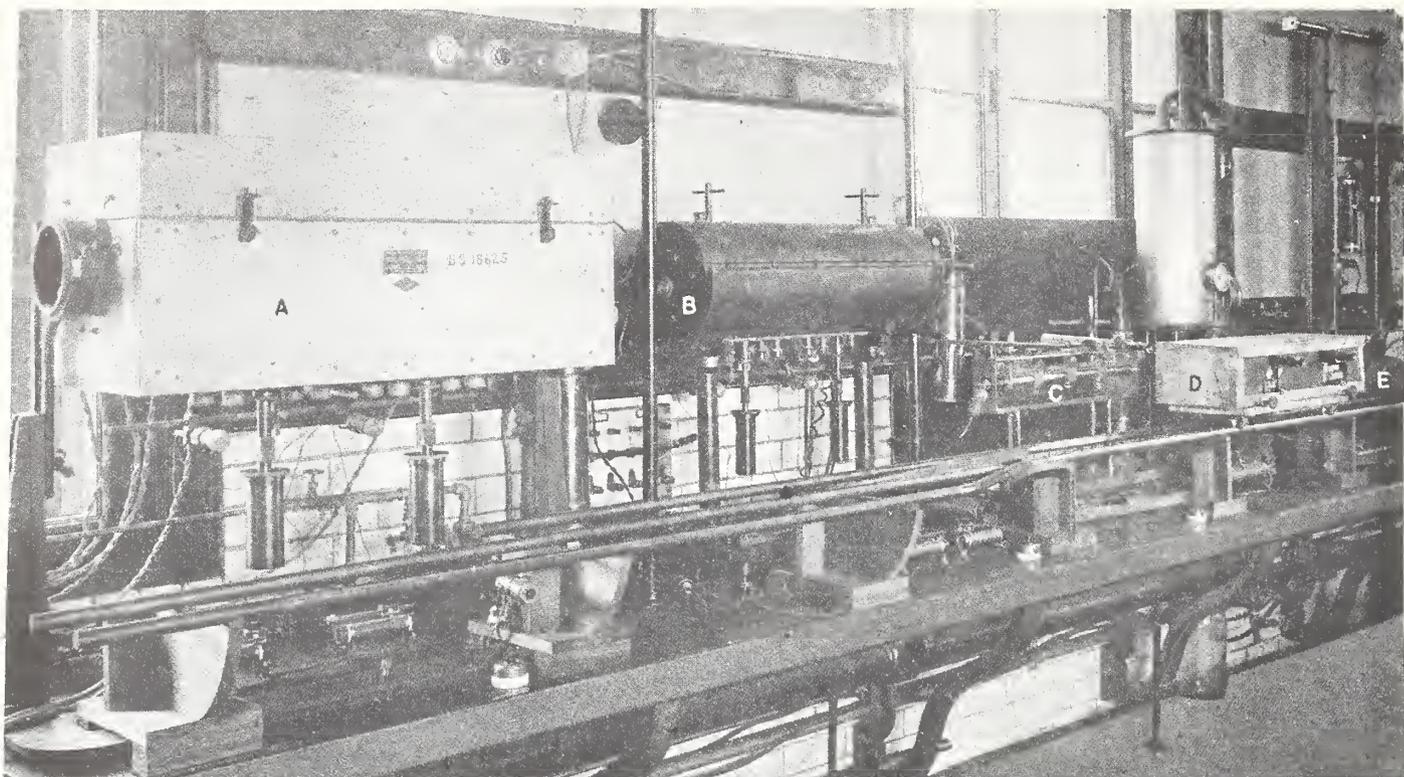


FIGURE 1. *Furnaces, standard bar and comparator of precision micrometric thermal-expansion apparatus.*
 A, Furnace (20° to $1,000^{\circ}$ C); B, furnace (20° to 600° C); C, standard bar; D, microscope comparator; E, bath (-150° to $+300^{\circ}$ C).

micrometer microscopes for short rods (10 cm).

When a sample must be heated in the air furnace to a temperature at which oxidation or sealing is expected, the furnace may be filled with an inert gas. When such gas is not available, it is possible to minimize the effects of oxidation or sealing by placing the observation wires in sharp V-grooves cut around the rod near each end.

A vane (5 to 25 g), attached to the bottom of each observation wire in the air-type heating chamber, is suspended in oil to damp out vibrations from the building and surroundings. The wires are annealed before observations are started by passing sufficient electric current to heat them to a visible glow. For determinations below 700° C, 0.001- or 0.002-in. diameter Chromel wires may be used. For determinations from 20° to $1,000^{\circ}$ C, platinum-osmium, platinum-rhodium or platinum-ruthenium wires 0.002 in. in diameter may be used.

With the air furnace it is possible to make measurements between 20° and $1,000^{\circ}$ C. The stirred liquid bath is used for temperatures between -150° and $+300^{\circ}$ C. The stirred liquid bath, by reason of the circulation and intimate

contact of the heated or cooled liquid, permits more rapid observations than the air furnace. Observations are recorded when the sample has reached the temperature of the furnace. This condition may be established by waiting until the observation wires show no movement in the microscopes. After readings are completed the heating current is adjusted for the next temperature. The immersion of samples in the liquid bath is limited to those samples not attacked by the liquids used. The usual practice is to use a light engine oil (Esstie 42) for the temperature range 20° to 300° C. For the range -65° to $+60^{\circ}$ C Dow Corning Fluid No. 500 (kinematic viscosity 2.0 centistokes at 25° C) is used, and 2-methylpentane is used at low temperatures to about -150° C. A water coil in the liquid bath is used to hasten cooling between 300° and 20° C.

The observation wires on which microscope settings are made are illuminated by 10-watt frosted spherical lamp bulbs placed behind them. Resistances in the lamp circuit are used to reduce the illumination to a satisfactory intensity. An excellent arrangement for high-precision measurements is secured by the parallel spider lines on the



FIGURE 2. *Sample, sample holder, and hinged fingers for wires in liquid bath.*

reticle frames moved by the microscope micrometers.

The air furnace has control rheostats for each side and end of the heating chamber. The external heat loss from these rheostats is reduced by using a transformer for the lower temperatures.

With these types of apparatus it is essential that the position of the samples be maintained horizontally during the determinations. A further requirement for the stirred liquid bath is that the readings be made at a known distance above each end of the sample, and that the ratio of distances (level of observation to upper supports of wires, and ends of sample to upper supports of wires) be known and maintained.

The arrangement of the furnaces, standard bar, and comparator is shown in figure 1. The air type heating chamber, or furnace, is shown at the extreme left of the figure. A smaller air furnace is shown at the right of the large furnace. The standard bar of fused quartz and the micrometer comparator appear in front of the window. The interchanger and stirred liquid bath are partially visible near the right margin of figure 1. Vacuum bottles filled with melting ice for maintaining the temperature of the cold junctions at 0° C appear at the right of the two furnaces in this figure. Connections to the potentiometer are made through lead-covered cables and a selector switch. Triple-junction thermocouples are placed in the air furnaces. A single thermocouple junction in the stirred liquid bath is sufficient. The forced circulation of the bath liquid by a motor and a propeller maintains uniformity of temperature. Platinum platinum-rhodium thermocouples are used for the 1,000° C air furnace. For the smaller air furnace (600° C), gold-palladium platinum-rhodium thermocouples are used. A copper-constantan thermocouple is employed in the stirred liquid bath. The interchanger is of the regular air-liquefying type. The cooled air, compressed to about 2,500 lb/in.², is expanded through coils in the stirred liquid bath, when temperatures between +20° and -150° C are required.

This method for determining linear thermal expansion is believed to be the most precise known. Under most favorable conditions, measurements of coefficients of expansion accurate to 0.1 percent are possible.

Additional information about this equipment is given in publications by Hidnert [1]¹ and Souder and Hidnert [2]. Blueprints giving details of construction are on file at the National Bureau of Standards and may be borrowed upon request.

2. Interference Method

When only small samples of material (about 10 mm or less in length) are available, the Fizeau-

¹ Figures in brackets indicate the literature references at the end of this Circular.

Pulfrich interference method is especially useful for determinations of linear thermal expansion. The method is also useful for determinations of the linear thermal expansion of crystals or other solids in different directions. The sample is placed vertically between two transparent fused-quartz plates, each about 4 mm thick and reasonably free from bubbles and other imperfections. The surfaces of each plate should be flat within one-fifth of a fringe, and should be inclined to each other at an angle of about 20' of arc. The sample with the two fused-quartz plates is set in an electric furnace or cooling chamber for heating or cooling. When the plates are illuminated normally with monochromatic light, a set of interference fringes is produced by the interference of the light reflected between the lower surface of the upper plate and the upper surface of the lower plate, when the angle between these surfaces is slight. The fringes are observed by means of a viewing device. Changing the temperature of the sample brings about a change in length, which causes the distance between the plates to change with a corresponding movement of the interference fringes past a reference mark on the lower surface of the upper plate. From the observed displacement of the fringes, the change in length, or linear thermal expansion, can be determined. The temperature of the sample is determined with a thermocouple, resistance thermometer, or interference thermometer [3]. This method may be used at low temperatures and at elevated temperatures to about 1,000° C.

If the sample is maintained in a vacuum during the determinations, the linear thermal expansion per unit length for a given temperature interval is computed from the equation

$$\frac{\Delta L}{L} = \frac{\lambda N}{2L}, \quad (13)$$

where L is the initial length of the sample, ΔL is the change in length or linear thermal expansion, λ is the wave length of monochromatic light, and N is the number of fringes that passed the reference mark. If the sample is heated or cooled in air, the following equation should be used:

$$\frac{\Delta L}{L} = \frac{\lambda N}{2L} + \frac{A}{L}, \quad (14)$$

where A is the "air correction". This correction varies with the pressure and the temperature of the air. Tables 2 and 3 of Merritt's publication [4] give values of A/L in microns per centimeter for 760 mm atmospheric pressure, and for various temperatures between -194° and +1,100° C. The average coefficient of expansion is computed by dividing $\Delta L/L$ by the change in temperature.

Austin [5] stated that the maximum errors of the interference method with his vacuum furnace

should not be greater than 1 percent at 300° C or 0.7 percent at 600° C, and the probable errors will be much less than these values. When the sample is heated in an air furnace, the errors may be greater as a result of the uncertainty caused by the presence of an air film between the sample and the interferometer plate and the oxidation or scaling of the sample.

Additional information about the interference method has been published by Peters and Cragoe [6], Merritt [4], and Saunders [7].

Trowbridge [8], Arnulf [9], Sinden [10], Nix and MacNair [11], and Saunders [12] described methods of photographing fringes.

In 1916 Meggers [13] indicated how the determination of the exact order of interference or the optical measurement of length can be made with the aid of certain wavelengths of neon radiation. In 1919 Peters [14] described the use of the interferometer in the measurement of small dilations by observing fractional orders due to several wavelengths by the method described by Meggers. Willey and Fink [15] have recently used this method in determinations of coefficients of expansion of aluminum alloys. The constant attention of the observer and the counting of the interference fringes that pass the reference mark during heating or cooling, or the photographing of fringes are not required. The method has been used for many years at the National Bureau of Standards for determinations of the lengths of gage blocks at room temperature.

3. Fused-Quartz Tube and Dial-Indicator Method

A fused-quartz-tube thermal-expansion apparatus may be used for determinations of linear thermal expansion for various temperature ranges between -190° and $+1,000^{\circ}$ C. This type of apparatus, as improved by Hidnert and Sweeney [16] over a similar type used abroad, is recommended for commercial laboratories where data of the highest precision are not required. With the apparatus it is possible to obtain an accuracy of 2 percent.

Figure 3 shows a fused-quartz tube closed at the bottom, with a sample (20 cm) in the tube ready for heating or cooling. A movable fused-quartz rod rests on top of the sample and extends above the open end of the tube. The bottoms of the tube and the movable rod are ground concave and the ends of the sample, convex, in order to secure satisfactory contacts. The top of the movable rod, on which a dial indicator rests, is flat. Heating is effected by placing the tube containing the sample in a water or oil bath, or in an electric furnace (fig. 4) extending well above the top of the sample. Low temperatures are secured by using a cooling cabinet or suitable cooling baths, such as liquid air, solid carbon

dioxide in a mixture of equal parts of carbon tetrachloride and chloroform, ice and sodium chloride, and melting ice. A thermocouple, placed inside the fused-quartz tube near the center

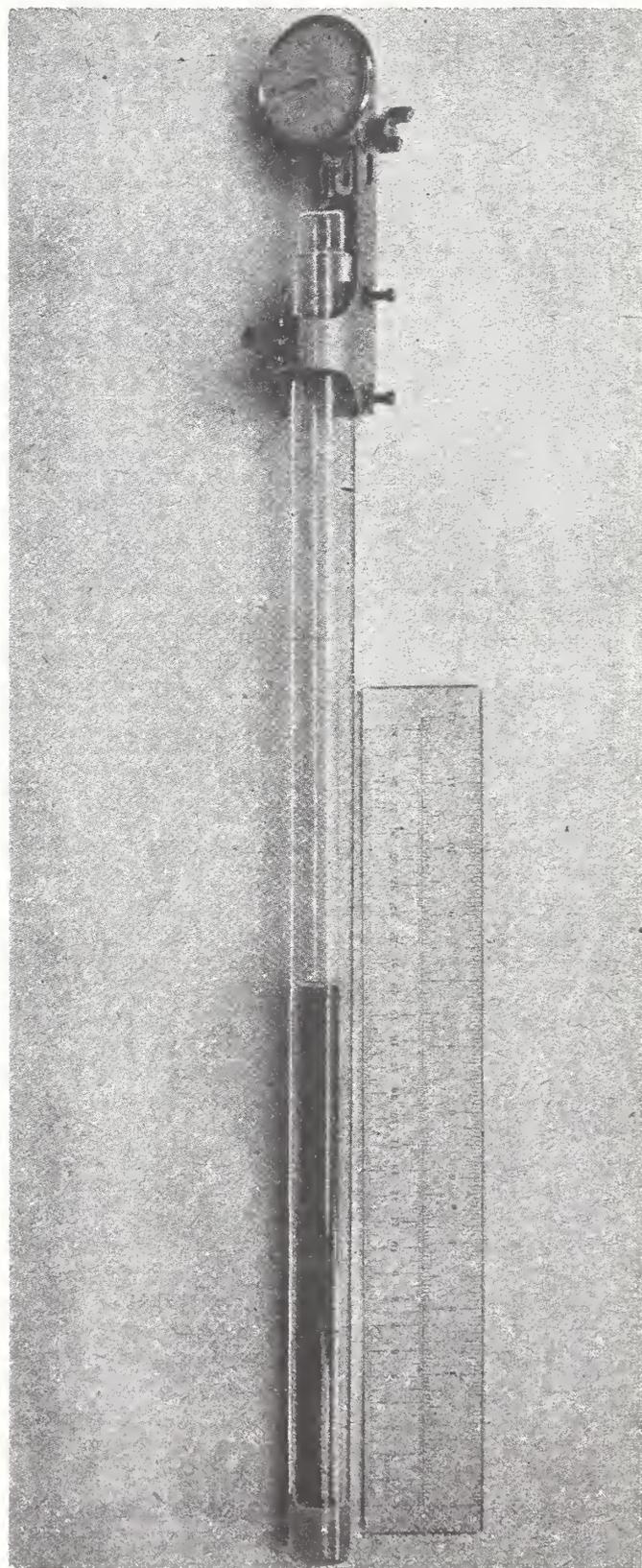


FIGURE 3. Fused-quartz tube, dial indicator, and sample.



FIGURE 4. Fused-quartz tube and dial indicator thermal-expansion apparatus.

of the sample, indicates the temperature. A dial indicator, fastened near the top of the tube, registers the differential expansion between the sample and an equivalent length (20 cm) of fused quartz. A small correction for the linear expansion of fused quartz is made. Prints showing details of this apparatus may be secured from the National Bureau of Standards.

Modifications of the fused-quartz-tube thermal-expansion apparatus for use with shorter samples or with a load on the samples, have been described by Hidnert [17] and by Hidnert and Dickson [18].

Walters and Gensamer [19] modified this type of apparatus so that the expansion of a sample can be determined in a vacuum or an inert gas between -200° and $+1,000^{\circ}$ C. Kingston [20] modified the apparatus to a recording dilatometer by means of a contact mechanism, transmission shaft, recorder and electronic relay. He also made provision for the use of various atmospheres.

4. Autographic Optical-Lever Method

Souder, Hidnert, and Fox [21] designed and constructed an autographic optical-lever thermal-expansion apparatus for use in commercial laboratories. It was found to give more accurate results than previous apparatus of similar type. With this apparatus it is possible to obtain expansion curves photographically or to observe points on the expansion curves of materials during the progress of the test.

A general view of the autographic expansion apparatus is shown in figure 5. The apparatus consists essentially of a furnace, *F*, mounted on a common base with a camera box, *C*, and a source of illumination, *I*. The back of the camera is provided with a hinged bookform plate holder for 8- by 10-in. photographic plates, films, sensitized paper, or a ground-glass screen for photographic or visual observations. The over-all length of the apparatus is approximately 64 in., height 19 in., and width 14 in.

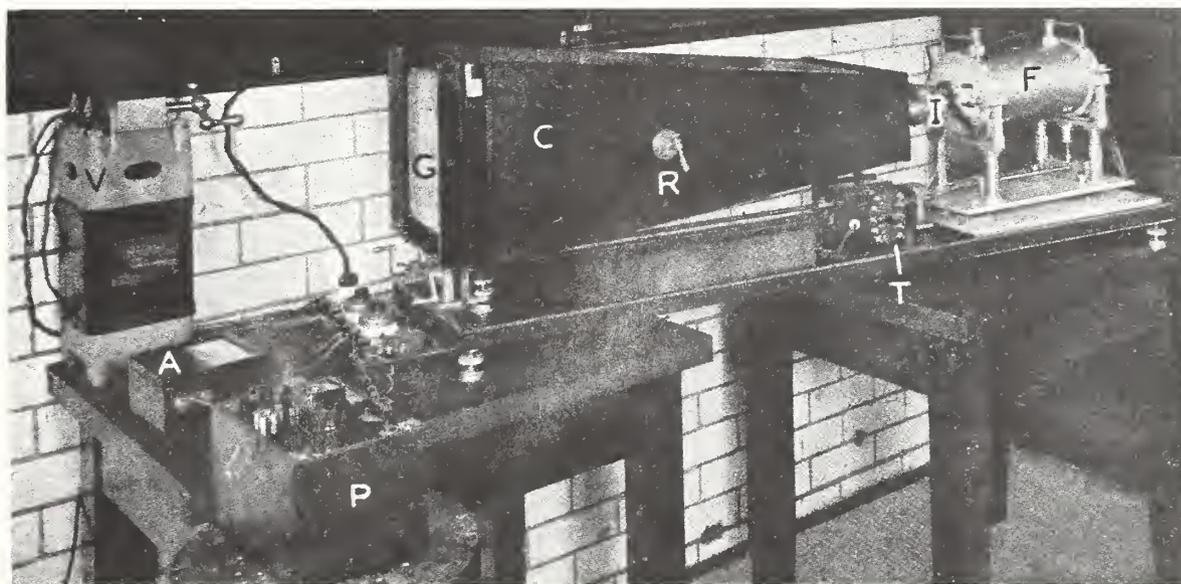


FIGURE 5. Autographic expansion apparatus.

A, Ammeter; C, camera box; F, furnace; G, glass screen; I, illumination; P, potentiometer indicator; R, internal reflecting mirror; T, transformer; V, induction voltage regulator.

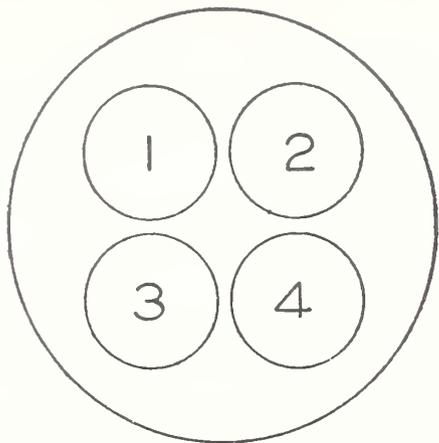


FIGURE 6. Cross section of alumina cylinder in furnace indicating the holes and the positions of "stationary" axis, temperature axis, sample, and thermocouple.

1, "Stationary" axis; 2, temperature axis; 3, sample (expansion axis); 4, thermocouple.

The observer is located at the glass screen, *G*, of the camera box (fig. 5) and is looking toward the furnace.

The following materials are inserted in the four horizontal holes of the furnace (fig. 6):

Hole 1—A long, fused-quartz rod, which represents a "stationary" axis.

Hole 2—A rod of Chromel *A* having uniform rate of expansion, 150 mm in length, which represents a temperature axis.

Hole 3—A sample 150 mm in length.

Hole 4—A Chromel-Alumel thermocouple with 3 junctions, 1 near the center of the sample in hole 3, and the other junctions near the ends of the sample.

The ends of the rod of Chromel and the sample in holes 2 and 3, respectively, are pointed. The angle at each pointed end is about 80°.

A fused-quartz plug is in contact with each end of the Chromel rod (temperature axis) and of the sample. The pointed ends of these 4 plugs and of the long, fused-quartz rod ("stationary" axis) extend beyond the ends of the furnace. The rear of the mirror *M* (fig. 7), held in position by means of a spring, is in contact with the fused-quartz plugs and the "stationary" axis (fused-quartz rod) extending from the left face of the furnace.

Fused-quartz plugs are in contact with the right ends of the Chromel rod and the sample. At each end of these plugs there is a 90° pivot bearing. One end of each of these plugs and one end (90° pivot bearing) of the "stationary" axis extend beyond the right face of the furnace. Each of these ends is held against an adjustable screw.

When the furnace is heated, the sample and the Chromel rod (temperature axis) expand. The mirror which is in contact with the plugs extending from the sample, Chromel rod (temperature axis) and "stationary" axis therefore moves. The expansion of the sample causes the bottom of the mirror to move and deflect the spot of light upward. The expansion of the Chromel rod (temperature

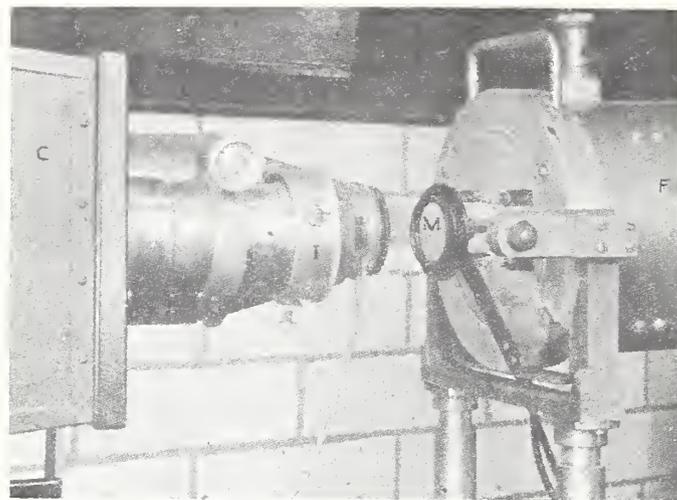


FIGURE 7. Stellite mirror in contact with the fused quartz rods and the "stationary" axis extending from the left face of the furnace.

C, Camera box; *F*, furnace; *I*, illuminator; *M*, optical lever mirror.

axis) causes one side of the mirror to move and deflect the spot horizontally. The expansion of the fused-quartz plugs, extending on each side of the sample, should balance the expansion of the fused-quartz plugs extending on each side of the Chromel rod (temperature axis), and also the expansion of an equivalent length of the "stationary" axis. A spot of light focused on the mirror and reflected on a glass screen, *G*, or photographic plate, indicates the expansion both of the sample and of the Chromel rod and describes a curve, the resultant of these two expansions vertically and horizontally. The abscissas indicate the expansion of the Chromel rod and may be evaluated to represent temperature. The ordinates represent the expansion of the sample. The curve therefore represents the expansion of the sample versus temperature.

For materials having coefficients of expansion approximately equal to those for ordinary steel, the error of the autographic expansion apparatus is about 6 percent for the range from 20° to 100° C, and about 3 percent for the range from 20° to 500° C. The apparatus is not sufficiently sensitive for tests on low-expanding materials. It is not satisfactory at temperatures where softening occurs in the sample or for materials that bend at elevated temperatures.

Chevenard [22] and other investigators used other forms of the optical lever method for determinations of the linear thermal expansion of materials.

5. Liquid-Micrometer Method

A liquid-micrometer method for determining linear thermal expansion of solids has been described by Andrew, Rippon, Miller, and Wragg

[23]. The sample (in the form of a hollow cylinder 2 in. long, $\frac{3}{4}$ in. in diameter with a $\frac{3}{8}$ -in. hole through the center) is placed horizontally in a silica tube of 1-in. internal diameter. The heating coil consists of a platinum wire wound around the silica tube over a length of 9 in. On the ends of the sample, which have been ground parallel and polished, two silica disks (also accurately ground) are placed. These disks are pressed against the ends of the sample by means of two silica tubes, which transmit changes in the length of the sample. One of the latter silica tubes is butted up against a heavy cast-iron block, and the other silica tube is pressed against a measuring device butted to a large lead block.

The relative movement of the silica tube is measured by a hydraulic device. This consists of a thick steel disk 8 in. in diameter, having a saucer-like depression on its face over which is clamped a thin disk of saw steel. The space so enclosed is filled with colored water. A pipe runs through the back of the steel disk, connecting this liquid with a horizontal glass tube to which a scale is attached. A small reservoir with a stopcock is also fitted for adjusting the amount of liquid in the system. This liquid micrometer is bolted to the lead block, so that the thin steel disk is vertical. A steel disk with a flat surface 1 in. in diameter transmits the movement of the silica tube to the thin steel diaphragm. With a glass tube of about 2-mm bore, the movement of the meniscus magnifies the displacement of the silica tube two thousand times. The temperature of the sample is measured by means of a thermocouple placed at the center of the sample. Figure 8 shows a diagrammatic sketch of the apparatus.

A flexible gas-tight joint is made by means of tubes of thin rubber fixed on the ends of the furnace tubes and then wired to the moving silica tubes. Through holes drilled in these two silica

tubes, gas may be circulated around the sample to prevent oxidation.

It is necessary to calibrate this apparatus with samples of known expansion.

The apparatus was modified by Haughton and Griffiths [24], the changes being the method of rendering the apparatus autographic, the method of mounting so as to enable adjustment of zero and resetting to be easily performed, and various minor modifications which simplify its arrangement and use. With this modified dilatometer, using a 2-in. sample, it is possible to record changes of the order of 10^{-6} in., although neither the sensitivity nor the accuracy is normally as high as this.

Because it is under pressure both in the original apparatus and in that modified by Haughton and Griffiths, the sample is likely to become deformed at high temperatures.

6. Induction-Furnace and Dial-Indicator Method

A high-frequency induction furnace was used by Heindl [25] for determinations of the linear thermal expansion of refractories between 20° and $1,800^{\circ}$ C. The heat was generated through the medium of a cylindrical graphite muffle, 12 in. long, 6 in. in diameter, and having a wall $\frac{1}{2}$ in. thick. A dial indicator, independent of the furnace, was supported by a framework with fused-quartz tubes for legs.

The arrangement of the sample (about 6 in. in length) is indicated in figure 9. This set-up formed a continuous system which expanded and contracted, independently of the furnace. Tungsten plates, $\frac{7}{8}$ in. square and 0.015 in. thick were placed between the sample and the graphite parts in a reducing atmosphere. The sample was placed within a muffle prepared from zirconium silicate.

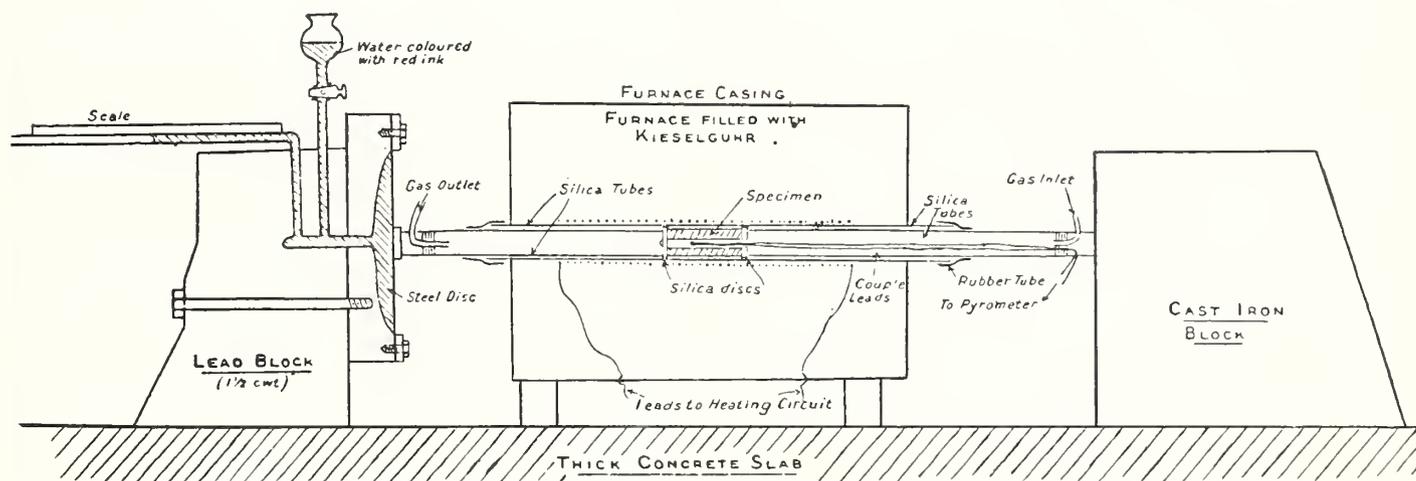


FIGURE 8. Diagrammatic sketch of liquid-micrometer dilatometer (Andrew, Rippon, Miller, and Wragg).

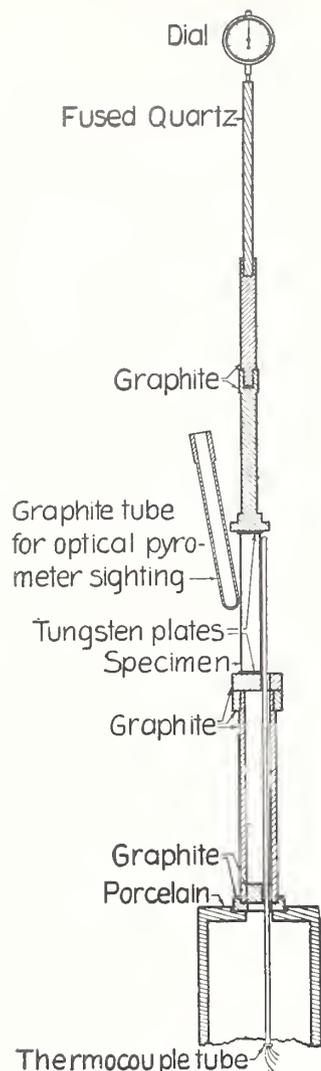


FIGURE 9. Specimen, specimen support, and the system transmitting the expansion in the induction furnace (Heindl).

The fused-quartz rod does not extend into the furnace.

Temperatures were measured with two platinum platinum-rhodium thermocouples up to about $1,500^{\circ}\text{C}$. Then the couples were removed from the apparatus and higher temperatures observed with an optical pyrometer.

From a comparison of dial readings, with this apparatus, on a bar of artificial graphite between 20° and $1,800^{\circ}\text{C}$ and on a bar of fused quartz of known expansion between 20° and $1,000^{\circ}\text{C}$, a curve was derived which gives corrections to be applied to the apparent expansion of a sample at various temperatures between 20° and $1,800^{\circ}\text{C}$. The portion of the correction curve from $1,000^{\circ}$ to $1,800^{\circ}\text{C}$ was extrapolated, since the apparent expansion of artificial graphite showed no irregularities between $1,000^{\circ}$ and $1,800^{\circ}\text{C}$.

7. Capacitance Method

A capacitance dilatometer was developed by Prytherch [26] for use in the Metallurgy Department of the National Physical Laboratory. An

oscillatory circuit is used and the sample (about 2 cm in length) is made to actuate the moving plate of a small capacitor, which constitutes part of the series grid capacitance of the circuit. Small changes in this capacitance bring about relatively large changes in the mean steady plate current of the tube. A continuous record of the plate current of the tube is made on a recorder, which also simultaneously records the temperature of the sample. The length changes of the sample are correlated with temperature. Haughton and Adcock [27] altered this apparatus somewhat to examine samples at temperatures up to $1,300^{\circ}\text{C}$ in an inert atmosphere. The circuit arrangements of the oscillator are those described by Prytherch, but Haughton and Adcock found it advantageous to screen the oscillator completely by means of a copper-lined box.

8. X-Ray Method

With the X-ray method it is possible to determine the coefficients of expansion of crystals in different directions over large temperature ranges. A very small amount of material is sufficient for a sample.

When a crystal is heated, the perpendicular distance between its atomic planes parallel to its crystal face generally increases. This distance d at any temperature may be determined by an X-ray method from Bragg's law [28]

$$n\lambda = 2d \sin\theta, \quad (15)$$

where λ is the wavelength of a monochromatic beam of X-rays incident upon the cleavage face of the crystal, θ is the angle between the incident X-ray beam and the atomic reflecting plane or the angle between the reflected beam and the same plane, and n is a small integral number that represents the order of reflection. From eq 15, the following equation may be obtained for the average coefficient of linear thermal expansion for a temperature range from 0° to t° :

$${}_0a_t = \frac{\sin\theta_0 - \sin\theta_t}{t \sin\theta_t}, \quad (16)$$

where ${}_0a_t$ is the average coefficient of linear expansion between 0° and t° , and θ_0 and θ_t are the angles indicated previously, at temperatures 0° and t° , respectively. From eq 16 it will be noted that it is not necessary to know the value of λ .

Hull [29] and Debye and Scherrer [30] modified Bragg's method so that a polycrystalline material (thin sheet or powder) can be used. Some of the crystals in this sample will be oriented so that when monochromatic X-rays are normally incident, they will be reflected from every plane

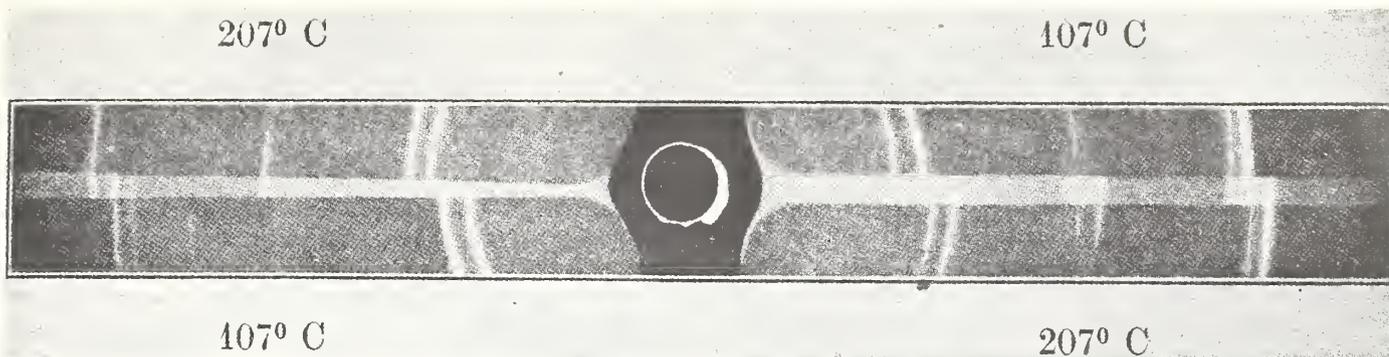


FIGURE 10. Shifts of the Hull-Debye-Scherrer rings of the reflections (114) and (103) of magnesium, FeK radiation (Hanawalt and Frevel).

set that is characteristic of the crystal. A diffraction pattern consisting of concentric circles may be obtained by placing a photographic plate in back of the sample. The innermost circle is caused by those crystals presenting the plane set having the largest lattice constant at the proper angle for reflection. When the sample is heated, the change in d will cause a corresponding reciprocal change in $\sin\theta$ (see eq 15), and hence in the diameter of the circle.

If the sample consists of a speck of powder, it may be supported by a fine wire and rotated so that complete lines are obtained on a cylindrical photographic film when the X-rays passing through a slit system are diffracted from the sample. The shift in the position of the lines at different temperatures indicates the change in the distance d , from which the linear thermal expansion may be computed.

Figure 10 shows the shifts of the Hull-Debye-Scherrer rings of the reflection (114) and (105) of magnesium (FeK radiation), obtained by Hanawalt and Frevel [31] at 107° and 207° C. The sample consisted of a thin disk of magnesium which was mounted in an electric furnace heated by storage batteries. The X-ray beam penetrated an aluminum foil covering that served as a window for the heating chamber. An iron-constantan thermocouple was attached to the surface of the sample. Two strips of lead covered the quadrants 1 and 3 of the film during the low temperature exposure, and quadrants 2 and 4 during the high temperature exposure. The films were measured on a comparator and the data used to evaluate the coefficients of expansion of magnesium, parallel to and perpendicular to the c -axis.

The X-ray tube used by Shinoda [32] and shown in figure 11, was designed to take Hull-Debye-Scherrer photographs with a large angle of reflection. The body of the X-ray tube made from a brass block 5.5 by 5.5 by 8 cm is shown in figure 12. A hole, F , 3.4 cm in diameter, was bored from one end of the block to the central part of the flat surface at the other end. The anticathode shown by b , and A , in figure 12, was

made from a flat copper or iron block 4.5 by 5.5 by 0.7 cm, having a slightly raised part on one side. This anticathode fits on the flat end of the body of the X-ray tube and the raised part of the anticathode enters hole F . As the electrons emitted from the hot cathode are focused on the center of hole F , the raised part of the anticathode becomes the source of X-rays. A narrow groove extends from hole F and forms a slit for the X-ray beam. A cellophane window covers the slit. The sample, S , is placed very close to this window, as shown in figure 12. The distance between the sample and the source of the X-ray beam is about 2.5 cm. The specimen holder is made from a copper block 1 by 1 by 0.5 cm, and the sample, usually a foil, is attached on one surface and covered with layers of cellophane and paper. The other surfaces of the specimen holder are covered with asbestos. For determinations at 100° C, steam is introduced through an opening in the specimen holder.

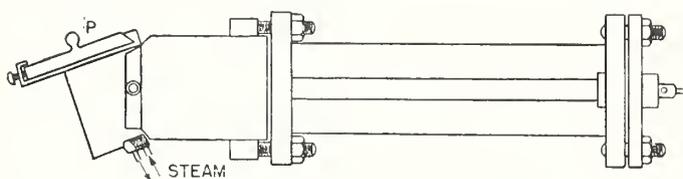


FIGURE 11. X-ray tube (Shinoda).

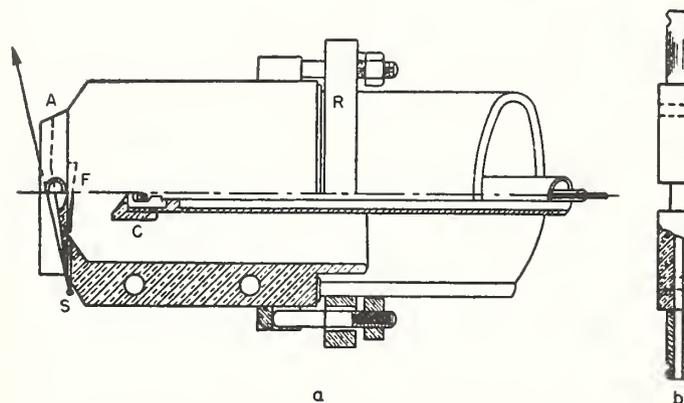


FIGURE 12. Body of X-ray tube a , and anticathode b (Shinoda).

The cathode is an ordinary hot cathode with a tungsten filament covered by a cap, *C*. This cap serves as a focusing device. Its anticathode side is V-shaped to secure a sharp line-focus. Insulation between the cathode and the body of the X-ray tube is attained by a glass tube 17.5 cm in length and 5 cm in diameter. This glass tube is connected to the cathode and the body by bolts and nuts through the brass ring *R*, with rubber rings used as packing.

The plate holder *P* in figure 11 can move up and down so that four photographs can be taken in succession. With this apparatus, an exposure of 3 to 5 min is sufficient for each photograph. The X-rays are excited by 5 to 7 ma and 40 kv.

Becker [33], Jay [34], and Wilson [35] described X-ray cameras used in determinations of the coefficients of linear thermal expansion of materials. The X-ray camera and auxiliary equipment used by Wilson are shown in figure 13. This camera consists of two circular brass plates, the upper (*A* in fig. 13) about 19 cm in diameter, the lower (*B*) somewhat larger containing a groove to receive the cover *C*. The plates are held about 1.1 cm apart by two brass V-blocks *D* and *E*. Each V-block carries a pair of stainless steel knife-edges that mark out definitely the length of film exposed to the X-rays. The film, backed by a sheet of black paper, is held firmly against the upper plate and the boss of the lower plate by two strips of steel ribbon on each side. The strips are attached to the back V-block, *D*, and are clipped firmly to the front V-block, *E*, by phosphor-bronze springs. The lower diagram of figure 13 shows, on the left side of the camera, the film, paper and steel strips lying loosely; on the right side it shows them clipped to the front V-block.

Each plate contains two water-cooling channels, *F*, and *G*, about 1-cm² section. The water enters through one of four tubes, *H*, at the back of the camera, passes through V-block *D*, to the upper plate, where it flows half-way round the outer channel until it meets a baffle, through U-tube *J* to the inner channel, where it flows completely round and through another U-tube to the other half of the outer channel and finally emerges through another of the tubes, *H*. The circulation in the lower plate is similar.

The furnace consists of two bobbins, *K* and *L* (fig. 13), made of oxidation-resistant steel. The windings are of platinum insulated from the bobbins by mica. The bobbins are fastened by screws in two soapstone members held to the plates of the camera by circular brass clamps *M* and *N*. The leads from the furnace are taken to small Bakelite terminal boards on the clamps. The lower clamp, *N*, supports a platinum-platinum-rhodium thermocouple, *P*. The upper clamp, *M*, contains a ball-race carrying a device, *Q*, for centering the sample. The sample is contained in

a thin-walled silica tube that is mounted in a brass rod screwed into a small brass block. This contains two mutually perpendicular grooves into which fit small disks on the ends of two screws. The sample is easily brought into the axis of rotation of the camera by adjusting these screws. A third screw clamps the block in position after centering. The sample is turned four times a minute by a small synchronous motor, *R*, that can be swung back out of the way, when it is necessary to remove the specimen holder. The leads from the motor and the upper half of the furnace pass out of the camera through four holes in the front V-block, *E*.

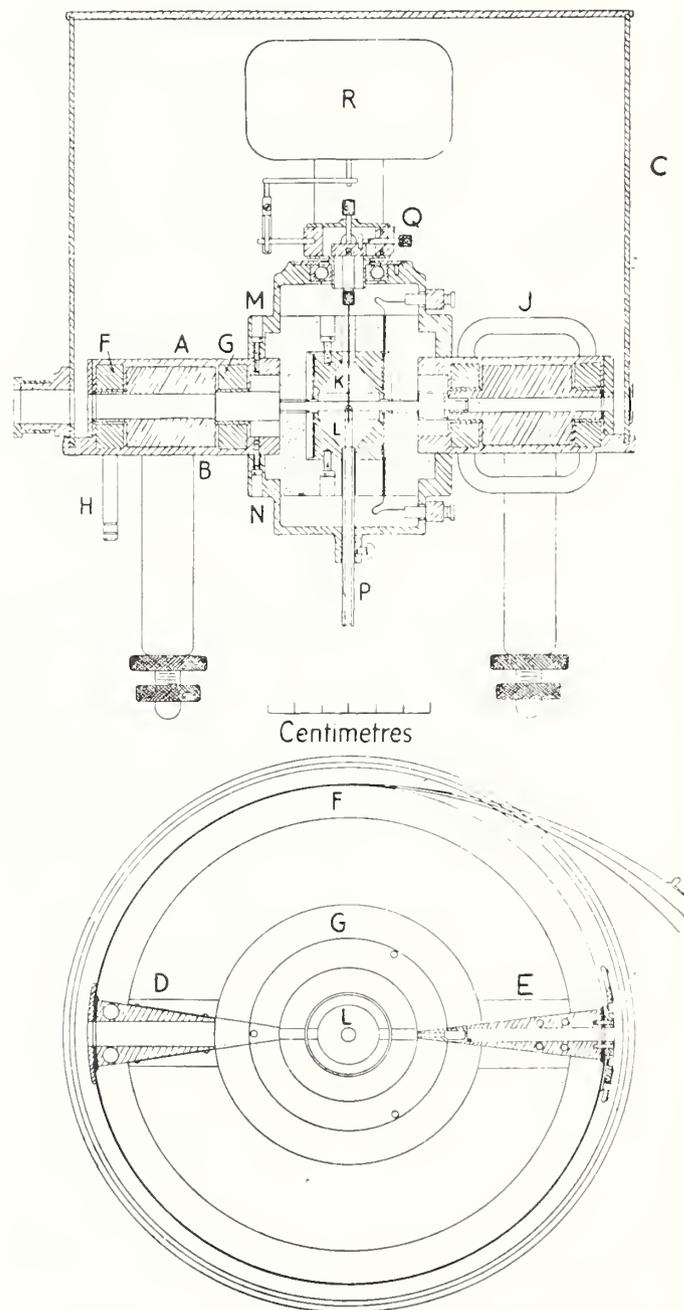


FIGURE 13. X-ray camera and auxiliary equipment (Wilson).

The X-rays enter the camera (fig. 13) through a small hole in the front of the cover. Filters are placed in a small pocket on the outside of the cover when necessary. The X-rays pass through a hole in the block, *E*, and strike the slit system, a small removable brass block fitting the inner end of the hole. The slit itself, about 0.8 by 3 mm, is at the outer end of this block; the wider part toward the center of the camera prevents rays scattered by the edges of the slit from striking the film. The rays then pass out of the camera through a hole in block *D* larger than that in block *E*. Two strips fastened to the outside of *D* prevent any rays scattered by it from striking the film.

The heating currents in the two furnace bobbins, *K* and *L* (fig. 13), can be varied independently. At high temperatures it is necessary to place an opaque screen between the furnace and the film to prevent fogging by the light emitted.

From Bragg's law, Shinoda [36] derived the following equation for a sample placed at the center of a circular film:

$$\frac{\Delta d}{d} = \frac{1}{2r} (\cot \theta) \Delta l \quad (17)$$

where Δd is a small variation of d , r is the radius of the film, and Δl is the displacement of the line (l is the distance between the line and the zero position). If a plate perpendicular to the X-ray beam is substituted for a circular film, eq 17 becomes

$$\frac{\Delta d}{d} = \frac{1}{r} \cot \theta \frac{1}{1 + \left(\frac{l}{r}\right)^2} \Delta l \quad (18)$$

The coefficient of linear expansion of a cubic crystal may be obtained by dividing $\Delta d/d$ by the change in temperature. Shinoda gives the following relations for other crystals:

For tetragonal

$$\frac{\Delta d}{d} = \frac{1}{1 + (h_1 c_1)^2} \left[\frac{\Delta a}{a} (h_1 c_1)^2 + \frac{\Delta c}{c} \right], \quad (19)$$

where $c_1 = \frac{c}{a}$, $h_1^2 = \frac{h^2 + k^2}{l^2}$ and h, k, l are indices of planes.

For rhombic

$$\frac{\Delta d}{d} = \frac{1}{h^2 + \frac{k^2}{b^2} + \frac{l^2}{c^2}} \left[\frac{h^2 \Delta a}{a^2} + \frac{k^2 \Delta b}{b^2} + \frac{l^2 \Delta c}{c^2} \right]. \quad (20)$$

For hexagonal

$$\frac{\Delta d}{d} = \frac{1}{1 + (h_1 c_1)^2} \left[\frac{\Delta a}{a} (h_1 c_1)^2 + \frac{\Delta c}{c} \right], \quad (21)$$

where $c_1 = \frac{c}{a}$ and $h_1^2 = \frac{4}{3} \left(\frac{h^2 + hk + k^2}{l^2} \right)$

From the values of $\Delta d/d$ in the preceding equations, it is possible to calculate $\Delta a/a$, $\Delta b/b$ and $\Delta c/c$ that correspond to the linear expansion of the a -, b -, and c -axis, respectively. The coefficients of expansion for these axes may be obtained by dividing $\Delta a/a$, $\Delta b/b$, and $\Delta c/c$ by the temperature change.

Becker [33] determined the coefficients of linear thermal expansion of materials from measurements of the angle θ at room temperature, and at elevated temperatures up to 2,200° C. Table 1 gives an example of his data and derived values for silicon. The following equation was used in computing the average coefficient of expansion:

$$1 + a (t_2 - t_1) = \frac{\sin \frac{\theta_1}{2}}{\sin \frac{\theta_2}{2}}, \quad (22)$$

where a is the average coefficient of linear thermal expansion between temperatures t_1 and t_2 , θ_1 is the angle at t_1 , and θ_2 is the angle at t_2 .

TABLE 1. Data and derived values for silicon

(hkl)	18° C		950° C			1+a(t ₂ -t ₁)
	$\frac{\theta_1}{2}$	$\sin \frac{\theta_1}{2}$	$\frac{\Delta \theta}{2}$	$\frac{\theta_2}{2}$	$\sin \frac{\theta_2}{2}$	
111.....	21° 30'	0.3665	0° 5'	21° 25'	0.3651	1.0038
220.....	36 40'	.5972	9	36 31	.5951	1.0035
311.....	44 25	.6999	10	44 15	.6978	1.0030
400.....	57 40	.8450	17	57 23	.8423	1.0032
420.....	71 0	.9455	31	70 29	.9425	1.0032
				Avg.....		1.0033

The average coefficient of linear expansion between 18° and 950° C is

$$a = \frac{1.0033 - 1}{950 - 18} = 3.5 \times 10^{-6} \text{ per deg C.}$$

Becker stated that the accuracy of his determinations is 8 percent for electrically conducting materials and 15 percent for nonconducting materials. Wilson [35] found that the systematic and random errors of his determinations of d are each about 0.0001 Å, and that the errors of the coefficients of expansion are from 1 to 2 percent.

9. Density Method

The density method has been used for determinations of the coefficients of cubical expansion. If the densities of a sample are determined by weighing it in a liquid of known density at two temperatures, 0° and t °, the average coefficient of cubical expansion may be computed from the equation

$${}_0\alpha_t = \frac{D_0 - D_t}{D_t t}, \quad (23)$$

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where ${}_0\alpha_t$ is the average coefficient of cubical expansion between 0° and t° , and D_0 and D_t are the densities of the sample at 0° and t° , respectively. If the sample is isotropic, the coefficient of linear expansion may be taken equal to one-third the coefficient of cubical expansion.

If the volumes of the sample at 0° and t° are obtained during the density determinations or by another method, the average coefficient of cubical expansion may be calculated from the equation

$${}_0\alpha_t = \frac{V_t - V_0}{V_0 t}, \quad (24)$$

where V_0 and V_t are the volumes of the sample at 0° and t° , respectively.

The density method has been used previously by Matthiessen [37, 38] and Dewar [39], for determinations of the coefficients of cubical expansion of various materials. By this method it is possible to make determinations with small irregular pieces of materials.

The methods used in determinations of the densities of materials are given in Bureau Circular 487.

10. Methods for Determinations of Volume Changes in Metals and Alloys During Casting

The foundryman, in producing a satisfactory casting, has a threefold interest in the shrinkage problem—first, he must make the dimensions of his pattern greater than the dimensions of the desired casting; second, he must employ all possible means to attain the maximum density in the casting and to produce a metal free from porosity, piping, and gas holes; and third, he must consider the design of his casting in its relation to the tendency of the casting to crack in the mold, as a result of stresses set up by differential shrinkage, particularly at sharp angles or abrupt changes in cross section. Determinations of volume changes of metals and alloys during casting are therefore important in design, pattern making, and foundry practice.

When a molten metal or alloy is cooled to room temperature, a change of volume takes place. This change is the algebraic sum of three consecutive volume changes that may be defined as follows:

1. Liquid shrinkage is the volume contraction of a metal or alloy on cooling from any temperature in the liquid state to the freezing point of a metal, or to the beginning of the freezing range of an alloy.

2. Solidification shrinkage (or expansion) is the volume contraction (or expansion) of a metal or alloy on passing from the liquid state at the freezing point to the solid state at the melting point. In the case of a pure metal, the freezing point and the melting point occur at the same temperature. When an alloy solidifies over a

freezing range, the solidification shrinkage (or expansion) is the volume contraction (or expansion) that occurs from the beginning (or end) of freezing. The solidification shrinkage of a eutectic or an intermetallic compound, which freezes at a constant temperature, is similar to that of a pure metal.

3. Solid shrinkage is the volume contraction of a metal or alloy on cooling from the melting point in the solid state to any lower temperature, usually room temperature.

Liquid shrinkage is a definite physical property of a metal or alloy. Solidification shrinkage is essentially a constant for a pure metal, eutectic, or intermetallic compound. However, in alloys of the solid-solution type, the solidification shrinkage is complicated due to the fact that the solid that separates from the melt during freezing continually changes in composition. During this selective crystallization stresses may be set up on account of different coefficients of expansion. Solid shrinkage of a metal or alloy may also be variable. Stresses may exist in a cast alloy of the solid-solution type as a result of progressive solidification and structural transformations after solidification. Such stresses may also be due partly to temperature gradients set up during cooling.

As a result of these stresses in a cast alloy, the observed linear contraction of a bar, designated as patternmaker's shrinkage, may differ from that calculated from available data on linear thermal expansion of the alloy. Another factor which influences the difference between the linear thermal expansion of a metal or alloy and the patternmaker's shrinkage, is the apparent failure of a casting to register the true shrinkage taking place at and slightly below the melting point. The cause of this may possibly be due to the fact that material at the grain boundaries does not possess sufficient rigidity to register the actual contraction of the grains or crystallites.

The contraction of liquid metal as it cools and solidifies is distributed between external pipe and internal voids. Except where conditions are such that a completely rigid shell is formed about a cooling mass of molten metal before the interior has reached the freezing temperature, the shrinkage caused by contraction of the liquid interior will result in a depression in the upper surface (external pipe). Shrinkage that takes place during the change of state may contribute largely to the production of internal voids, when such tendency is exhibited by the metal or alloy.

The distribution of the shrinkage, or contraction, during solidification, in the form of an external pipe, internal pipe, or small cavities (porosity) throughout the casting, will depend largely on the following factors: (a) The magnitude of the shrinkage during solidification, (b) the freezing temperature of the metal, (c) type of metal,

whether it is pure, eutectic, intermetallic compound, solid solution, or any combination of these, (d) fluidity of the metal, (e) thermal conductivity, heat capacity, and pouring temperature of the metal, (f) location and design of gates and risers, (g) ratio of surface area to volume, and (h) heat capacity, thermal conductivity, and initial temperature of the mold material.

In 1932 Saeger and Ash [40] described a method for determining the volume changes occurring when a molten metal is cooled to room temperature. They obtained data from which specific volume²-temperature curves could be constructed for the range from the molten state to room temperature.

In figure 14, $V_a - V_b$ represents the change in specific volume with change in temperature of a metal in the liquid state as it cools from some temperature, a , to the freezing temperature, b . The interval $V_b - V_c$ represents the change in specific volume of a metal in passing from the liquid state at temperature b , to the solid state at temperature c . The interval $V_c - V_d$ represents the change in specific volume of the solid metal as it cools from the melting point c , to room temperature, d . The change in volume, in percent, for any interval can be calculated from the difference in specific volume for the interval; thus $100(V_a - V_b)/V_a$ represents this change on cooling the metal from temperature a to temperature b .

(a) Liquid Shrinkage

Saeger and Ash [40] devised an apparatus for determining the specific volumes of molten metals and alloys over a wide range of temperatures. The apparatus is shown in figures 15 and 16. It is essentially a pycnometer, but is designated a "crucible immersion apparatus." The crucible, lid, and supporting rods were made of Acheson graphite.

Graphite is unsatisfactory for the sampling of cast iron because of the reaction between the iron and the graphite, which changes not only the dimensions of the crucible but also the composition of the iron. It was found, however, that graphite can be protected from the action of molten cast iron by surfacing it with a refractory "wash." Zirconium-oxide powder to which had been added a small amount of colloidal clay for bond and mixed with water to a "consistency of cream" was found to be satisfactory.

The temperature of the molten metal is measured by means of a platinum-platinum-rhodium thermocouple within a glazed-porcelain protection tube placed inside a graphite tube 1 in. in diameter. The graphite tube is treated with the refractory coating described previously, when it is to be immersed in cast iron.

² Specific volume is the reciprocal of the density, or the volume in milliliters per gram.

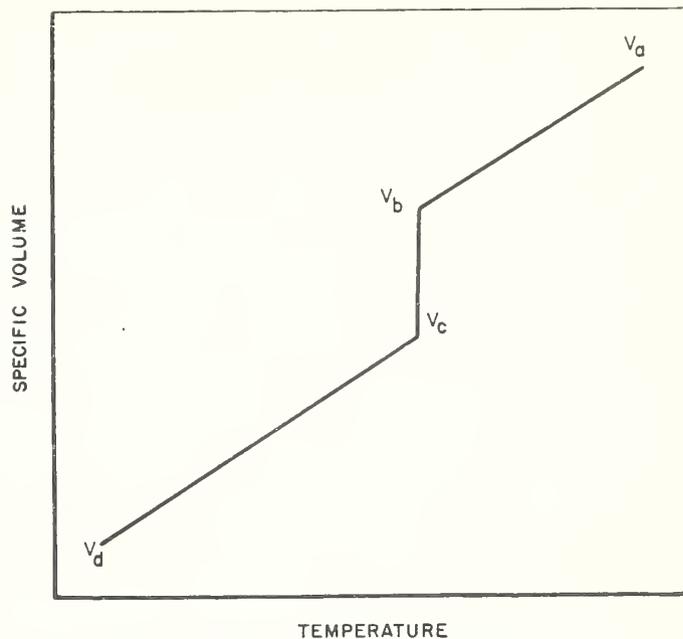


FIGURE 14. Typical specific volume-temperature curve of a metal (Saeger and Ash).

(b) Solidification Range

For alloys that solidify over a freezing range, it is necessary to determine the freezing point (the temperature at which incipient crystals form on cooling) and the melting point (the temperature at which incipient fusion begins on heating). These temperatures may be determined by a thermal-analysis method. The interval between the freezing point and the melting point is the solidification range.

With this apparatus it is possible to isolate a known volume of liquid metal at a definite and predetermined temperature from a ladle of molten metal. The procedure is as follows: The crucible is filled by immersing it in a ladle of molten metal and sufficient time is allowed for the crucible to come to the temperature of the surrounding metal, or to allow both crucible and metal to cool to the temperature at which it is desired to obtain a sample. When the desired temperature is reached the lid is pressed down firmly on the crucible, and the whole assembly removed from the ladle of metal.

The sample of metal thus obtained is allowed to freeze within the crucible and to cool to room temperature. Obviously the mass of the metal does not change on cooling. The resulting ingot is weighed at room temperature. The specific volume of the metal at the temperature of sampling is calculated by dividing the volume of the crucible at that temperature by the weight of the ingot.

The volume of the crucible at room temperature was determined by weighing the mercury required to fill it, when the lid was pressed down firmly. The volumes of the graphite crucible at elevated temperatures were calculated from data on linear thermal expansion of graphite.

Saeger and Ash [40] used cast samples 5 in. long and 1½ in. in diameter to determine the solidification range. A ⅜-in. hole, 4 in. long, was drilled along the longitudinal axis of the cylindrical sample into which was inserted a platinum-platinum-rhodium thermocouple protected by means of a glazed-porcelain tube. The sample was mounted in a 4-in. coil of a high-frequency induction furnace and the intervening space filled with zircon sand. The electromotive force developed by the thermocouple was measured by means of a potentiometer. The time in seconds required for each successive change of 0.1 mv was recorded, and inverse-rate heating and cooling curves of the sample were obtained. The freezing point was taken as that temperature at which the first break occurred in the cooling curve, and the melting point as the first break in the heating curve.

(c) Solid Shrinkage

In addition to determining the specific volume-temperature curve for the liquid metal, it is neces-

sary to determine a similar curve for the metal on cooling in the solid state. Thermal-expansion data for many metals are available in the literature, and can be used for calculating the specific volume-temperature relations for solid metals. In the case of gray cast iron, however, thermal-expansion data obtained by heating a gray-iron casting will not answer the purpose because heating causes permanent growth as indicated in figure 17. It is necessary, therefore, to obtain data on the contraction of gray cast iron as it cools from the melt.

The dimensional changes occurring during the cooling of cast iron are affected by the separation of graphite. On freezing and during cooling, the cementite undergoes a partial transformation to ferrite and graphite. As the specific volume of graphite is about three times that of cementite, the transformation results in an increase in volume. This increase varies with the amount of cementite decomposed and therefore is dependent on the composition of the cast iron, particularly the carbon, silicon, and manganese contents, and on the rate of cooling. The dimensional changes

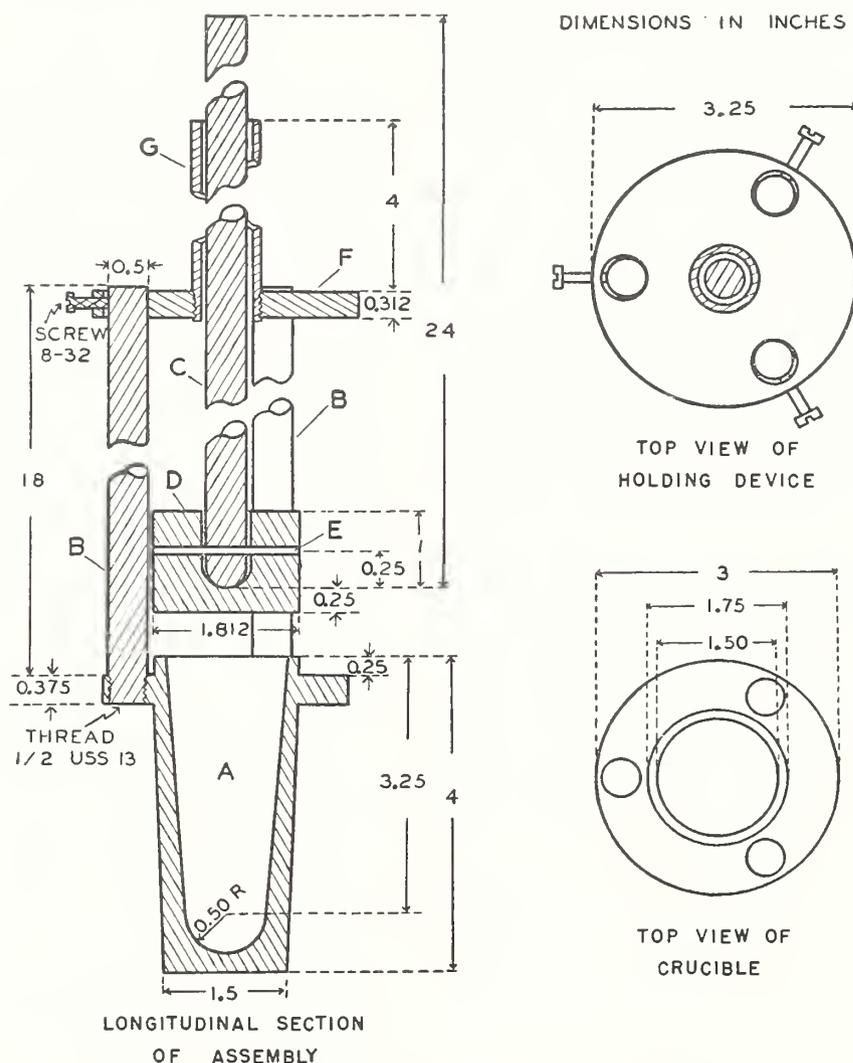


FIGURE 15. Crucible immersion apparatus (Saeger and Ash).

A, Crucible; B, support rods; C, control rod; D, lid; E, sustaining pin (4-mm carbon arc rod); F, metal base; G, metal handle.

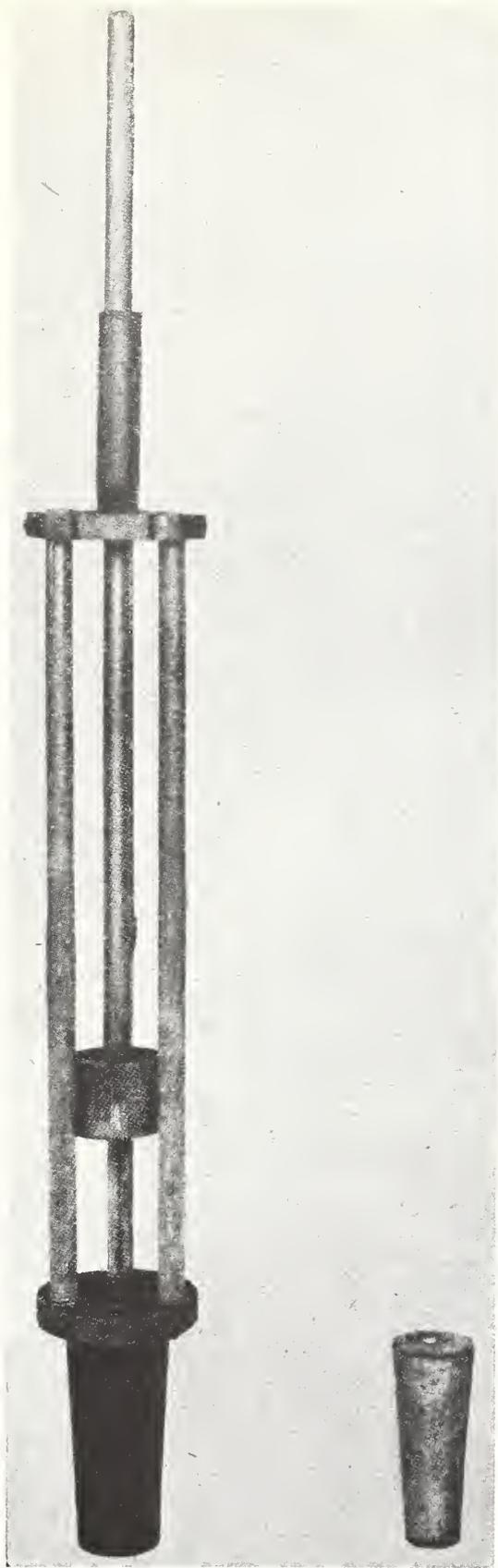


FIGURE 16. Crucible immersion apparatus and ingot (Saeger and Ash).

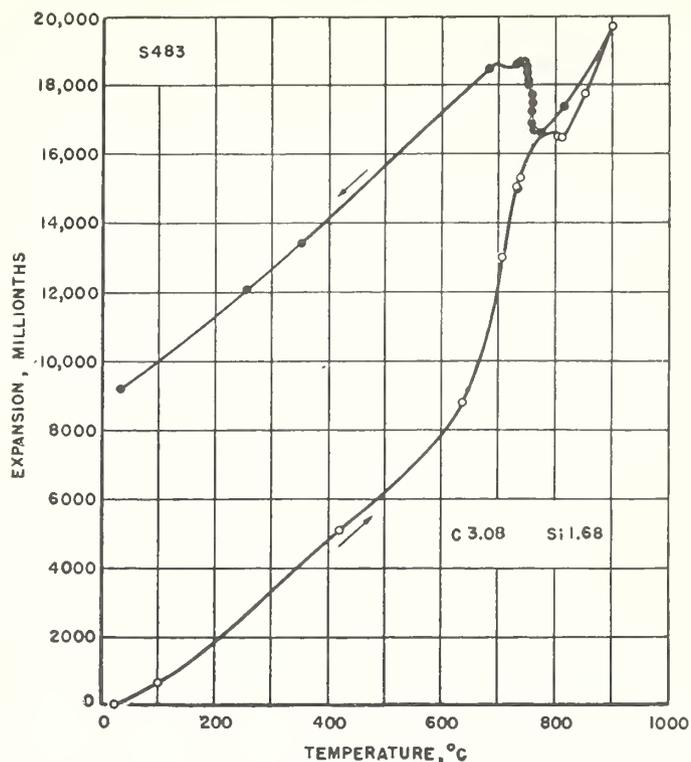


FIGURE 17. Curve showing linear thermal expansion and growth of cast iron.

resulting from the graphite separation are affected by factors that affect the cooling rate, such as pouring temperature, thermal conductivity of the mold and metal, temperature of the mold, size of casting (total heat content), and thickness of cross section.

Figure 18 is a sketch of the apparatus used by Saeger and Ash [40] for determining the linear contraction of cast metals and alloys. The portion of the cast bar beyond the fixed pin can be of any convenient length, and the cross section can be made to any convenient dimensions.

A fused-quartz rod was used to indicate the linear movement of the cast bar on cooling. The rod was allowed to project into the mold cavity about $\frac{1}{8}$ in. to insure that it would be firmly gripped by the metal on casting. The small movement of the cast bar as transmitted through the fused-quartz rod was measured by means of a microscope mounted on a micrometer slide.

It was essential that the average temperature of the cast bar be obtained. In order to secure this, three thermocouples were located in the 12-in. cast bar at distances $1\frac{1}{2}$, 6, and $10\frac{1}{2}$ in., respectively, from the free end of the bar. Saeger and Ash assumed that an average of the three temperatures measured in this manner represented the average temperature of the bar. In practice, the three thermocouples, all of the same length and size, were joined in parallel, and the average temperature of all three was obtained by one

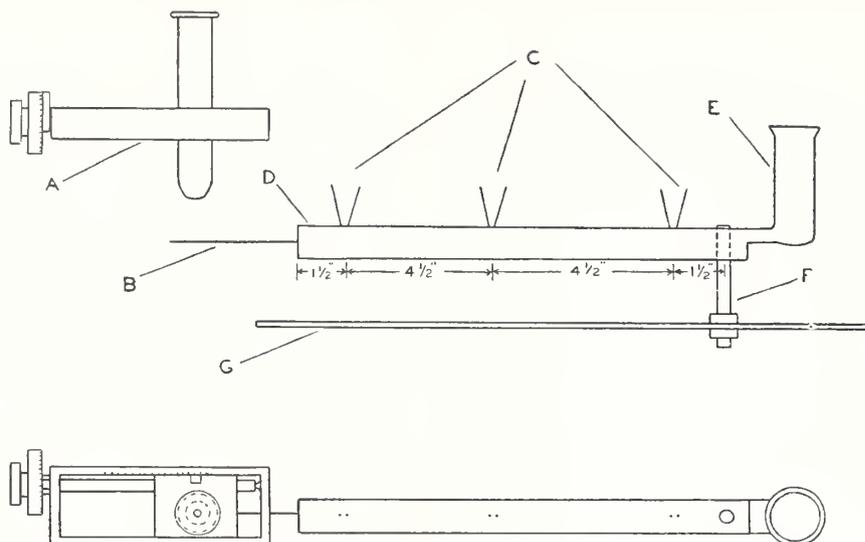


FIGURE 18. Sketch of apparatus for determining linear contraction of metals and alloys (Saeger and Ash).

A, Micrometer slide and microscope; B, fused-quartz rod; C, thermocouples; D, shrinkage bar; E, pouring gate; F, fixed steel pin; G, steel plate.

reading of the instrument. Chromel-alumel thermocouples and a portable potentiometer indicator were used to indicate temperatures. The wires of the thermocouples were insulated from each other and from the molten metal by means of a sodium silicate silica-flour cement. The welded tip of each thermocouple extended into the mold cavity to one-half the thickness of the resulting bar.

The procedure was as follows: A shrinkage bar was cast by pouring molten metal from a ladle into a green-sand mold. The temperatures of the cast bar were read simultaneously with observations on the movement of the fused-quartz rod. The contraction curve was obtained from these data.

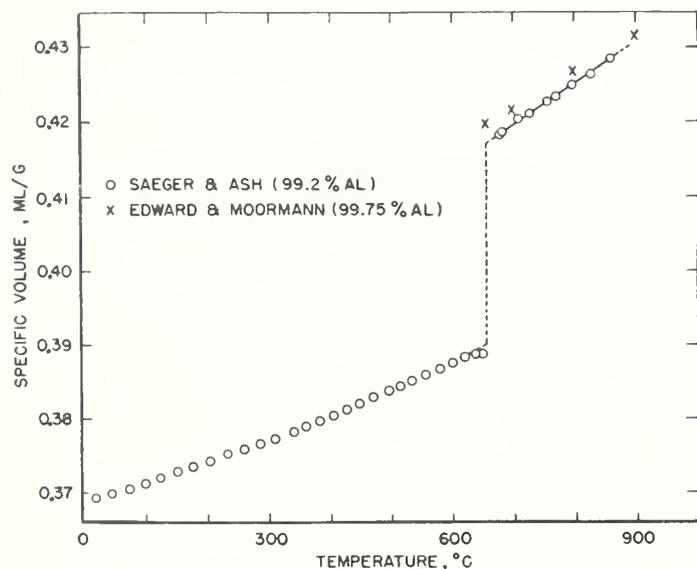


FIGURE 19. Specific volume-temperature curve for aluminum (Saeger and Ash).

Solidification shrinkage 6.6 percent; solid shrinkage (658° to 25° C) 5.5 percent.

(d) Specific Volume-Temperature Curves

Saeger and Ash [40] determined the specific volume-temperature curves for some nonferrous metals and cast irons. The procedure used in obtaining the specific volume-temperature curve for aluminum will be briefly indicated as an example.

The specific volume of liquid aluminum at 770° C was calculated as follows from experimental data obtained with the crucible immersion apparatus:

Specific volume of aluminum at 770° C

$$\begin{aligned} & \frac{\text{Volume of liquid aluminum sample at } 770^{\circ} \text{ C}}{\text{Weight of liquid aluminum sample at } 770^{\circ} \text{ C}} \\ &= \frac{\text{Volume of graphite crucible at } 770^{\circ} \text{ C}}{\text{Weight of aluminum ingot at room temperature}} \\ &= \frac{68.18}{160.99} = 0.4235 \text{ ml/g.} \end{aligned}$$

In a similar manner, the specific volume of liquid aluminum was determined at other sampling temperatures. By plotting these values, the upper portion of the curve in figure 19 was obtained. This portion of the curve represents the specific volume of liquid aluminum at various temperatures.

Measurements of changes of length with temperature were made on a 1-in. square bar of aluminum of 99.2 percent purity during the period of cooling from its melting point (658° C) to room temperature. As may be observed from the lower portion of the curve in figure 19, the

solid specific volume-temperature curve tends to flatten out just below the melting point, as the cast bar may not have been sufficiently rigid at this high temperature to register the true contraction taking place. This condition was overcome by extrapolating the main portion of the curve to the melting point. The observed linear contraction of the bar (12 in. long at the casting temperature) from the melting point to room temperature was 0.213 in. (1.78 percent) and as corrected for the extrapolated portion, 0.222 in., or 1.85 percent.

The cubical contraction was taken as three times the linear contraction. The value 5.55 percent (3×1.85 percent) represents the decrease in volume undergone by aluminum in cooling from the melting point to room temperature, and agrees closely with the value 5.57 percent for the cubical contraction of aluminum of the same purity calculated, by extrapolation from linear thermal-expansion data given by Hidnert [41].

The specific volume of a sample cut from this cast bar of aluminum, after forging at 400°C and annealing at 450°C , was determined as 0.3683 ml/g at 20°C . The corresponding specific volume of aluminum in the solid state at 658°C is 0.3899 ml/g, computed on the basis of the specific volume of the metal at 20°C and the cubical contraction of 5.55 percent on cooling from 658° to 20°C .

By extrapolating that portion of the curve (fig. 19) representing the specific volume of liquid aluminum, to the freezing point (658°C), the specific volume of liquid aluminum at the freezing point was found to be 0.4173 ml/g. The difference between the specific volumes of liquid and solid aluminum at 658°C , the change in the specific volume caused by solidification shrinkage, is equal to $0.4173 - 0.3899 = 0.0274$ ml/g. This is equivalent to 6.6 percent, based on the specific volume of the liquid at the freezing point.

The specific volume-temperature curve of aluminum from about 900°C to room temperature is shown in figure 19. Several values reported by Edwards and Moormann [42] were also plotted in this figure for comparison.

The linear contraction of commercial aluminum in the solid state, reported by Anderson [43] is appreciably less than the value calculated from data on linear thermal expansion. He found that his results for the linear contraction of aluminum and many of its alloys varied as follows: other conditions being equal, (a) the smaller the cross section of a cast bar of given length, the less the contraction, (b) the greater the length for a given cross section, the less the contraction, and (c) the contraction was less in chill molds than in sand-cast molds.

(e) Other Methods

Coleman [44] found a linear casting shrinkage of 1.25 percent for a gold-copper alloy containing 10 percent of copper, whereas the linear contraction of the solid alloy from the melting point to 20°C , computed from thermal expansion data, was 1.62 percent. The apparatus used for measuring the linear casting shrinkage is shown in figure 20. The procedure was as follows: The large caps were screwed tightly into the ends of the flask, and the small hollow plugs screwed into the caps and locked in position with lock nuts. A metal rod, inserted through and supported by the hollow plugs, served as the pattern. The sprue was attached to the pattern with wax, the crucible former placed in position and the flask filled with dental investment. After the investment had set, the crucible former, sprue, and pattern rod were removed. The hollow plugs were replaced with the solid plugs, thus forming a mold, the length of which is determined solely by the distance between the inside

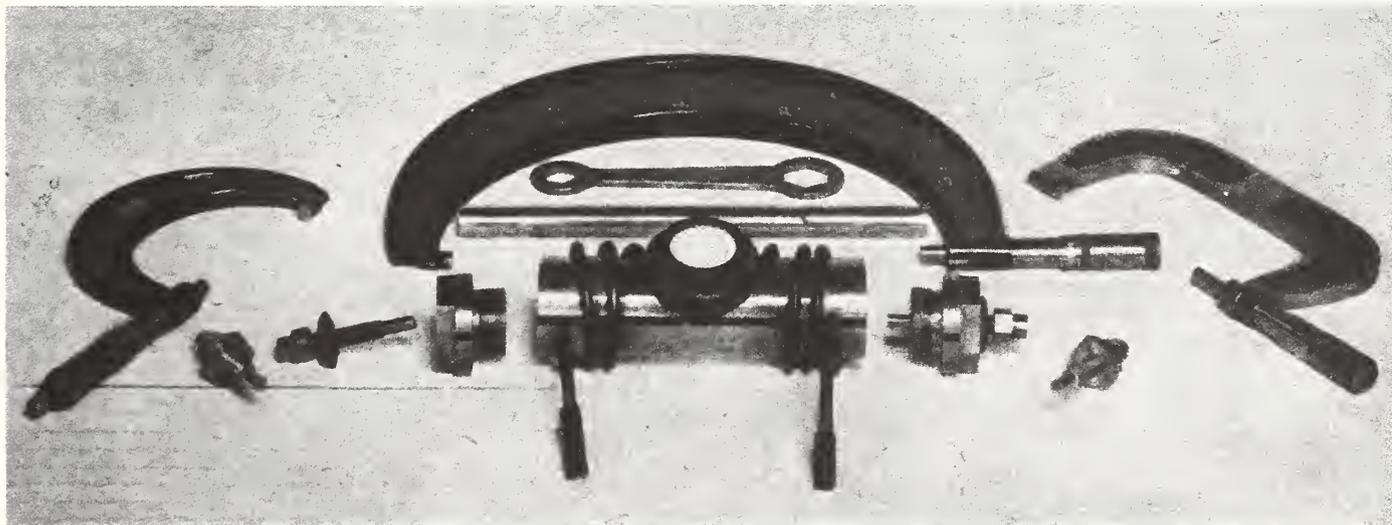


FIGURE 20. Apparatus for determining the linear casting shrinkage of gold alloys (Coleman).

faces of the solid plugs. The flask was then placed in an oven and "burned out" at the desired temperature. Castings approximately 3¼-in. long and 0.12 in. in diameter, were made with an air-pressure machine.

The metal parts of the apparatus are of an oxidation-resisting steel, the coefficient of linear thermal expansion of which is known. The temperatures of various parts of the flask were measured with small thermocouples and a potentiometer. The length, at room temperature, of each of the solid plugs was determined accurately. The distance between the outside faces of the solid plugs was measured with a micrometer. From these values the distance between the inside faces of the solid plugs, or the length of the mold, was computed.

Two possible explanations of the difference between the observed linear casting shrinkage of the gold alloy, and the calculated linear contraction from thermal-expansion data were suggested by Coleman [44]: (1) There may be

sufficient friction or interlocking between the casting and the walls of the mold to hold and stretch the casting while it is cooling through that range of temperature within which the metal is very soft or weak, thus preventing the full normal shrinkage, and (2) the compensation of part of the total shrinkage of the solid metal may be dependent upon a difference in the rates of cooling of different parts of the casting. If part of the metal in the mold solidifies and cools to some temperature below the melting point before the metal in the sprue freezes, the shrinkage caused by the cooling of this solid metal may be compensated by the addition of metal from the crucible.

Other methods that have been employed for measuring the volume changes undergone by a metal in cooling from the liquid state to room temperature have been reviewed by Saeger and Ash [40] under four headings—dilatometric; buoyancy; pycnometer; sand-cast sphere, cone, or cylinder.

III. Data on Thermal Expansion

From observations on the linear thermal expansion of a sample of material it is possible to derive an empirical equation showing the relation between linear expansion and temperature. For example, the following second-degree equation was derived for molybdenum (fig. 21) by the method of least squares:

$$\Delta L = 4.090(t + 142.5)10^{-6} + 0.00226(t + 142.5)^2 10^{-6}, \quad (25)$$

where ΔL represents the change per unit length from the length at the initial temperature -142.5°C , and t represents any temperature between -142.5° and $+305^\circ\text{C}$. The probable error per unit length of molybdenum was found to be $\pm 8.3 \times 10^{-6}$. This value may be affected by the departure of the expansion curve from the assumed parabolic law.

The first derivative of eq 25 gives

$$\frac{d}{dt} [\Delta L] = 4.090 \times 10^{-6} + 0.00452(t + 142.5)10^{-6} \quad (26)$$

and represents the tangent or instantaneous coefficient of expansion a_t , at any temperature t , between -142.5° and $+305^\circ\text{C}$.

Eq 25 can be transformed into the following form:

$$L_t = L_0 [1 + (4.73t + 0.00226t^2)10^{-6}], \quad (27)$$

where L_t is the length of the sample at any temperature t , between -142.5° and $+305^\circ\text{C}$, and L_0 the length at 0°C . In this equation, 4.73×10^{-6}

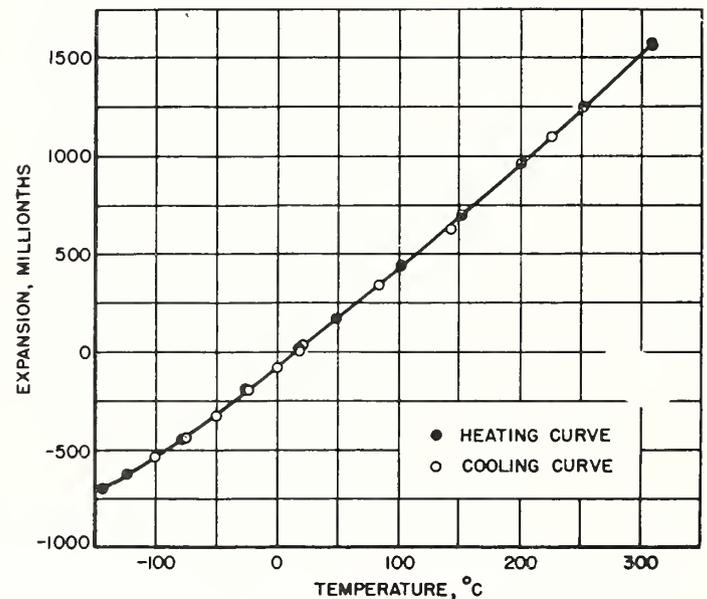


FIGURE 21. Linear thermal expansion of molybdenum.

represents the instantaneous coefficient of expansion at 0°C .

From observations with the initial temperature at any temperature (usually room temperature), Souder [45] in 1918 derived two normal equations from which it is possible to compute directly two constants of a second-degree equation in terms of L_0 , the length of a sample at 0°C . These normal equations are

$$a \sum (\Delta t)^2 + b \sum \Delta t^2 \Delta t = \sum e \Delta t \quad (28)$$

$$a \sum \Delta t^2 \Delta t + b \sum (\Delta t^2)^2 = \sum e \Delta t^2, \quad (29)$$

where a and b are constants, $\Delta t = t_n - t_1$, or the change in observed temperature from the initial temperature t_1 , $\Delta t^2 = t_n^2 - t_1^2$, or the change in the square of the observed temperature from the square of the initial temperature t_1 , and e is the corresponding observed linear expansion per unit length from the initial temperature.

The constants a and b may be obtained by solving these normal equations by determinants. This method gives

$$a = \frac{\sum (\Delta t^2)^2 \sum e \Delta t - \sum \Delta t^2 \Delta t \sum e \Delta t^2}{\sum (\Delta t^2)^2 \sum (\Delta t)^2 - [\sum \Delta t^2 \Delta t]^2} \quad (30)$$

and

$$b = \frac{\sum (\Delta t)^2 \sum e \Delta t^2 - \sum \Delta t^2 \Delta t \sum e \Delta t}{\sum (\Delta t^2)^2 \sum (\Delta t)^2 - [\sum \Delta t^2 \Delta t]^2} \quad (31)$$

IV. Relations between Thermal Expansion and Other Properties

From the data obtained on a sample by means of one of the dilatometric methods, it is possible to plot a curve which shows the relationship between

The values computed for a and b may then be substituted in eq 3.

Coefficients of expansion of various materials are given in the following publications:

International Critical Tables.

Landolt-Börnstein Physikalisch-Chemische Tabellen.

Smithsonian Physical Tables.

National Bureau of Standards Circular C447, Mechanical Properties of Metals and Alloys.

Handbooks.

A chart indicating the linear thermal expansion of 18 materials on heating from 0° to 100° C is shown in figure 22.

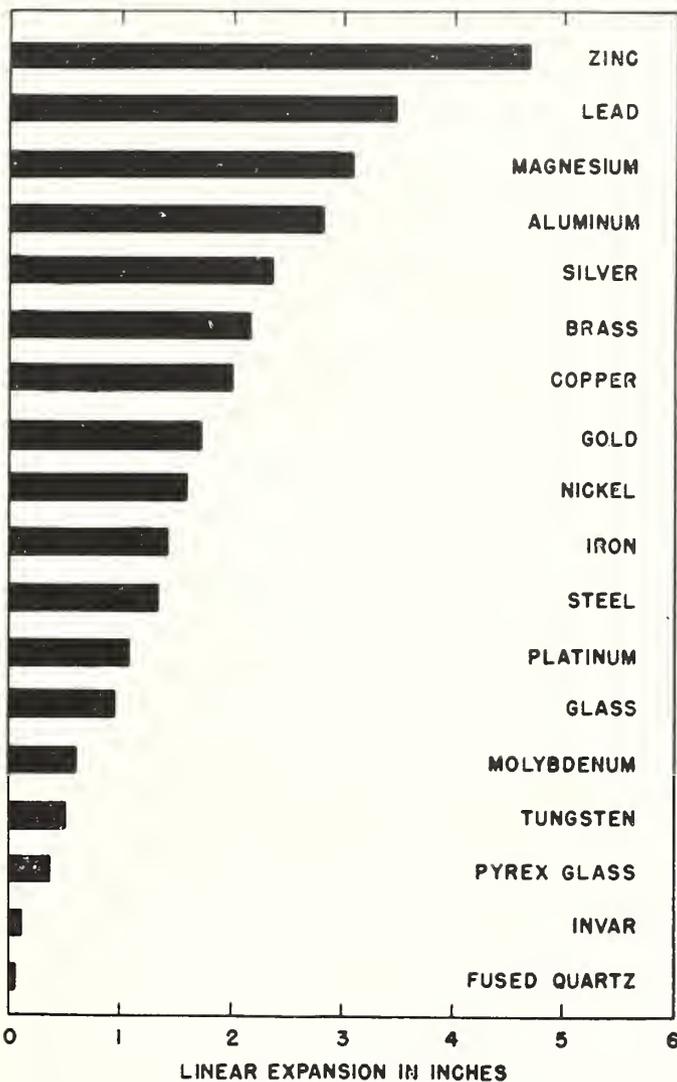


FIGURE 22. Linear thermal expansion of 100 feet of various materials from 0° to 100° C.

expansion and temperature, on heating and cooling. The curve on cooling may or may not coincide with the curve on heating, depending on the nature and the previous treatment of the sample. From the expansion curve it is possible to compute average coefficients of expansion for various temperature ranges, or an empirical equation may be derived from the original data.

Dilatometric methods permit the investigation of slow or rapid reactions, and a quantitative description of transformations. These changes observed during the heating and cooling of a sample are caused by transformations of the constituents (allotropic phase transformations, anomalous transformations without change of phase, and decomposition of phases) and by the reactions between phases. Iron is an example of a metal which undergoes a transformation with change of phase. When alpha iron (body-centered cubic crystal structure) is heated, it undergoes an allotropic transformation $\alpha \rightarrow \gamma$ at about 910° C, with a contraction for several degrees higher. On being further heated above this temperature (A_{c3}), gamma iron (face-centered cubic crystal structure) expands. On cooling, gamma iron undergoes an allotropic transformation $\gamma \rightarrow \alpha$ at about 900° C with an expansion. On further cooling below this temperature (A_{r3}), alpha iron contracts.

Figure 23 shows the linear expansion of a carbon steel on heating and cooling between 20° and 900° C. This steel expanded regularly to 729° C (A_{c1}) and then contracted to 759° C (A_{c3}). During this range from 729° to 759° C, alpha iron transformed to gamma iron, and iron carbide went into solution in gamma iron. At 759° C, the steel resumed its expansion at higher temperatures. On cooling, the steel contracted regularly to 685° C (A_{r3}), when expansion started with a transformation to alpha iron. The expansion continued on cooling to 659° C (A_{r1}). On further

cooling to 20° C, the steel contracted in a normal manner.

Dilatometric data may be used not only for obtaining relationships between thermal expansion and temperature, and for investigations of the constitution of metals and alloys, of dimensional changes on hardening steels, aging phenomena of alloys and steels, kinetics of transformations of austenite during cooling of steels and during isothermal holding below the eutectoid temperature, and of graphitization of cast iron; but also for obtaining relationships between thermal expansion, chemical composition, thermal and mechanical treatments of materials, and the like. For example, the following equation shows the relationship between linear thermal expansion and chemical composition of cold-rolled copper-zinc alloys:

$$a_{50} = (22.923 - 0.06833X + 0.0000695X^2)10^{-6} \quad (32)$$

where a_{50} is the instantaneous coefficient of expansion at 50° C, and X is the copper content between 62 and 97 percent by weight.

Grüneisen's law [46], first deduced empirically in 1908, states that for a metal the ratio of the coefficient of linear expansion to its specific heat at constant pressure is constant at all temperatures. Various theories of the solid state lead to the relation

$$3a = dbkc., \quad (33)$$

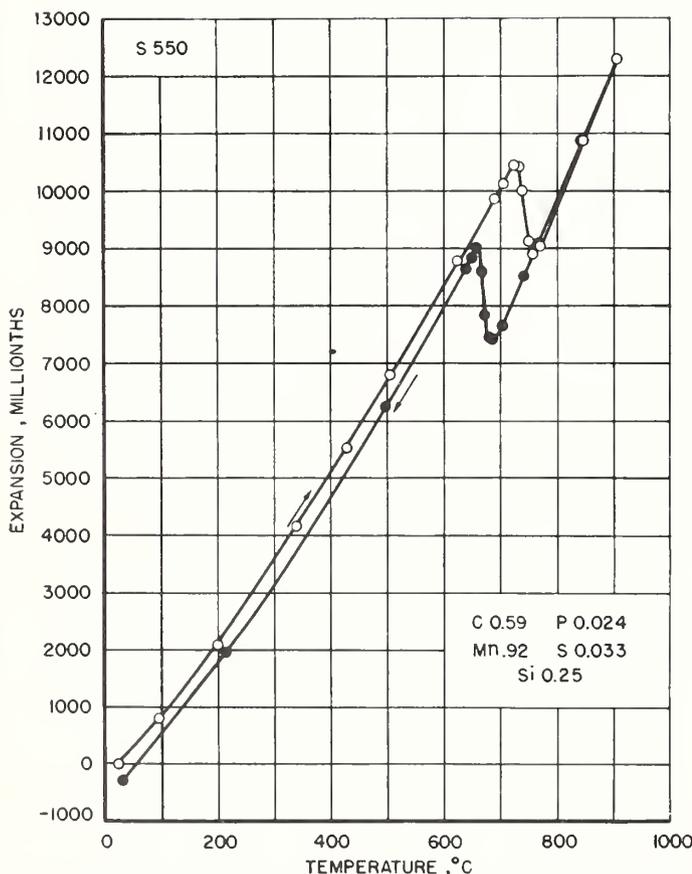


FIGURE 23. Linear thermal expansion and transformations of steel.

where a is the coefficient of linear expansion, d is the density, b is the compressibility, k is approximately a constant, and c_v is the heat capacity at constant volume. Wilson [35] showed that this relation may be derived by a method which is thermodynamical, except for the assumption of the Debye or some similar expression for c_v . Neither Grüneisen's law or eq 33 is well obeyed at high temperatures.

Hume-Rothery [47] adopted the following modification of Grüneisen's relation used by Simon and Vohsen [48]:

$$\frac{V_T - V_0}{V_0} = \frac{E_T}{Q_0 - kE_T}, \quad (34)$$

where

V_0 = volume at absolute zero

V_T = volume at $T^\circ K$

$$E_T = \int_0^T c_v dT$$

$$Q_0 = \frac{c_p}{3a} + 2kE_T \quad (\text{where } c_p \text{ and } a \text{ are the}$$

values of the specific heat and coefficient of expansion at room temperature)

$$k = \text{constant} = \gamma + \frac{2}{3},$$

where

$$\gamma = \frac{-V \frac{\partial V}{\partial T}}{c_v \frac{\partial V}{\partial p}}$$

Hume-Rothery stated that for most metals Q_0 is of the order 10^4 to 10^5 cal, k is a small number of the order 1 to 3, and E_T is of the order 1,000 at room temperature and increases by about 600 for each 100 deg rise of temperature. He found very good agreement between the observed and calculated changes in volume of the cubic metals silver, copper, and aluminum between absolute zero and a temperature of the order two-thirds of the melting point on the absolute scale, but for iron he found good agreement only up to the temperature at which the magnetic transformation begins.

Carnelley [49] and Lémeray [50] showed an approximate relation between the coefficients of linear expansion and the melting points of the chemical elements. Available coefficients of linear thermal expansion of the chemical elements at room temperature or for the range from 20° to 100° C versus their melting points (° K) are plotted in figure 24. The hyperbolic curve was derived from data on body-centered cubic and face-centered cubic elements, except manganese and the alkali metals. The curve indicates that the coeffi-

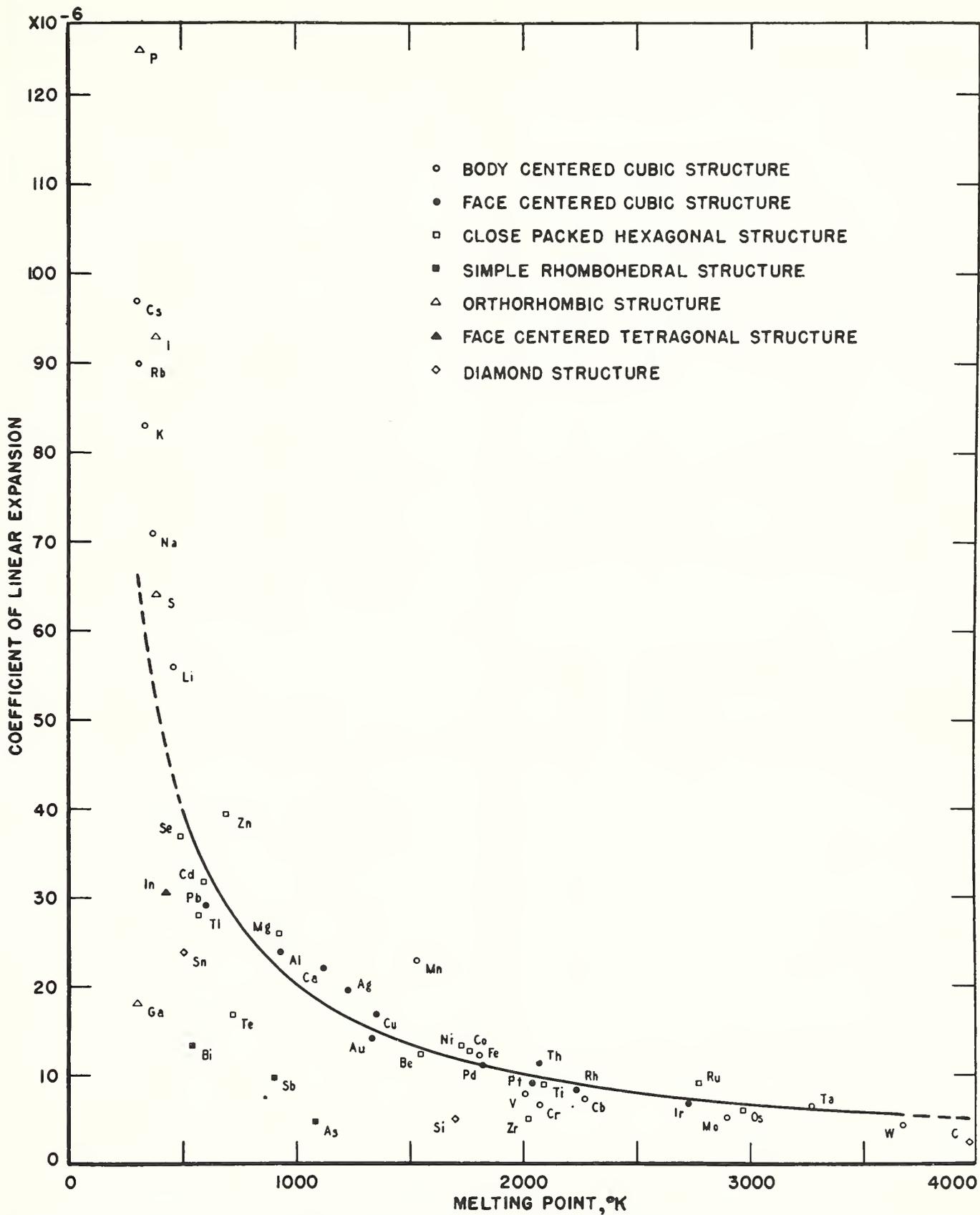


FIGURE 24. Relation between coefficients of linear thermal expansion (at room temperature or for the range from 20° to 100° C) and melting points of chemical elements.

Curve derived from data on body-centered cubic and face-centered cubic elements, except manganese and the alkali metals.

coefficients of linear expansion of the elements decrease as their melting points increase. The coefficients of expansion of the low-melting elements are relatively very large compared to those of the high-melting elements. Most of the elements having body-centered cubic, face-centered cubic or close-packed hexagonal structures lie close to the curve. The elements In, Sn, Ga, Bi, Te, Sb, As, and Si lie appreciably below the curve. Most of the elements that do not lie close to the curve have melting points below 1,000° K. The curve may be represented by the equation

$$a = \frac{0.020}{T}, \quad (35)$$

where a is the coefficient of linear expansion of a chemical element and T is its melting point (° K).

Wiebe [51] found the following relation between the coefficients of cubical expansion, specific heats, melting points, and atomic weights of the chemical elements that crystallize in the regular system:

$$\alpha = \frac{1}{2.6AcT}, \quad (36)$$

where α is the coefficient of cubical expansion, A is the atomic weight, c is the specific heat, and T is the melting point (° K). Since the coefficient of cubical expansion is three times the coefficient of linear expansion,

$$a = \frac{1}{7.8AcT}, \quad (37)$$

Table 2 gives a comparison of the observed and computed coefficients of linear expansion of 28 chemical elements that showed a difference of less than 6×10^{-6} . These elements have body-centered cubic, face-centered cubic, or hexagonal crystal structures (only one exception). The average difference between the observed and computed coefficients of expansion of these elements is $\pm 2.1 \times 10^{-6}$. The differences for other chemical elements on which data (atomic weight, specific heat, melting point, and coefficient of expansion) are available are considerably greater than the differences indicated in the last column of table 2.

With the aid of the law of Petit and Dulong [52], which states that the product of the atomic weight and the specific heat of an element is approximately constant, Wiebe [53] transformed eq 36 into

$$\alpha = \frac{1}{16.6T}, \quad (38)$$

where α is the coefficient of cubical expansion, and T is the melting point (° K).

TABLE 2. Comparison of observed and computed (eq 37) coefficients of linear expansion of 28 elements

Element	Crystal structure ^a	Observed coefficient of linear expansion, 20° to 100° C	Computed coefficient of linear expansion ($a = \frac{1}{7.8AcT}$) ^b	Difference
Aluminum.....	F	$\times 10^{-6}$ 23.8	$\times 10^{-6}$ 22.5	$\times 10^{-6}$ +1.3
Cadmium.....	H	31.8	34.9	-3.1
Calcium.....	F (H)	^c 22	18	+4
Chromium.....	B (H)	6.6	9.9	-3.3
Cobalt.....	H (F)	12.6	12.5	+0.1
Copper.....	F	^d 16.8	16.2	+0.6
Gold.....	F	14.1	15.7	-1.6
Iridium.....	F	6.8	7.6	-0.8
Iron.....	B (F)	12.2	11.8	+0.4
Lead.....	F	29.1	34.4	-5.3
Lithium.....	B	56	51	+5
Magnesium.....	H	26.0	22.9	+3.1
Molybdenum.....	B	5.2	7.1	-1.9
Nickel.....	H (F)	13.3	11.3	+2.0
Osmium.....	H	^e 6.1	7.3	-1.2
Palladium.....	F	11.1	11.1	0.0
Platinum.....	F	9.1	10.0	-0.9
Rhodium.....	F	8.3	9.3	-1.0
Ruthenium.....	H	^e 9.1	7.5	+1.6
Selenium.....	H	^c 37	39	-2
Silver.....	F	19.6	17.2	+2.4
Sulfur.....	O	^c 64	59	+5
Tantalum.....	B	6.6	6.0	+0.6
Thorium.....	F	11.3	9.5	+1.8
Titanium.....	H (B)	8.8	9.0	-0.2
Tungsten.....	B	4.3	5.6	-1.3
Vanadium.....	B	^e 7.8	10.9	-3.1
Zirconium.....	H (B)	5	10.5	-5.5

^a B=body-centered cubic; F=face-centered cubic; H=close-packed hexagonal; O=orthorhombic. The designation in parenthesis indicates a modification at higher temperatures.

^b In this equation, a =coefficient of linear expansion, A =atomic weight, c =specific heat, and T =melting point (° K). Specific heats at room temperature were used in computing the values given in this column.

^c At 20° C.

^d From 25° to 100° C.

If the coefficients of linear expansion (at room temperature or for the range from 20° to 100° C) versus the atomic numbers of the chemical elements are plotted, the periodic curve shown in figure 25 is obtained. Lithium, sodium, phosphorus, potassium, rubidium, iodine, and cesium appear at the maxima of the curve. Carbon, silicon, chromium, molybdenum, and tungsten are some of the elements that appear at the minima of the curve. A similar relationship is obtained if the products of the atomic volumes³ and the coefficients of linear expansion versus the atomic numbers are plotted.

Cork [54] gives an equation for the difference of the specific heats of solids at constant pressure and constant volume. This equation may be written as follows:

$$a = \frac{1}{3} \sqrt{\frac{(C_p - C_v)J}{eVT}}, \quad (39)$$

where a is the coefficient of linear expansion at $T^\circ K$, C_p is the specific heat at constant pressure, C_v is the specific heat at constant volume, J is

³ The atomic volume of a chemical element is equal to its atomic weight divided by its density.

the mechanical equivalent of heat, e is the coefficient of volume elasticity or bulk modulus, and V is the volume occupied by a gram or a gram-mole of the material (depending whether the specific heat at constant pressure is per gram or per gram-mole).

From available data on eight metals, Pictet [55] in 1879 derived a relation between thermal expansion, melting point, density, and atomic weight. The following equation was derived in 1947 from available data on the body-centered cubic and face-centered cubic elements except the alkali metals:

$$a = \frac{0.0465 \sqrt[3]{\frac{d}{A}}}{T}, \quad (40)$$

where a is the coefficient of linear expansion, d is the density (g/cm^3), A is the atomic weight, and T is the melting point ($^\circ\text{K}$). Table 3 gives a comparison of the observed and computed coefficients of linear expansion of these body-centered and face-centered cubic elements. Calcium, chromium, and manganese show large differences between the observed and computed coefficients of expansion. The average difference between the observed and computed coefficients of expansion of the 19 elements is $\pm 2.5 \times 10^{-6}$.

In connection with an investigation of bonding between plastics and metals, Turner [56] developed the following formula for the coefficient of cubical expansion, α_v , of a mixture in terms of the coefficients of cubical expansion, α , fraction

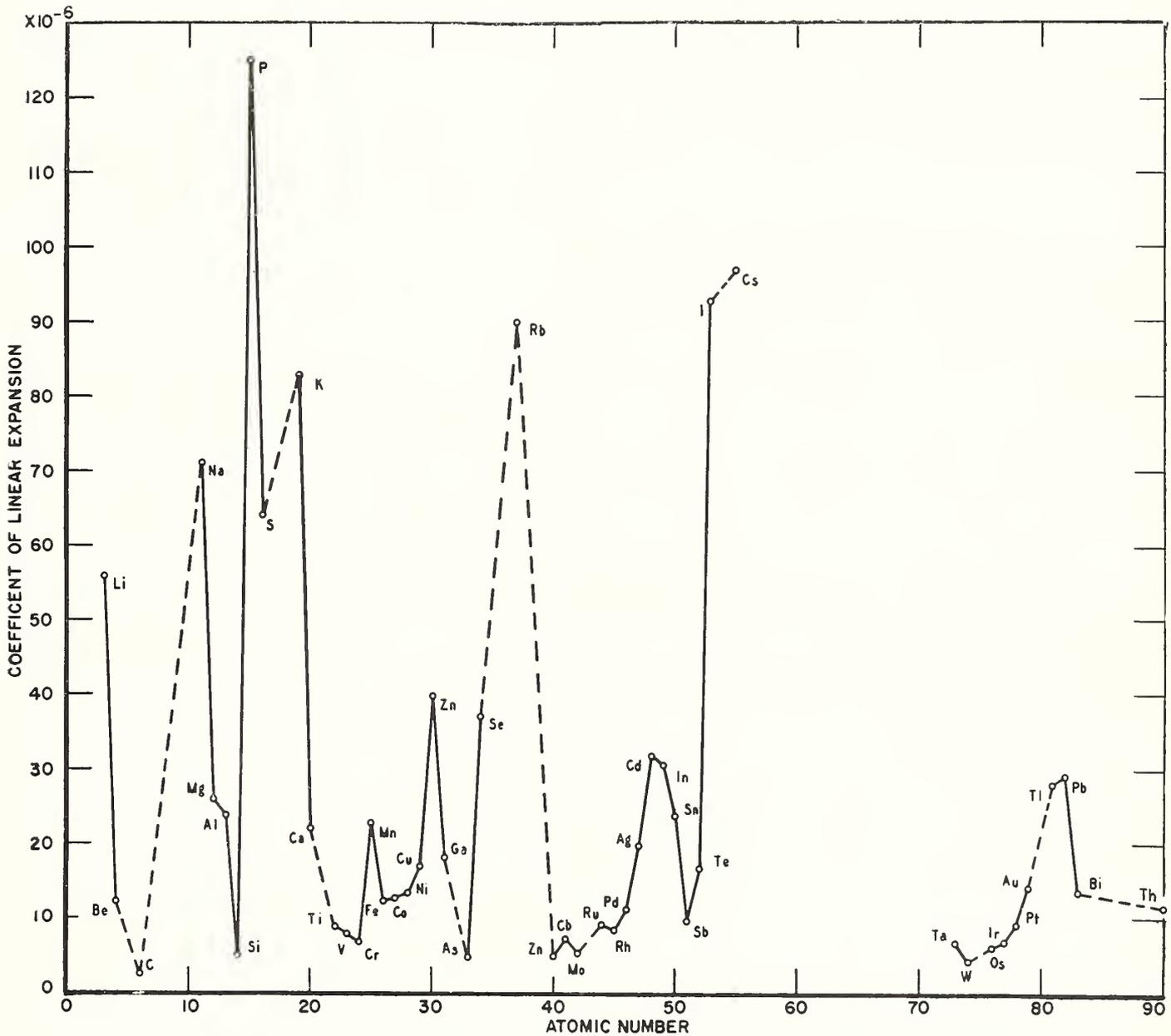


FIGURE 25. Relation between coefficients of linear thermal expansion (at room temperature or for the range from 20° to 100°C) and atomic numbers of chemical elements.

TABLE 3. Comparison of observed and computed (eq 40) coefficients of body-centered cubic and face-centered cubic elements (except alkali metals)

Element	Crystal structure ^a	Observed coefficient of linear expansion, 20° to 100° C	Computed coefficient of linear expansion $\left(a = \frac{0.0465 \sqrt[3]{\frac{d}{A}}}{T}\right)^b$	Difference
Aluminum	F	×10 ⁻⁶ 23.8	×10 ⁻⁶ 23.1	×10 ⁻⁶ +0.7
Calcium	F(H)	^c 22	14.0	+8.0
Chromium	B(H)	6.6	11.6	-5.0
Columbium	B	^d 7.2	9.2	-2.0
Copper	F	^e 16.8	17.8	-1.0
Gold	F	14.1	16.0	-1.9
Iridium	F	6.8	8.3	-1.5
Iron	B(F)	12.2	13.4	-1.2
Lead	F	29.1	29.4	-0.3
Manganese	B(T)	22.8	15.6	+7.2
Molybdenum	B	5.2	7.6	-2.4
Palladium	F	11.1	12.3	-1.2
Platinum	F	9.1	10.9	-1.8
Rhodium	F	8.3	10.3	-2.0
Silver	F	19.6	17.3	+2.3
Tantalum	B	6.6	6.4	+0.2
Thorium	F	11.3	8.2	+3.1
Tungsten	B	4.3	6.0	-1.7
Vanadium	B	^c 7.8	11.1	-3.3

^a B=body-centered cubic; F=face-centered cubic; H=close-packed hexagonal; T=face-centered tetragonal. The designation in parentheses indicates a modification at higher temperatures.

^b In this equation, a =coefficient of linear expansion, d =density, A =atomic weight, and T =melting point (°K).

^c At 20° C.

^d From 0° to 100° C.

^e From 25° to 100° C.

or percentage by weight, P , bulk modulus, K , and density, d , of the individual components:

$$\alpha_r = \frac{\frac{\alpha_1 P_1 K_1}{d_1} + \frac{\alpha_2 P_2 K_2}{d_2} + \dots + \frac{\alpha_n P_n K_n}{d_n}}{\frac{P_1 K_1}{d_1} + \frac{P_2 K_2}{d_2} + \dots + \frac{P_n K_n}{d_n}} \quad (41)$$

If the coefficients of cubical expansion of all components are equal to the same constant times their coefficients of linear expansion, then a can be substituted for α in eq 41 to obtain

$$\alpha_r = \frac{\frac{a_1 P_1 K_1}{d_1} + \frac{a_2 P_2 K_2}{d_2} + \dots + \frac{a_n P_n K_n}{d_n}}{\frac{P_1 K_1}{d_1} + \frac{P_2 K_2}{d_2} + \dots + \frac{P_n K_n}{d_n}} \quad (42)$$

where a is the coefficient of linear expansion.

Equation 41, based on stress equilibrium, reduces to a percentage by volume calculation if the ingredients have the same bulk moduli. If the ingredients have the same modulus to weight ratios, the calculation amounts to a percentage by weight interpolation.

The size and shape of the filler particles in plastic mixtures have an effect on the resultant coefficient of expansion of the mixture. Equations 41 and 42 do not take this phenomenon into consideration. Difficulty is also encountered because the bulk moduli of some materials are not available. To solve these problems, eq 41 and 42 may be modified by substituting an empirically determined constant C for K/d for each material. Constant C is interpreted as proportional to the modulus-density ratio rather than being equal to it.

For a mixture with components having nearly equal values of Poisson's ratio the bulk moduli are nearly proportional to the corresponding Young's moduli. For such mixtures Young's modulus, E , may therefore be substituted for bulk modulus in eq 42 to yield the following expression for the coefficient of linear expansion of a mixture:

$$\alpha_r = \frac{\frac{a_1 P_1 E_1}{d_1} + \frac{a_2 P_2 E_2}{d_2} + \dots + \frac{a_n P_n E_n}{d_n}}{\frac{P_1 E_1}{d_1} + \frac{P_2 E_2}{d_2} + \dots + \frac{P_n E_n}{d_n}} \quad (43)$$

Thus, in many cases where the bulk moduli are not available, eq 43 may be used instead of eq 42.

V. Applications of Thermal Expansion

The problems in which the thermal expansion of materials must be recognized are as varied as our industries. The precision chronometer is useless without proper temperature compensation. The elaborate suspension bridge carrying thousands of tons of traffic hourly would be unsafe if in its design and construction provisions for changes in dimensions of its members incident to temperature changes had not been made. Precision instruments such as indicators for measuring lengths, composed of parts having differing expansivities may give erratic indications unless the temperature is maintained constant.

One of the most important uses of thermal expansion is in thermostats of various types (differential expansion, bimetallic flexure, and fluid

expansion). Thermostats may be used in actuating circuit controllers for electrical devices such as electric furnaces, electric irons, electric refrigerators, vulcanizers, alarm devices, and stack controls, in the operation of mechanical controls such as means for regulating the flow of gas to gas ovens, and in pyrometers for indicating temperatures.

1. Thermostats by Differential Expansion

A large differential thermal expansion between two materials offers a basis for controlling and indicating temperature over a useful temperature range. For example, the change may be linear by

placing one material within the other, as an aluminum rod within a fused-quartz tube. If the two materials are attached at one end and heated or cooled, a differential motion is obtained at the other end. The linear motion may be converted into angular movement that may be magnified mechanically, if necessary.

2. Thermostats by Bimetallic Flexure

Thermostat metal (or bimetal) may be prepared from two strips of metals or alloys having widely different coefficients of expansion, by welding the strips throughout their entire length. Heating or cooling a thermostat metal produces a change of curvature of the thermostat metal, as indicated in figure 26. Heating a straight narrow piece of thermostat metal will cause it to bend and form an arc of a circle with the low-expanding metal on the inner side. Conversely, if the straight narrow piece of thermostat metal is cooled, the high-expanding metal will be on the inner side of the arc of a circle. The action caused by the change of curvature of thermostat metal can be converted into a linear or angular movement.

Commercial types of thermostat metals are available for various temperature ranges between -50° and $+1,200^{\circ}$ F (-46° and $+649^{\circ}$ C). These thermostat metals may be classified as "low-temperature" and "high-temperature" types. The low-temperature group includes invar in combination with brass or bronze. The high-temperature group includes all those thermostat metals that can be used at higher temperatures than those using brass or bronze. For high-temperature use, brass or bronze has been replaced by stronger alloys such as nickel-copper alloys and nickel-chromium stainless alloys, to increase the temperature range of uniform deflection. It is desirable that the thermal expansion and contraction of the metals or alloys selected for use in a thermostat metal, should be reversible on heating and cooling in the temperature range in which the thermostat metal can be subjected in use, in shipment, and in the process of mounting by welding, soldering, or brazing. Thermostat metal should be properly heat treated in order to relieve internal stresses set up during the working and forming of the metal.

The fundamental relation between the properties of the two metals or alloys (elements 1 and 2) of a narrow⁴ thermostat metal when heated or cooled, may be expressed by the equation⁵

$$\frac{1}{R} = \frac{6(\Delta a)(\Delta T)(t_1 + t_2)t_1t_2E_1E_2}{3(t_1 + t_2)^2t_1t_2E_1E_2 + (t_1E_1 + t_2E_2)(t_1^3E_1 + t_2^3E_2)}, \quad (44)$$

⁴ A flat, wide strip of thermostat metal when heated or cooled will assume the shape of a portion of a curved tube.

⁵ The equations in this subsection were obtained from catalog (1935 edition) by the H. A. Wilson Co., Newark, N. J. and publication by Hood [57].

where

- R = radius of curvature of thermostat metal
- Δa = difference in coefficients of expansion of elements 1 and 2
- ΔT = difference in temperature
- t_1 = thickness of element 1
- t_2 = thickness of element 2
- E_1 = elastic modulus of element 1
- E_2 = elastic modulus of element 2.

When the elastic moduli are equal, eq (44) reduces to

$$\frac{1}{R} = \frac{6(\Delta a)(\Delta T)t_1t_2}{(t_1 + t_2)^3}. \quad (45)$$

When the elastic moduli are equal and $t_1 = t_2$, eq 44 reduces to

$$\frac{1}{R} = \frac{3(\Delta a)(\Delta T)}{2t}, \quad (46)$$

where t is the total thickness of the thermostat metal.

The coefficients of thermal expansion and the elastic moduli of most materials are not uniform over wide temperature ranges. For a limited temperature range the curvature can be expressed by the equation

$$\frac{1}{R} = \frac{2k(\Delta T)}{t}, \quad (47)$$

where k is a constant depending on the difference in the coefficients of thermal expansion and on the ratio of the elastic moduli of the two elements.

For a narrow straight strip of thermostat metal fastened at one end and free to move at the other end, the deflection or distance moved by the free end may be represented approximately by

$$d = \frac{L^2}{2R}, \quad (48)$$

where d is the deflection and L is the effective length of the strip. If the value of $\frac{1}{R}$ from eq 47 is substituted in eq 48, the following equation is obtained for the deflection:

$$d = \frac{k(\Delta T)L^2}{t}. \quad (49)$$

If the narrow strip of thermostat metal is shaped in the form of a U with arms of equal length, the deflection of the free end is given by

$$d = \frac{k(\Delta T)L^2}{2t}, \quad (50)$$

where L is the developed length of the strip.



FIGURE 26. Effect of heating and cooling thermostat metal (Hood).

For thermostat metal shaped in the form of a circular ring, the deflection of the free end is

$$d = \frac{k(\Delta T)L^2}{\pi t}, \quad (51)$$

where L is the developed length of the ring. This shape is used for restricted spaces in which auxiliary arms or levers are employed to obtain additional motion.

The force exerted at the end of a straight strip of thermostat metal fastened at one end and touching a stop at the other end may be represented by

$$P = \frac{k(\Delta T)wt^2E}{4L}, \quad (52)$$

where P is the force, and w is the width of the strip.

For a thermostat metal in the form of a helix or spiral, the angular rotation between the ends may be obtained approximately from the following equation:

$$\theta = \frac{360k(\Delta T)L}{\pi t}, \quad (53)$$

where θ is the angular rotation in degrees. The torque of a coil is

$$M = \frac{k(\Delta T)wt^2E}{6}, \quad (54)$$

where M is the torque.

Thermostat metal is also used in the form of a round disk pressed into a concave or convex shape so that on heating or cooling, the disk will buckle from one side to the other.

Figure 27 shows some of the shapes of thermostat metals. Additional information about these materials may be obtained from manufacturers of thermostat metals.

Methods of testing thermostat metals have been published by American Society for Testing Materials [58].

3. Thermostats by Fluid Expansion

The large cubical thermal expansion of some liquids and gases has been applied for thermostats and for pyrometers. In the mercurial thermostat the contact is made by the mercury column, which rises when heated and contacts electric wires embedded in the glass and projecting into the capillary tube. Another type of liquid thermostat consists of a bulb, capillary tube, and bellows filled with a suitable liquid. The expansion or contraction of the liquid on heating or cooling actuates the bellows. The mercurial thermometer is the most common application for indicating temperature. The tin pyrometer is another application in which a chemical element in the liquid state has been used in the measurement of high temperatures.

Gas-filled thermostats have wider application than liquid-filled thermostats on account of the greater temperature range of the former. The sensitive bulb may be connected by capillary tubing to a Bourdon tube spring wound into the form of a helix. When the thermostat is heated, the gas expands and exerts a pressure that causes the spring to unwind. In some fire-alarm systems, the operation depends on the expansion of air from the heat of the fire or air within a vent-compensated system, which deflects diaphragms forming one side of connected air cells to make an electrical contact instrumental in sounding the alarm. The operating pressure and vent are so adjusted that pressures built up from ordinary temperature changes will not cause false operation of the system. The air volume concerned can be contained in long runs of fine tubing or can be consolidated in clusters of tubing or in bulbs of relatively large volume.

A thermostat using ether vapor in a balanced mercury column system, similar to that described by Green and Loring [59] is used in the gage block constant-temperature room of the National Bureau of Standards and has been found to be highly sensitive. Green and Loring's thermostat bulb has been replaced by a spiral to increase the sensitivity. A displacement of $\frac{1}{2}$ in. corresponds to a change of about 1° C.

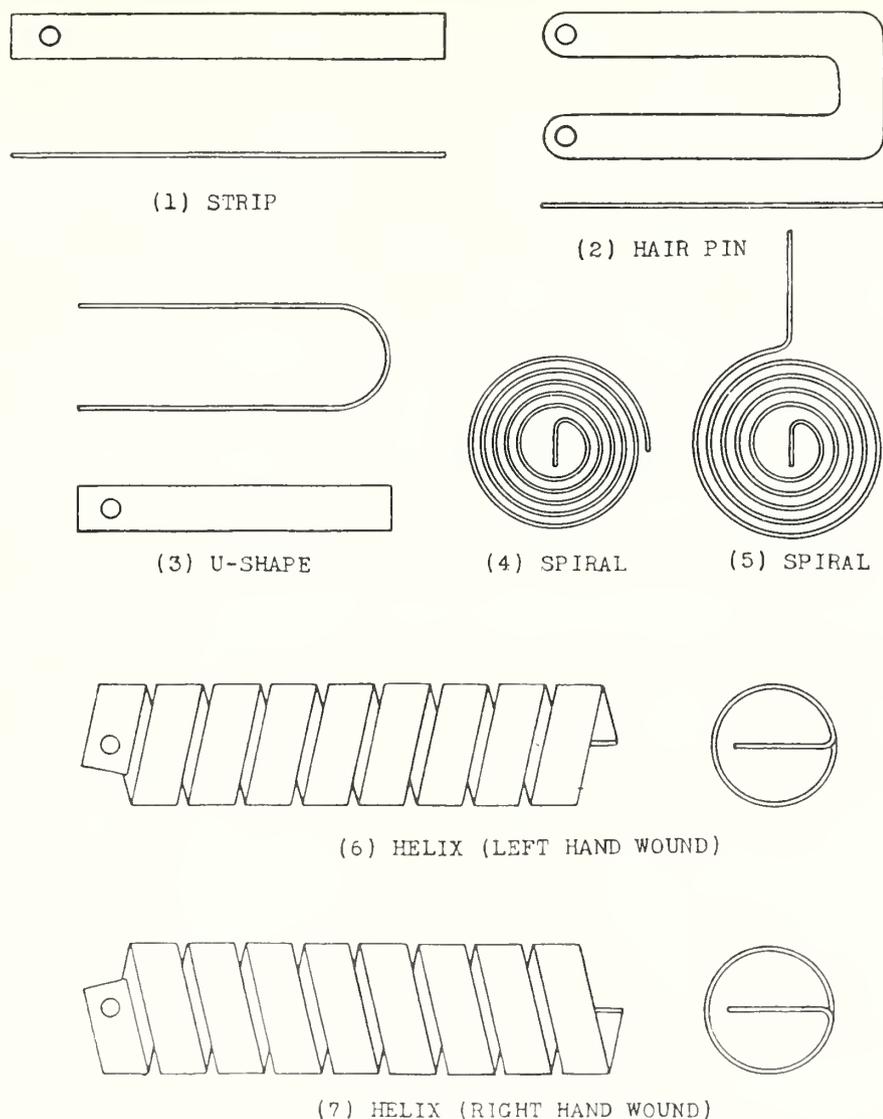


FIGURE 27. *Typical shapes of thermostat metal (Catalog, W. M. Chace Co., 1934).*

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WASHINGTON, April 4, 1949.

Spectrophotometry

(200 to 1,000 millimicrons)

By Kasson S. Gibson



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Preface

The spectrophotometer has long been recognized as a valuable analytical and standardizing instrument. For over 100 years photographic methods in the ultraviolet region of the spectrum, and visual methods in the visible region have afforded valuable means of research in the properties of matter, and the study of chemical structure. At first the spectrophotometer was seldom found outside of the college laboratory. Later it became a basic instrument also in the standardizing laboratory. With the development of the photoelectric cell, and the resulting design and construction of photoelectric instruments—direct reading, rapid to use, some of them automatically operated—the spectrophotometer found widespread use in the commercial laboratory, and thousands of them are now in daily use in research or control work.

Spectrophotometry is not only a research and an analytical tool in physics, chemistry, engineering, and technology. One of its most important uses relates also to the subject of color. It is the fundamental basis of colorimetric analysis, standardization, and specification. This has become axiomatic in psychophysical colorimetry. It is the only fundamental means of analyzing a color for research or other purposes. It is the only means of standardizing a color that is independent of material color “standards” (always of questionable permanence) and independent of abnormalities of color vision (existing among even so-called normal observers), and in this sense it is the fundamental basis for color specification.

The National Bureau of Standards has applied the spectrophotometer to its color research, development, and testing, and to various other work, for nearly 40 years. It pioneered in the development of the photoelectric spectrophotometer and in its application to the colorimetry of diffusing materials. With the advent of commercial photoelectric spectrophotometers, the Bureau began preparing and issuing spectrophotometric standards of various kinds, and hundreds of these standards are now in use, affording checks on the reliability of the various instrumental scales.

This Circular is issued to make available to users of spectrophotometers the experience, techniques, and data that have resulted from the Bureau's work in this field. Various instruments and methods are described, with emphasis on the basic principles, and various possible errors and the means used to eliminate them are considered in some detail. The various types of spectrophotometric standards are also described.

E. U. CONDON, *Director*.

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Spectrophotometry

(200 to 1,000 millimicrons)

By Kasson S. Gibson

This paper describes various spectrophotometric instruments and methods for use in the ultraviolet, visible, and near infrared spectral regions. These include photographic, visual, and photoelectric methods, but not the radiometric methods so widely used throughout the infrared. Errors and their elimination are discussed, and various types of spectrophotometric standards are described.

I. Introduction

1. Definition

Spectrophotometry is a term that by practically universal usage is taken to refer to the relative measurement of radiant energy or radiant flux as a function of wavelength. By strict derivation the term might be restricted to such measurements made visually, but in actual practice the word has long been applied also to photographic and photoelectric methods of measurement, in both the ultraviolet and the visible regions of the spectrum. More recently the term has been extended in application to cover also all such relative measurements in the infrared—throughout the region where the term “spectroradiometric” had heretofore been widely used, because the detectors necessarily used were of the type conveniently covered by the word “radiometer.” The present extended usage is good, however, as it frees the word “spectroradiometric” for the absolute measurement of spectral distributions, as contrasted with the relative measurements for which the word “spectrophotometry” is so generally applied.

The significance of the word *relative* in this connection is that in spectrophotometry the measurements are always made relative to some standard. What this standard may be in any case depends on the type of measurement. For example, in spectral reflection measurements it may be a standard magnesium oxide surface; in transmission measurements it may be either the blank beam or a similar material or cell free of the absorbing constituent; in measuring the spectral distributions of light sources it may be a source of known spectral distribution. But in all cases what is measured is merely the ratio of two quantities, wavelength by wavelength, throughout the spectral range of interest.

2. Applications

The numerous applications of spectrophotometry may be grouped into three main categories:

1. Spectrophotometry may be used as a general research tool in investigations of the properties of materials. These investigations may be concerned with the relation between absorption and constitution or structure, the eye-protective qualities of glasses and other materials, the connection between absorption and fluorescence, the study and selection of optical filters, the bleaching or rancidity of vegetable oils, the fading of dyed materials, and many other types of work.

2. Spectrophotometry is of great usefulness in the rapid chemical analysis of solutions, and other materials. Such measurements in the ultraviolet and visible spectral regions have long been valuable for this purpose, and in recent years similar measurements in the infrared have been found of immense importance, particularly in the petroleum industries. In the visible spectrum this type of analysis is often referred to as color analysis or chemical colorimetry, but such work should not be confused with the type of colorimetry referred to below in the third application.

It should be pointed out in this connection that the present paper is concerned primarily with instruments and instrumental techniques, particularly as bearing on the reliability of the data obtained, and not with the treatment of spectrophotometric data for any of the three categories listed. The applications and use of such data have been thoroughly covered in numerous publications in physical, chemical, and optical literature.

3. Spectrophotometry is the fundamental basis of colorimetric analysis, standardization, and specification. This has become axiomatic in psychophysical colorimetry. It is the only fundamental

means of analyzing a color for research or for other purposes. It is the only means of standardizing a color that is independent of material color "standards" (always of questionable permanence), and independent of abnormalities of color vision (existing even among so-called normal observers), and in this sense it is the basis for fundamental color specification. But while serving as the *basis* for color specification, a spectrophotometric curve (or table of data) is quite inadequate as a color specification in itself, particularly if colorimetric tolerances are desired as part of the specification. For adequate color specifications with tolerances the spectrophotometric data must be converted to some form of tristimulus specification by means of methods and data defining a standard observer, a standard coordinate system, and a standard illuminant.

Only in the last application above is the spectral range of interest limited to the visible spectrum. In the first two applications any division of the spectrum into ultraviolet, visible, and infrared is essentially arbitrary and pointless. For example, the Beckman quartz photoelectric spectrophotometer covers the range from about 200 to about 1,200 $m\mu$, and its usefulness is in no wise limited as we enter or leave either end of the visible spectrum.

However, in the neighborhood of 1,000 or 1,200 $m\mu$ (1 or 1.2 μ) there is a natural division, particularly as to instruments and methods. Neither the sensitized photographic plate nor the common types of photoelectric cells show much useful response above this wavelength. Radiometric methods on the other hand, with the thermopile, bolometer, or other similar detector, which are universally used throughout most of the infrared, are but little used below 1 μ , particularly since the development of the cesium oxide and other phototubes of high sensitivity. Accordingly the techniques used above and below 1 μ are in general widely different, even though the type of measurement is often essentially the same. To a less extent this wavelength marks also a division in the types of materials investigated—for example, petroleum products above 1 μ , dyes and camouflage materials below 1 μ —but with many other materials such as glasses no such division is apparent. This circular therefore covers the range from 200 $m\mu$ to 1,000 $m\mu$, approximately.

3. Historical

As already implied, the spectrophotometric methods to be considered here include the visual, the photographic, and the photoelectric. However, consistent with the purposes of the circular this is not primarily an historical résumé of the subject. Rather, an effort has been made to make the treatment such as to have the greatest utility to present-day users of spectrophotometers. Nat-

urally this results in considering spectrophotometers commercially available and widely used to a much greater extent than various spectrophotometers that have been individually designed and used in various laboratories, but are not readily available to others.

It is accordingly desirable to list various published articles on spectrophotometry or related subjects that treat the matter historically, are of résumé nature, or give extensive bibliographies, so that those who wish may pursue the subject to any desired extent. Listed chronologically, with comments, these are as follows, the complete reference being given in section VIII.

Spectrophotometry, Report of Optical Society of America Progress Committee for 1922-23. A fairly complete treatment of the science as it existed at that time [1].¹

The use of the photoelectric cell in spectrophotometry, K. S. Gibson. A résumé of the subject up to 1930 [2].

The practice of absorption spectrophotometry with Hilger instruments, F. Twyman and C. B. Allsopp (1934). Special emphasis on photographic methods, but describes other instruments [3].

Current advances in photographic photometry, G. R. Harrison (1934). Includes absorption spectrophotometry; contains references to work in extreme ultraviolet [4].

Visual spectrophotometry, K. S. Gibson. Describes visual instruments in common use in 1934 [5].

Measurement of Radiant Energy, W. E. Forsythe, Editor (1937). Contains the following chapters of interest in spectrophotometry: I, Fundamental concepts and radiation laws (Forsythe); II, Sources of radiant energy (Worthing); III, Analysis of radiation (Adams); IV, Spectrometric instruments and their adjustments (Kurtz); V, The spectrometer as an optical instrument (Barnes); VII, Measurement of spectral radiation by means of the photoelectric tube (Koller); VIII, Measurement of radiant energy with photographic materials (Jones); XI, (Visual) Spectrophotometry (Gibson); XIII, Photometry (Forsythe and Ives) [6].

The role of spectrophotometry in colorimetry, M. G. Mellon (1937). Of primary interest to the chemist [7].

Photoelectric methods in analytical chemistry, R. H. Müller (1939). Much of the information given would be useful in photoelectric spectrophotometry [8].

Spectrophotometric methods in modern analytical chemistry, S. E. Q. Ashley (1939). Of primary interest to the chemist [9].

Survey of spectrophotometers, Kasson S. Gibson (1940). Discusses the measurement of spectral directional reflectance [10].

¹ Figures in brackets indicate the literature references at the end of this paper.

Chemical spectroscopy, Wallace R. Brode (1943). Includes absorption spectrophotometry by visual, photographic, and photoelectric methods [11].

Spectrophotometry in medicine, Ludwig Heilmeyer, translated by A. Jordan and T. L. Tippell (1943). Absorption spectrophotometry [12].

Photoelectric photometers: Their properties, use and maintenance, J. S. Preston (1943). Much of the information given would be useful in photoelectric spectrophotometry [13].

Photometry and spectrophotometry, David L. Drabkin. A chapter in Medical Physics (1944). Absorption spectrophotometry, including filter photometry [14].

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metric methods of analysis for inorganic ions, J. W. Stillman (1945). Of primary interest to the chemist [15].

Absorption spectrophotometry, G. F. Lothian (1949). This is a revision and extension of Twyman and Allsopp's treatise, reference [3] above. [16].

Spectrophotometry and colorimetry, Wallace R. Brode. A chapter in Physical Methods in Chemical Analysis [17].

Practical Spectroscopy, G. R. Harrison, R. C. Lord, and J. R. Loofbourow (1948). Includes chapters on photographic photometry and absorption spectrophotometry [18].

Analytical Absorption Spectroscopy, M. G. Mellon, editor [127].

II. Nomenclature—Quantities Measured

In order to discuss the applications of spectrophotometry in the measurement of the spectral energy distribution of sources of light and radiant energy, and in the measurement of the spectral transmission and reflection characteristics of materials, it is necessary to define clearly various terms used in such work, and to assign symbols to these terms for convenient use.

Various groups and individuals have done this in the past, and various systems of terminology are thus available. Many of these are excellent in themselves, and they have served their purposes well. For example, may be noted (1) in radiation, the definitions and symbols proposed and used in the book entitled, Measurement of Radiant Energy, chapter 1 [6] (2) the photometric terminology prepared and issued by the Illuminating Engineering Society and the American Standards Association [19], and (3) various reports prepared by special committees of the Optical Society of America, dealing with colorimetry and spectrophotometry [1, 20, 21].

Naturally, however, such divergent groups and purposes have resulted in systems that are often inconsistent with one another. Furthermore, because of the extent and detail of some of these systems, recourse has often been made to Greek, bold-face, script, or other symbols that are inconvenient to use. Encouraging in this connection is the fact that the Illuminating Engineering Society Nomenclature Committee, and the Optical Society of America Colorimetry Committee have recently come into agreement on many terms and definitions relating to radiometry and photometry. In spectrophotometry, however, there is still no extensive agreement on terminology. In 1947, accordingly, a restricted set of terms for use in transmission and absorption measurements appeared in Letter Circular LC857 of the National Bureau of Standards [22]. These terms were agreed upon by various members of the optics and

chemistry groups of the Bureau and appear to be a satisfactory compromise of the various terms and symbols used in the past.

These transmission and absorption terms of LC857 are given in this Circular, together with a few other terms useful in the spectrophotometry of light sources and reflecting materials.

1. Spectral Energy Distribution

In any fundamental consideration of the radiation characteristics of light sources, or of sources emitting radiant energy at any wavelengths in the ultraviolet, visible, or infrared regions of the spectrum, one must consider the space and time variants involved in the radiation processes. This has been done to a greater or less extent in the nomenclatures and terminologies to which reference has just been made. In such we find, for example, definitions and terms for radiant energy, the time rate of flow of radiant energy, the solid-angular flux density in a given direction, the areal density of radiant flux from a source or on a surface, the space density of radiant energy, and so on, together with the luminous analogues for the same terms.

For our purposes *radiant energy* is defined as energy traveling through space in the form of electromagnetic waves of various wavelengths. It is the entity that is emitted from radiators; that is reflected, refracted, absorbed, or transmitted by various objects or materials; and that is incident upon the various detectors such as the thermopile, the photographic plate, the eye, and the phototube.

Radiant flux (power) is the time rate of flow of radiant energy.

In some cases the integrated amount of radiant energy is what is primarily measured by the detector; this is true of the photographic plate as customarily used and of the phototube if con-

ected to certain electrometers or "photon-counters". In other cases the radiant flux is the quantity which the detector primarily indicates. This is true of the eye, and of the phototube and thermopile with galvanometers or other devices as ordinarily used. It is often convenient, therefore, to consider the radiant flux as the entity to be measured or discussed.

In spectrophotometry, where at any frequency or wavelength only the ratio of two quantities is measured, it is usually possible and convenient to ignore the spatial and temporal variants of radiant energy and speak simply of the radiant energy itself, denoted graphically by an arrow and symbolically by the letter I . If I_0 refers to the standard at any wavelength and I to the unknown at the same wavelength, the quantity I is derived immediately from the measured ratio, I/I_0 , and expressed in whatever units are used or known for I_0 . In deriving the transmission terms given below, it will be noted that I and I_0 are no longer needed after the initial definitions of transmittance.

It is occasionally desirable, however, even in spectrophotometry, to refer to certain spatial variants of radiant flux and these are accordingly defined here. The terminology agreed to by the Optical Society of America and the Illuminating Engineering Society committees is followed.

Radiant intensity of a source of radiant energy is defined as the radiant flux per unit solid angle in a given direction.

Radiance is the radiant intensity per unit projected area of the source.

Irradiance is the radiant flux per unit area incident upon a surface.

Before considering the transmission and reflection terms, a few other comments may be made.

Radiant energy and light. Light is the aspect of radiant energy of which a human observer is aware through the visual sensations arising from stimulation of the retina of the eye. Quantitatively light is radiant energy evaluated in terms of the standard luminosity function (see section IV-1 below). Usually radiant energy must have wavelengths between approximately 380 and 770 $m\mu$ if it is to have a luminous aspect. But it is not proper to state that radiant energy of wavelengths 380 to 770 $m\mu$ is synonymous with light. Consistent with this definition of light, the expressions "ultraviolet light" and "infrared light" are incorrect, and the word "light" is seldom used in this Circular even when visual spectrophotometry is under consideration.

In this connection may be noted the luminous analogues for the radiation terms just defined, starting with light or luminous energy, the standard luminosity function being the correlating factor in every case. Thus may be defined *luminous flux*, *luminous intensity* (candlepower), *luminance* (photometric brightness) and *illuminance*

(illumination) [21, 19]. It may be further noted that new photometric units were introduced in this and other countries as of January 1, 1948 [23].

It is unfortunate that a short single word for radiant energy, analogous to light, has not come into common use. Lacking such a word we shall often use simply the word "energy", where the context makes clear that radiant energy is meant. The use of "radiation" or "radiations" for radiant energy, as for example in the expression "radiations of various wavelengths," is inconsistent with the modern usage that words ending in -tion (or -sion) are to be reserved for the designation of processes or for general and adjectival use.

Wavelength and frequency. The symbol for the wavelength of radiant energy is λ . The units in which wavelengths are expressed are restricted to those conventionally used for each spectral range; i. e., millimicrons ($m\mu$) or angstroms (\AA) for the ultraviolet and visible and microns (μ) for the infrared. The symbol for frequency is ν . In place of true frequency a term designated as *wave number* is widely used, defined as $1/\lambda_{\text{vac}}$ (the unit of λ in this usage being usually the centimeter).

The relation between wavelength and frequency is $\nu=c/\lambda$, where c is the velocity of propagation of radiant energy. It may perhaps be emphasized that both c and λ change whenever radiant energy of frequency ν passes from a medium of one refractive index to a medium of different index. The frequency itself does not change, however, and is thus more fundamental than wavelength for theoretical studies.

2. Spectral Transmission and Absorption

In this section all of the terms derived relate to the rectilinear transmission of radiant energy and no consideration is given to radiant energy that may be deviated or scattered from its original direction. By far the greater amount of spectral transmission and absorption measurements are of this type, made upon materials such as liquids, solutions, glasses, or other nonscattering materials. If the material is turbid or translucent, the radiant energy scattered from its original direction must be considered. If this scattered energy is slight in amount and unavoidable, it may need consideration along with other sources of error. If the material is more highly diffusing the measured transmittance will be importantly dependent upon the geometrical conditions of irradiation and reception; the situation is then analogous to that considered in some detail in section II-3 for reflection measurements.

1. *Terms relating to the rectilinear transmission of homogeneous radiant energy (that is, radiant energy of a "single" frequency) through a homoge-*

neous, isotropic, nonmetallic medium having plane, smooth, parallel surfaces.

In figure 1 let I_1 be the radiant energy incident upon the first surface of the sample, I_0 that entering the sample, I that incident upon the second surface, I_2 that leaving the sample.

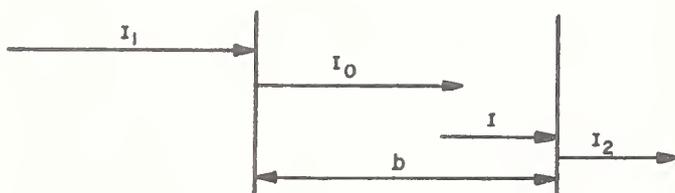


FIGURE 1. Transmittance, T , is defined as the ratio, I_2/I_1 ; internal transmittance; T_i , as the ratio, I/I_0 .

$T = I_2/I_1 =$ transmittance of the sample. (100 $T =$ percentage transmittance)

$T_i = I/I_0 =$ internal transmittance of the sample.

$A_i = -\log_{10} T_i = \log_{10} 1/T_i = \log_{10} I_0/I =$ absorbance of the sample.

Let b represent the length of absorbing path in the sample.

$a_i = A_i/b =$ absorbance index of the material.

Other quantities derived from the above are occasionally used. These are $T_i^{1/b}$, which has been called *transmissivity*, $1 - T_i^{1/b}$, which has been called *absorptivity*, and $1 - T_i$, which has been called *absorptance*.

In the above, the thickness, b , or the unit of thickness, should always be given. The temperature of the sample during the measurements should also be stated when this is a matter of importance.

2. *Terms relating to the rectilinear transmission of homogeneous radiant energy through a homogeneous, isotropic, nonmetallic medium such as a liquid, mixture, solution, vapor or gas confined between the windows of a cell.*

Let T_{soln} be the (over-all) transmittance of a given cell containing a solution or homogeneous mixture of solids, liquids, vapors, or gases, of which the compound of interest is the solute or one constituent. Let T_{solv} be the (over-all) transmittance of the same or a duplicate cell containing pure solvent, or containing the same mixture in the same relative proportions minus the constituent of interest.

$T_s = T_{\text{soln}}/T_{\text{solv}} =$ transmittancy of the sample. (100 $T_s =$ percentage transmittancy.) (T_s does not precisely equal $(T_i)_{\text{soln}}/(T_i)_{\text{solv}}$. However, with end plates of refractive index not greater than 1.5, the error is negligible for most purposes.)

$A_s = -\log_{10} T_s = \log_{10} 1/T_s =$ absorbancy of the sample.

Let b represent the length of absorbing path between the boundary layers of the solution, and

c the concentration of the solute or constituent of interest.

$a_s = A_s/bc =$ absorbancy index of the material.

a_M is the molar absorbancy index, ϵ being expressed in moles per liter, and b in centimeters, and the temperature being given.

In the above, the thickness and concentration, or the units, should always be given. The temperature of the sample during the measurements should also be given when this is a matter of importance.

In connection with these definitions it is desirable to point out certain alternative symbols and terms that have been widely used in chemical literature or elsewhere. Thus d, l , and x have often been used for thickness, rather than b . The symbol, T is often used for transmittancy, rather than T_s , particularly by chemists who in general have little interest in the concepts of transmittance and internal transmittance as defined above. Analogously, A and a would be used in place of A_s and a_s . However, the term, specific extinction, with symbol k , is widely used by chemists instead of the term, absorbancy index, a_s . Similarly the term, molar extinction, with symbol ϵ , is preferred in place of the term, molar absorbancy index, a_M . The molar extinction is usually recorded or plotted as $\epsilon \times 10^{-2}$ or as $\log_{10} \epsilon$.

3. Spectral Reflection

Differing from the case of spectral transmission measurements, where the great majority of substances measured are not importantly scattering, and accordingly where only rectilinear transmission was considered, most materials whose spectral reflecting properties are of interest are more or less highly diffusing. Such materials include papers, textiles, structural glass, paints, ceramics, etc., for which the analysis and specification of color is so important. However, the derivation of reflection terms herein is limited to those that are useful for spectrophotometry. Those who wish to consider the subject in greater detail should consult other papers on the subject [24, 25].

Three quantities are of primary importance in reflection spectrophotometry, designated as (total) reflectance, specular reflectance and directional reflectance. Again we are dealing with homogeneous radiant energy in these definitions.

Reflectance is defined as the ratio of the radiant energy reflected from an object to that incident upon the object. Since no restriction is placed upon the directions of the reflected energy this type of reflectance is often designated as the (total) reflectance. The (total) reflectance will vary with the direction or directions of the incident energy but cannot exceed unity.

Specular reflectance is the ratio of reflected to incident energy taken only in the direction of

mirror reflection. It is the type of reflection given by perfectly polished glasses, metals, or other mirror-like surfaces, without scatter. The specular reflectance of an object will vary with the angle of incidence but, again, cannot exceed unity.

Directional reflectance (formerly called apparent reflectance [24]) may be defined loosely as the reflectance that an object appears to have in any direction or group of directions. It is the type of reflectance measured by most reflection spectrophotometers and it is therefore of considerable importance that its meaning be fully understood, and the factors affecting its measurement be fully taken into account.

The concept is most easily defined in terms of a perfectly diffusing surface. With such a surface the ratio of the radiance to the irradiance (section II-1) is constant for all angles of reflection. Furthermore the magnitude of this ratio for a perfectly diffusing surface is independent of the angle or angles of irradiation provided only that the irradiance is constant. (If the radiant energy is in the visible spectrum, the perfectly diffusing surface will appear equally bright when viewed from any direction.)

The *directional reflectance* of a surface may accordingly be defined as the ratio of the radiance that the surface has under any specified set of irradiating and receiving conditions to the radiance that a perfectly reflecting, perfectly diffusing surface would have under the same conditions. Differing from the (total) reflectance or the specular reflectance, neither of which can exceed unity, the directional reflectance can have any value between zero and infinity.

Since no actual surfaces are perfectly diffusing the values of directional reflectance for any sample will depend on the geometrical conditions of both the irradiating and the receiving beams. The importance of the precise specification of these geometrical conditions depends on the purpose of the measurements and on the degree of departure of the surface from the completely diffusing condition. Which of the numerous possible conditions to use also depends on the purpose of the measurements and, in spectrophotometry, is determined partially by the degree of conservation of radiant flux which various conditions will afford with diffusing materials.

Symbols for reflection terms have varied from group to group and author to author. For purposes of this circular, (total) reflectance is designated by R . If it is desired to indicate the angle of incidence of the irradiating energy, a subscript may be added, as R_0 or R_{45} . If the irradiation is perfectly diffused the symbol is R_D ; if nearly diffused, the symbol is R_d .

Specular reflectance is often equivalent to (total) reflectance and the above symbols may be used. If it is desired to designate specifically that the reflectance is specular, the symbol R_s may be

used, combined if necessary with the angle of irradiation, as R_{s45} .

Directional reflectance is designated by the symbol R with subscripts attached to indicate the directions of the respective irradiating and receiving beams as $R_{0,45}$, $R_{D,60}$, $R_{45,-45}$, $R_{d,0}$, etc. In this connection it must be emphasized, however, that only the subscript D is specific. The angle subscripts refer to the directions of the axes of the respective beams. The angular spreads of these beams are also in many cases of considerable importance and should be given in any complete specification of irradiating or receiving conditions.

Reflectance, total or directional, has been defined above as a property of the object. In some cases, for example with papers and textiles, the reflectances and directional reflectances will vary with the thickness or number of layers of the material used in the measurement and may be affected by the reflectance of the backing used. If the reflecting properties characteristic of the material are desired, it is customary to measure the reflectance in such a thickness that any increase in thickness will not change the measured value and the reflectance of the backing will accordingly have no effect on the value. Reflectance measured under this condition is termed *reflectivity* or *directional reflectivity* and is designated by the symbol r . Subscripts have the same meaning as when used with the symbol R .

An important relation in this connection is that known as the Helmholtz [26] reciprocal relation. As stated by McNicholas [24], "the loss in flux density which an infinitely narrow bundle of rays of definite wavelengths and state of polarization undergoes on its path through any medium by reflection, refraction, absorption, and scattering is exactly equal to the loss in flux density suffered by a bundle of the same wavelength and polarization pursuing an exactly opposite path". By this relation it is shown that, for example,

$$R_{45,0} = R_{0,45}$$

and

$$R_{D,60} = R_{60}$$

In other words, the numerical value of directional reflectance obtained with irradiation at 45° and reception at 0° is the same as that obtained with irradiation at 0° and reception at 45° (and similarly for any other pair of angles); and the value of directional reflectance obtained with perfectly diffused irradiation and reception at 60° is the same as the value of (total) reflectance obtained with irradiation at 60° (and similarly for any other angle).

No commercial spectrophotometer has been designed to measure directly the spectral distribution of radiant energy scattered from diffusing

materials, except as the measurement of spectral directional reflectance may be so considered. Those wishing to investigate the relation between

scattering, reflectance, and opacity should consult a paper by Judd [25] and papers to which reference is made therein.

III. Spectrometry

Every spectrophotometer includes at least 5 principal constituents: (1) a source of radiant energy adequately covering the spectrum range of interest for the measurements; (2) a spectral dispersing system by means of which radiant energy of any desired frequency or wavelength (in reality, usually a more or less narrow range of frequencies or wavelengths) may be isolated and used in the desired measurements; (3) a photometric system, by means of which the desired ratio of energies may be determined at the various wavelengths; (4) a suitable detector of radiant energy which may or may not be a direct part of the photometric system; and (5) a sample holder, for transmission and reflection measurements.

Many of the details connected with these divisions of spectrophotometry are considered in connection with the various instruments discussed below. Many others, however, can best be treated in sections by themselves largely independent of the instruments (because they are pertinent to all instruments). The present sections accordingly deal with item (2), the spectral dispersing systems of spectrophotometers (section III-1) and with means for the wavelength calibration or check of such instruments (section III-2). The other items are considered in the sections on Photometry (sections IV-1 and IV-2).

1. Elements of Spectrometer

Three methods are commonly used to effect spectral dispersion and isolation in the region from 200 to 1,000 $m\mu$. The dispersion may be obtained by means of refraction through a prism, or it may be obtained through the diffraction and interference associated with the fine-line diffrac-

tion grating; or there may be no dispersing system in the ordinary sense, the spectral isolation being effected by means of optical filters. The present section deals only with the prism and grating spectrometers. The use of spectral filters is considered in section V-4.

(a) Prism Spectrometers

Glass or quartz prisms of 60° refracting angle, or equivalent prisms, have been widely, almost universally, used in prism spectrophotometers for the ultraviolet and/or visible. Two types are shown in figure 2, taken from the O. S. A. Spectrophotometry Committee report [1].

One of the simplest forms is type A. Although simple, this illustrates all of the essential elements of a spectrometer. These consist of the dispersing prism, P , with the 60° refracting angle at r , a collimator, C , carrying a lens and an entrance slit, S_C , and a telescope or second collimator, T , with lens and exit slit, S_T . The slits, S_C and S_T , and the refracting edge of the prism at r are parallel to each other and extend (usually vertically) at right angles to the plane of deviation and dispersion.

By means of the collimator slit and lens, radiant energy from a suitable source, L , is formed into a beam, the various elements of which are approximately parallel. (These elements are closely parallel in the horizontal plane because of the narrowness of the slit, but in the vertical plane the direction of the elements may deviate from the horizontal by varying amounts determined by the length of the slit and the focal length of the lens.) This "parallel beam" undergoes dispersion by passage through the prism, the angle of refraction

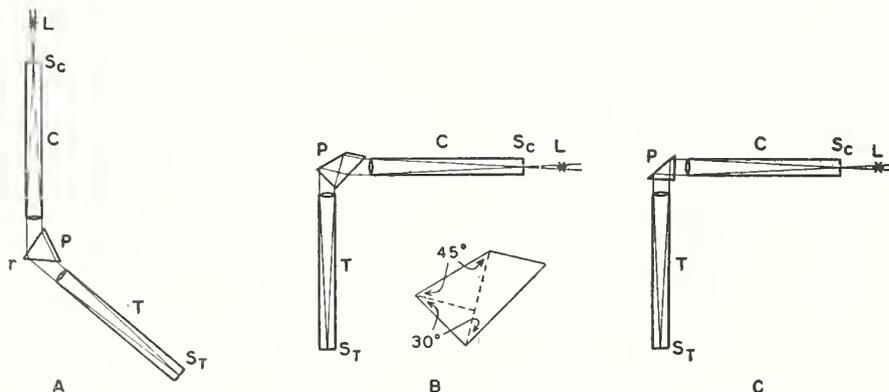


FIGURE 2. Three types of spectrometers that have been used in spectrophotometers.

A is the simple 60° prism type; B, the constant-deviation 60° prism type; C, the transmission-grating type. In C the replica grating is placed on one of the right-angle faces of P.

varying with the wavelength, and a spectrum image of S_C is formed in the plane of S_T .

By moving the telescope in the horizontal plane about a vertical axis located in the plane bisecting the angle r , the desired wavelengths may be selected at will for use in the observations. If the source, L , is one emitting radiant energy at certain wavelengths only (not a continuous spectrum), the images of S_C will appear as spectral "lines" characteristic of the particular source used. The width and length of these lines will vary with the width and length of S_C . If a source such as an incandescent-filament lamp is used (having a continuous spectrum), no lines are visible and the purity of the radiant energy isolated depends importantly on the widths of the slits, S_C and S_T .

Another type of spectrometer design is shown in B of figure 2. In this design the collimator and telescope are fixed at right angles to each other. Dispersion is effected by means of a constant-deviation prism. As shown in the enlarged diagram of P , this prism is equivalent to a 60° prism, consisting in effect of two 30° prisms separated by a right-angle totally reflecting prism. This type of spectrometer has two advantages over that shown in A . (1) Both telescope and collimator are fixed in position, and change of wavelength is effected by rotating the prism, P . (2) The rays accepted for measurement are closely at minimum deviation regardless of wavelength. Item (1) is of considerable advantage in connection with the construction of auxiliary equipment for use with the spectrometer; item (2) is less important in spectrophotometry. A disadvantage of this type of prism is the greater energy loss by absorption

within the dispersing prism caused by the increased length of path.

Modifications of these simple types of spectrometer are illustrated below in connection with certain of the spectrophotometers, such as the use of two spectrometers in series, and the use of a 30° dispersing prism with reflecting face. Still other designs of spectrometer, used principally in infrared work, are illustrated in references [6] and [21].

(b) Grating Spectrometers

The theory and use of diffraction gratings may be found in text books on optics. Concave reflecting gratings and plane transmission gratings have both been used in spectrophotometry, particularly in the form of replicas. A simple form of spectrometer of this type is shown in C of figure 2. The grating is cemented onto one of the faces of the right-angle glass prism, P . The prism also serves to redirect the beam as shown, giving a convenient form of constant-deviation spectrometer. Wavelength is varied by turning P .

(c) Transmission and Dispersion of Prism and Grating Instruments

In most applications the spectral transmittance of a spectrophotometer is of little importance so long as it transmits sufficiently to give adequate sensitivity. However, all prism material will absorb strongly at either end of its normal usable range and it is a matter of some interest to know what the transmittance of certain typical prism or grating instruments may be.

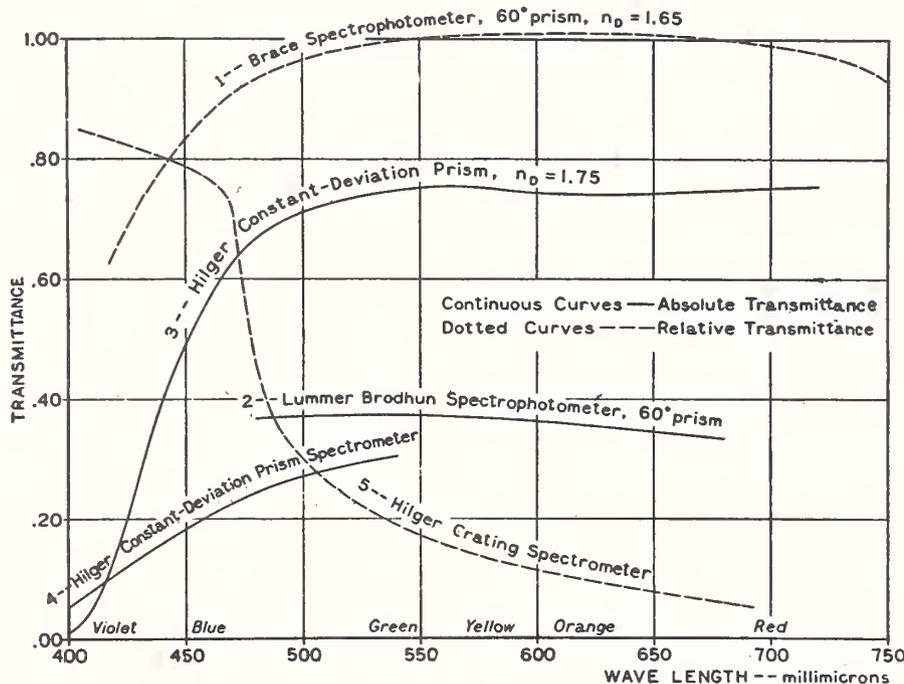


FIGURE 3. Spectral transmittance curves of various glass prisms or prism instruments, and of a grating spectrometer.

In figure 3, taken from the O. S. A. Spectrophotometry Committee report [1], are shown various transmittance curves of certain dispersing prisms and prism instruments, curves 1 to 4. Most of the absorption in the visible spectrum in a prism spectrophotometer is caused by the prism itself. The selectivity depends of course on the type of glass and the length of path in the prism. It is obvious from this illustration that any constant-deviation glass instrument cannot be used much below 400 $m\mu$.

An additional factor limiting the transmission of glass instruments in the ultraviolet is the Canada balsam used in the achromatic lenses of the collimators. A thin film of this absorbs strongly below 360 $m\mu$.

In figure 3, curve 5, is shown the relative transmittance curve of a replica-grating spectrometer, and in figure 4, curves 1 and 2, the absolute transmittance curves of a grating monochromator in the first and second order as determined by French, Rabideau, and Holt [27]. In actual use, of course, for any one setting of the grating, the two orders do not overlap as shown.

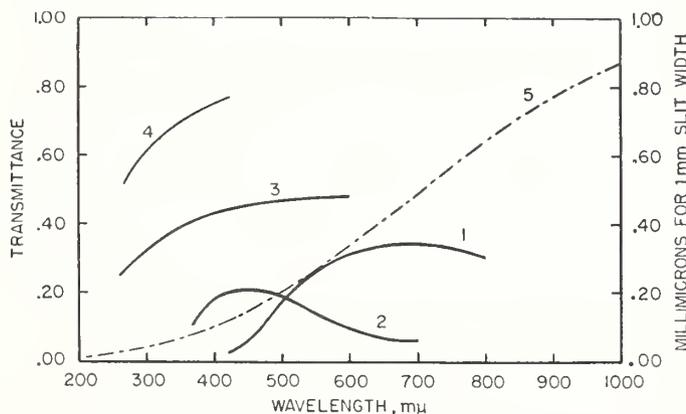


FIGURE 4. Spectral transmittance curves of certain spectrometers.

Curves 1 and 2, a grating spectrometer in the first and second orders, respectively; curve 3, a 2-prism crystalline-quartz monochromator; curve 4, a single-prism fused-quartz monochromator. Curve 5 is the dispersion curve of a quartz spectrophotometer.

Spectral transmittance curves for quartz-prism monochromators have been determined in the ultraviolet—for a crystalline-quartz 2-prism instrument by Forsythe and Barnes [28], and for a fused-quartz single-prism instrument by Kay and Barrett [29]. These are shown in figure 4, curves 3 and 4, together with the dispersion curve for a quartz-prism spectrophotometer, curve 5. The dispersion data are taken from one of the Beckman bulletins [30].

In figure 5, taken from the O. S. A. Spectrophotometry Committee report [1], are shown (1) a dispersion curve, dashed line, of a typical glass-prism spectrophotometer, (2) at top and bottom, the amounts of pure spectrum transmitted by this particular instrument for an ocular slit set

at 0.20 mm width, in terms of frequency and wavelength, and (3) wavelength and frequency calibration curves (which are considered further below). In figure 5 the dispersion is plotted as angular scale divisions per millimicron, in figure 4, as millimicrons per millimeter of slit width. For comparison it may be noted that at 525 $m\mu$ the glass-prism instrument transmits about 4 $m\mu$ of pure spectrum and the quartz-prism instrument about 5 $m\mu$ of pure spectrum, for an exit slit set at 0.20 mm.

Figures 4 and 5 illustrate how the extent of pure spectrum transmitted by an exit slit of constant mechanical width varies through the spectrum with a prism instrument. At the longer wavelengths the wavelength range thus transmitted is several times that transmitted at the shorter wavelengths. In general, therefore, the longer the wavelength, the smaller must be the mechanical width of slits to obtain the same purity of spectrum band for use in the measurements. Accordingly, if one wishes to maintain a constant spectral purity at the various wavelengths he must continually change the slit widths mechanically or use some form of cam arrangement that will do this automatically.

In a grating instrument, because of the normal dispersion, the amount of spectrum transmitted by slits of constant mechanical width is unchanging through the spectrum.

In the above, reference has been made to the amount of "pure spectrum" transmitted by the exit slit of a spectrophotometer, and this is the customary way of expressing the slit widths of such instruments. By a pure spectrum is meant one that is formed with the entrance slit set at an infinitesimal width. In order to secure sufficient spectral energy for practical use, however, the entrance slit must be set at finite width, and it has become customary to set the entrance and exit slits at the same width. With an incandescent or other continuous-spectrum source, however, this results in an extension of the wavelength range transmitted by the exit slit beyond that for a pure spectrum.

This is illustrated schematically in figure 6. In this figure, b represents the exit slit width, a the entrance slit width, both expressed in wavelength units. In A , slit a is very narrow and b is set at any nominal width. In B and C , slit b has been left unchanged and slit a widened to equal $0.5b$ and b , respectively, as shown. Relative amounts of energy transmitted by slit b are shown vertically as a function of wavelength, it being assumed that the spectral distribution of the energy arriving at slit b is constant in amount over the range $b+a$.

The important thing to note is the additional wavelength range that is transmitted by b as a is widened. This is shown by the triangle-shaped areas in B and C that are shown on either side

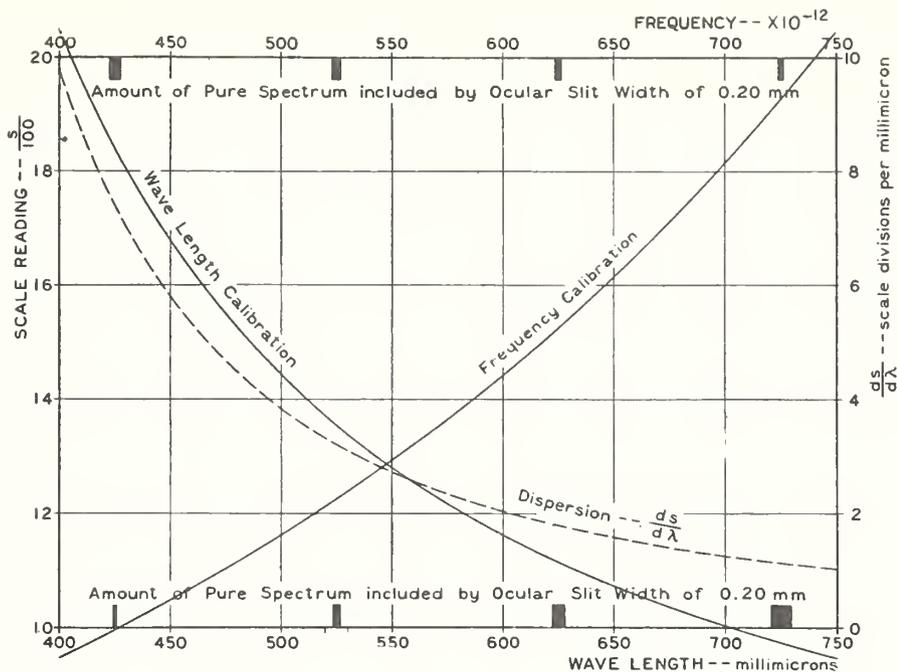


FIGURE 5. Wavelength, frequency, and dispersion characteristics of a typical glass-prism spectrophotometer not having a direct-reading wavelength scale.

beyond the extent of b . When the two slits are equal, case C , the total range of wavelengths transmitted by b is $2b$, although the fraction of energy transmitted by b at these additional wavelengths is only one-fourth of the total, and is weighted in accordance with the triangular outline. (For a prismatic spectrum the triangle of figure 6, C , is not perfectly symmetrical because of the variable dispersion, but the differences are negligible with any reasonable slit width.) Slit widths are considered further in section VI-6.

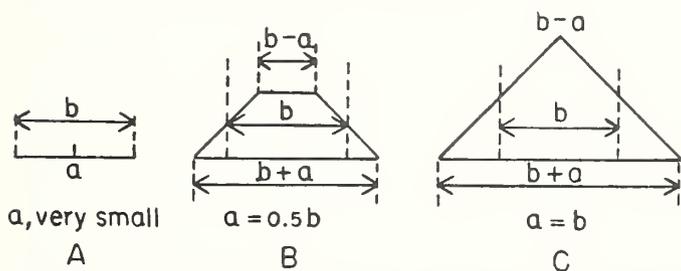


FIGURE 6. Relative transmittance of energy by a spectrophotometer at any nominal wavelength as the entrance slit width, a , is varied relative to the exit slit width, b .

Spectrophotometer slits are usually set at equal widths, illustrated in C , and the extent of spectrum transmitted is notably greater than that expressed in terms of either slit alone, as in A . It is customary to designate slit widths in terms of b alone, and designate them as nominal slit widths, and this is done in the present paper; but it is well to remember that the effective slit width is always somewhat greater than b , the nominal slit width.

2. Wavelength Calibration

The older spectrophotometers were usually designed with an arbitrary angular or linear scale for the wavelength setting. Such a scale had to be calibrated in terms of wavelengths or frequencies, somewhat as illustrated in figure 5 by the

respective continuous curves. The wavelength calibration curve can be more or less represented by various dispersion formulas, such as

$$\frac{1}{s-s_0} = A + B\lambda$$

or

$$\frac{1}{s-1} = A + B\lambda^2,$$

wherein s is the scale reading, λ is the wavelength, and A , B , and s_0 are constants to be determined. In general, however, these formulas will not give an adequate calibration except over a very short wavelength range. Their principal advantage is, perhaps, to facilitate calibration by enabling Δs , instead of s , to be plotted as a function of wavelength.

Most of the present-day spectrophotometers have a direct-reading wavelength scale; that is, the scale, instead of being divided in uniform linear or circular measure, is divided and engraved directly in millimicrons. This greatly facilitates setting the instrument at any desired wavelength. The accuracy of many of these direct-reading wavelength scales is remarkably good, when put in the best average adjustment, considering the difficulties of quantity production of such scales.

When so adjusted, it is not uncommon to find them in error by not more than $1 \text{ m}\mu$ throughout the ultraviolet and visible spectrum. However, if one wishes the uncertainties in his wavelength settings to be of the order of $0.1 \text{ m}\mu$, a careful check of these direct-reading scales is necessary.

The best procedure to use for checking the

wavelength scale of a spectrophotometer depends on whether it is a nonrecording or a recording spectrophotometer.

(a) For Nonrecording Spectrophotometers

The best procedure for checking the wavelength scale of a nonrecording spectrophotometer is by direct use of a source of radiant energy having spectral lines of suitable intensity and adequately spaced throughout the spectral range of interest. Various sources are available and can be recommended for such purpose. How many sources, or how many wavelengths, to use in such a calibration depends, of course, on the accuracy desired by the individual investigator.

In this connection it should be noted that the number of significant figures of importance in spectrophotometry (including "absorption spectroscopy") is of a different order of magnitude than that used in emission spectroscopy or in standard wavelength tables. In the visible spectrum with the usual type of spectrophotometer it seems impossible to maintain the wavelength calibration with uncertainties less than about 0.1 $m\mu$. While the uncertainty may be less in the ultraviolet with a prism instrument, there seems no purpose served in giving standard wavelengths to better than 0.01 $m\mu$ for spectrophotometric calibration.

Two of the most suitable sources for wavelength calibration of spectrophotometers are the quartz or glass mercury arc and the ordinary helium vacuum tube in glass. If the range from 220 to 1,014 $m\mu$ is of interest the quartz-Hg arc is by far the best single source. A glass-Hg arc gives the same wavelengths as the quartz arc above 310 $m\mu$.

The helium lines are especially well placed for wavelength calibration in the visible spectrum, and the strong lines at 388 and 1,083 $m\mu$ are also often very useful. Many other sources, flame or arc, are available for visual wavelength calibration [1, 31] but most of these are too unstable for accurate calibration with a photoelectric detector.

These same sources and many others are also useful for the wavelength calibration of spectrographs used in photographic spectrophotometry. Between 200 and 400 $m\mu$ the series of doublets obtained from the aluminum spark in air is very useful because they are so readily recognized.

The wavelengths characteristic of the above sources, with a few others that have proved useful, are noted in table 1. All values are consistent with those published in the MIT wavelength tables [31]. Not all of the lines for any of the sources are given but only those that are considered especially suitable for the purpose. Furthermore, not even all of those listed for any one source may be suitable for any one particular instrument. The mercury arc in quartz is a good example. All of the lines listed (and still others) could be used for wave-

length calibration of a photographic spectrophotometer over the range of sensitivity of the plate used. And all of the lines from 404.7 to 690.7 $m\mu$ can be used for visual calibration of a spectrophotometer. But not all of the lines are suitable for calibration of a photoelectric instrument, and those that prove adequate will depend on the sensitivity and slit widths characteristic of any particular instrument. One must be very careful that other lines are not included, in addition to the one on which the settings are supposedly being made, of sufficient intensity to affect the wavelength setting.

Special attention should perhaps be called to the use of a cesium arc at 852.1 and 894.3 $m\mu$ [32]. From table 1 it is apparent that suitable lines between 706.5 and 1,014.0 $m\mu$ are otherwise mostly unavailable, particularly from steady sources necessary or desirable in the calibration of photoelectric spectrophotometers. The neon discharge tube gives many lines between 750 and 1,000 $m\mu$ [31] but these have not been found satisfactory in the calibration of photoelectric spectrophotometers. In the orange and red the neon lines are useful for visual calibration and many of these can be used to calibrate photoelectric spectrophotometers [33] if the sensitivity is such that very narrow slits can be used. The relative intensities given [34] will help in case of overlapping.

The best technique to use in wavelength calibration of nonrecording spectrophotometers, given a suitable source, will vary from instrument to instrument and method to method. A few general principles can be given here, however.

In photographic spectrophotometry it usually is sufficient to photograph a known spectrum at the top and bottom of the plate, unless the source used for the absorption spectra itself carries such known reference lines. A few of these reference lines will then serve to correlate that particular plate with whatever complete calibration curve has previously been established by more extensive measurements with the various sources.

On visual and photoelectric nonrecording spectrophotometers, for highest precision, it usually is necessary to have a basic reference line to which all of the other wavelengths are compared by direct check. At the Bureau the Hg yellow lines have proved most suitable on the König-Martens visual spectrophotometer [35]. At the slit widths used the overlapping of the two lines gives a central brighter "line" taken as 578.0 $m\mu$ with a luminous background against which the slit jaws are readily seen. A luminous background, or slight illumination of the ocular slit, always facilitates calibration when an eyepiece is used. Visual calibration without an eyepiece is usually less precise unless very narrow slits are used.

Incidentally, the curvature of spectrum lines produced by a prism with straight entrance slit is readily observed in visual calibration with an eye-

TABLE 1. Sources and wavelengths suitable for the calibration of spectrophotometers

Wavelengths for				
Mercury arc in quartz (same wavelengths in glass above 300 $m\mu$)	Footnote	Aluminum spark in air	Helium discharge tube in glass	
$m\mu$		$m\mu$	$m\mu$	
205.29		216.88	318.77	
222.47		217.40	361.36	
223.41			363.42	
225.88		220.46	370.50	
230.21		221.00	381.96	
232.32		226.35	388.86	
235.25		226.91	396.47	
237.83			402.62	
239.94	1	236.71	412.08	
239.97		237.21	414.38	
		237.31		
244.69		237.34	438.79	
246.41		237.84	443.75	
248.20	2		447.15	
248.27		256.80	471.31	
248.38		257.51	492.19	
		257.54		
253.48	3		501.57	
253.65		263.16	504.77	
257.63			587.56	
260.32		265.25	667.81	
265.20	4	266.04	706.52	
				728.13
265.37		281.62	1083.0	
265.51				
269.95		308.22		
275.28		309.27		
275.97				
		358.69		
280.35	5		Neon discharge tube	
280.45			Wavelength	Relative intensity
284.78				
289.36				
292.54				
		Hydrogen, sodium, cesium arcs		
296.73	6		585.25	5.2
302.15			588.19	3.9
302.35			594.48	7.7
302.56			597.55	1.7
302.75			603.00	2.3
			607.43	7.8
312.57	7		609.62	13.0
313.15			614.31	24.6
313.18			616.36	5.9
334.15			621.73	3.9
349.28				
			626.65	11.4
365.01	8		630.48	4.3
365.48			633.44	19.9
366.29			638.30	22.8
366.33			640.22	100.0
390.64				
			650.65	38.6
398.40			653.29	7.9
400.63			659.90	11.8
404.66			667.83	23.4
407.78			671.70	14.0
435.83				
			692.95	23.0
491.60	9		702.41	2.-
546.07			703.24	44.9
576.96			705.91	-----
579.07			717.39	4.9
623.44				
			724.52	17.3
671.62			743.89	4.5
690.72			748.89	-----
1014.0			753.58	-----
1128.7			754.40	-----

1 A value of 239.95 is recommended for the unresolved pair.
 2 A value of 248.3 is recommended when the 3 lines are unresolved.
 3 The intensity of 253.48 is negligible compared to that of 253.65. The latter value should be used when the lines are unresolved.
 4 The 265.20 line is somewhat stronger than the others, and a value of 265.3 is recommended when the three lines are unresolved.
 5 These two lines are of approximately the same intensity and a value of 280.40 is recommended for the unresolved pair.
 6 The two shorter lines are considerably stronger than the other two. It is probable that a value of 302.25 should be used for the unresolved lines.
 7 A value of 313.16 is recommended for the unresolved pair.
 8 With the arc used on the Beckman spectrophotometer the ratio of intensities for 365.01: 365.48: 366.33 is 100:48:36, approximately. The intensity of the 366.29 line appears negligible relative to that of 366.33.
 9 These two lines are of approximately the same intensity and a value of 578.0 is recommended for the unresolved pair.

piece. This curvature is caused by the fact that the ray from the center of the slit is passed at less deviation than the rays from other parts of the slit. Some instruments are made with one or the other of the slits curved to remedy this defect. If both slits are straight there is an effective broadening of the slit width and, visually, a greater uncertainty of wavelength calibration unless the lengths are kept rather small.

Two techniques have been used at the Bureau in the calibration of nonrecording photoelectric spectrophotometers. On the Gibson spectrophotometer [36] the slits are always 0.1 mm or greater and the most reliable calibration is obtained by plotting galvanometer deflections at closely adjacent wavelengths. This is illustrated in figure 7. The most probable value for the wavelength reading is given by the intersection of the two straight lines as shown, the correction being given by the difference between this value and the true wavelength.

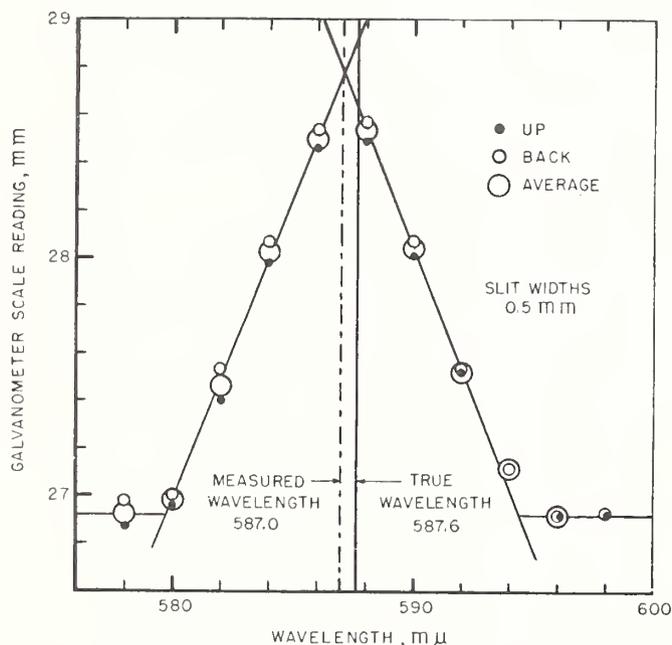


FIGURE 7. Illustrating one method of checking the wavelength calibration of a direct-reading nonrecording photoelectric spectrophotometer.

Zero deflection of the galvanometer happened to be at 26.92 mm in this illustration.

On the Beckman spectrophotometer the same method has been used [37], but at the Bureau it has seemed preferable, and is much more rapid, to calibrate with a narrow slit and note the farthest throw to the left (of the rapidly responding needle) as the wavelength dial is slowly turned. The most suitable reference line on two of the Bureau's instruments has proved to be the Hg green line [33].

(b) For Recording Spectrophotometers

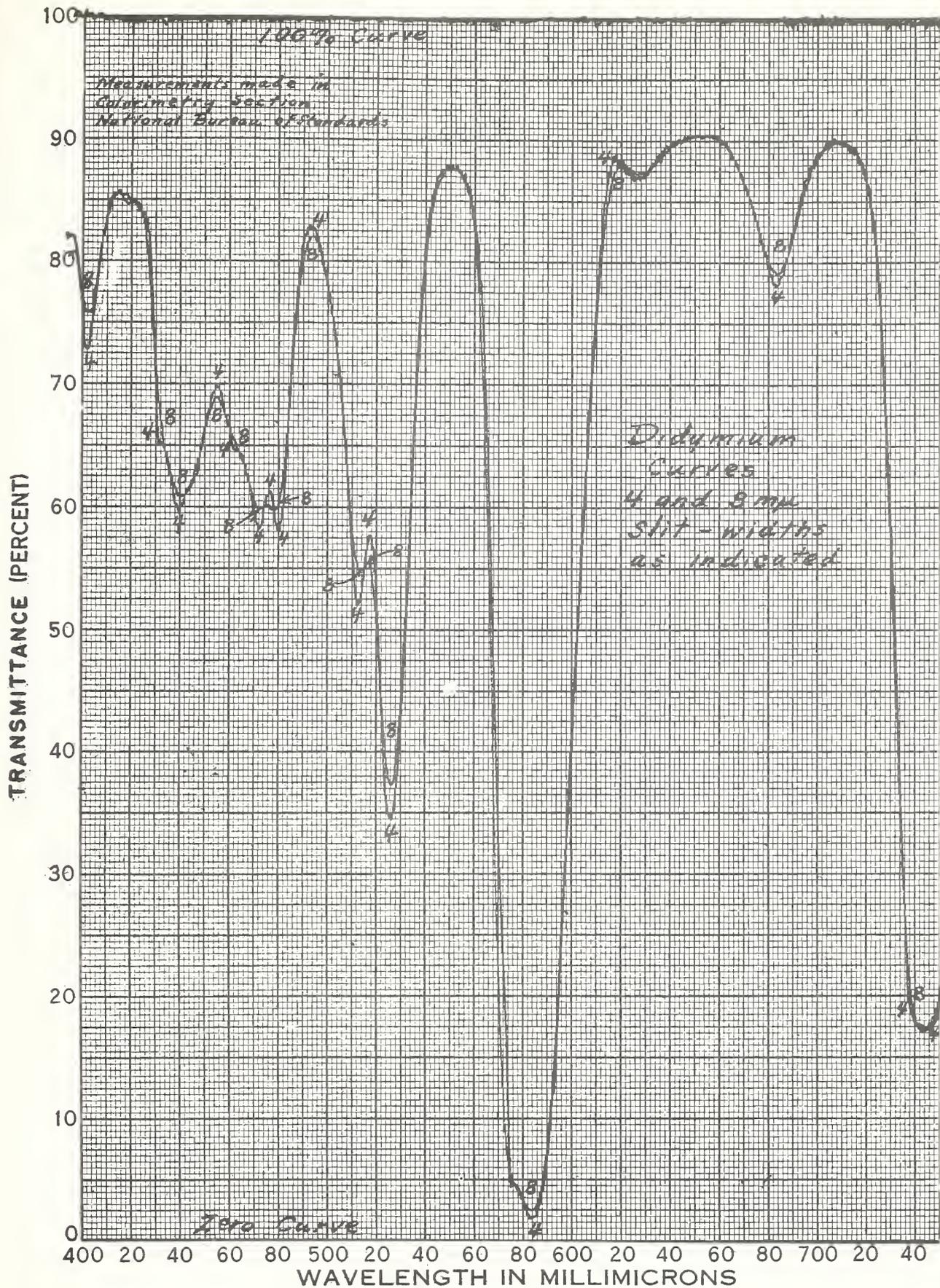
The initial wavelength calibration of a recording spectrophotometer, such as the manufacturer must carry out in connection with cutting his cams or preparing his reading scale, is not here considered, but only the check of such a calibration by the user of the instrument.

Such a user can, of course, follow the procedure prescribed above for checking the wavelength calibration of nonrecording spectrophotometers. However, there are two important reasons for following a different procedure for recording spectrophotometers. For such instruments it is desirable to have a calibration that is made with the instrument operating. It is further desirable in many kinds of work to have this calibration appear on the graph sheet so that worries connected with positioning of the sheet, expansion or contraction of the paper with humidity or temperature, or instrumental variations can be eliminated.

Wavelength calibrations of this kind can be made if a material is available having a number of strong and narrow absorption or transmission bands suitably spaced over the spectral range of interest. Two materials have been used or suggested for this purpose: (1) Didymium glasses have been used for many years at the National Bureau of Standards [38] and many have been calibrated and issued to others for similar use, (2) quartz-Polaroid combinations have been proposed [39] and may prove useful for such work.

The use of a didymium glass in this manner would not in general be accurate unless it is calibrated at closely the same slit widths at which it is to be used. Most of the absorption bands that are usable for the purpose are multiple bands and the wavelengths of maximum absorption often depend on the slit widths. This is illustrated in figure 8 where the curves recorded on a General Electric spectrophotometer [40, 41, 42] at the National Bureau of Standards [38] are shown for slits equivalent in width to 4 and 8 $m\mu$ of spectrum approximately.

The NBS didymium glass standards were carefully calibrated by point-by-point measurements on the König-Martens visual and Gibson photoelectric spectrophotometers with slit widths approximating the 4, 8, 10 and 20 millimicron slits used on the NBS General Electric spectrophotometers. Some of these values have been published [43]. Although these initial calibrations were tedious and time-consuming, the time and worry eliminated by the use of such standards are enormous. Once calibrated it has been very simple to derive corresponding values for glasses submitted for calibration by recording their transmittance curves on a sheet along with the curves for the NBS calibrated standards.



(Legend on page 15)

The most suitable didymium glass for the purpose, considering type of curve and availability, is a Corning 5120 glass of 3.0 mm thickness. While it is not known how much the wavelengths of maximum absorption of this 5120 glass might vary from melt to melt, glasses from at least three melts have been measured and there has never been any certain variation among the samples tested. For much work it is probably safe to use the values given in table 2.

While the use of a didymium glass for checking the wavelength calibration of recording spectrophotometers is highly recommended, as noted above, there are two other uses of the didymium glass which are not recommended. First, the didymium glass is not well suited for checking the photometric scale of any spectrophotometer, recording or nonrecording. Transmittances at the peaks of the absorption bands are too dependent on slit widths, and transmittances on the steep parts of the curve are too dependent on slight wavelength errors, both as illustrated by figure 8. Second, the use of a didymium glass to check the wavelength calibration of a nonrecording spectro-

TABLE 2. Wavelengths of maximum absorption for Corning 5120 glasses at 3.0 mm as obtained at the National Bureau of Standards for the slit widths indicated

Wavelength of maximum absorption	Over-all uncertainty	Approximate spectrum interval transmitted by slits
$m\mu$	$m\mu$	$m\mu$
441.0	± 1	10
475.5	1	10
528.7	1	10
585.0	1	10
684.8	1	10
743.5	1	10
745	2	20
808	2	20
883	2	20
1,067	2	20

photometer is considered much inferior from the standpoints of time, convenience, and reliability to the direct use of line sources as described above. The National Bureau of Standards has consistently refused to accept didymium glasses for calibration for either of these two purposes.

IV. Photometry

Another of the principal parts of a spectrophotometer is the photometric system, whereby the desired ratios of energy or flux may be determined at the various wavelengths isolated by the spectrometric system. Important parts of the photometric system are the source and detector of radiant energy, and these will be considered before discussing the photometric devices themselves.

1. Spectral Characteristics of Sources and Detectors

Various sources of radiant energy have proved adequate for spectrophotometry in the range from 200 to 1,000 $m\mu$. From 350 or 400 $m\mu$ to beyond 1,000 $m\mu$ the incandescent filament lamp is by far the most suitable source for most purposes, either as a standard of spectral energy distribution when measuring the distribution of other sources or as the source for spectral transmission and reflection measurements. Its advantages relate to the facts that (1) its radiant intensity under normal operating conditions is adequate for the purpose, (2) its intensity can be readily kept constant, to a high degree if necessary, (3) having a continuous spectrum it enables measurements to be made at any wavelength over its useful range.

In figure 9 are shown the relative spectral energy distributions of an incandescent source at color temperatures of 2,600 and 3,000° K. Most incan-

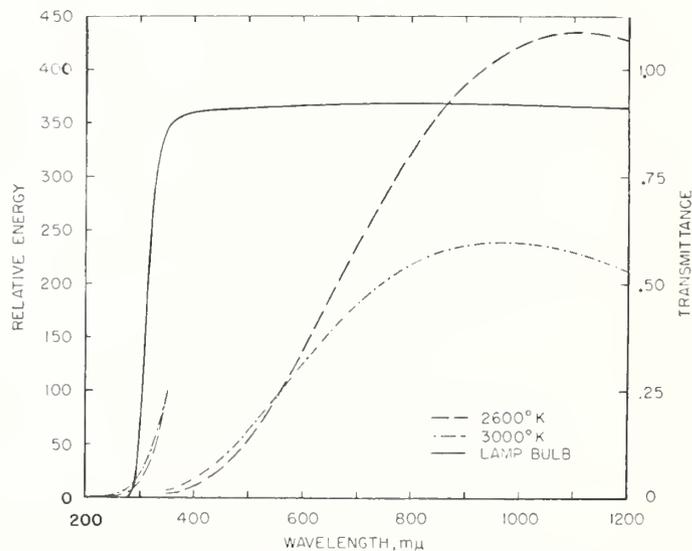


FIGURE 9. Spectral energy distribution curves of black-body at 2,600° and 3,000° K, adjusted to 100 at 560 and 350 $m\mu$.

These curves are closely like those from the incandescent lamp filaments used on spectrophotometers, except that in the ultraviolet below 350 $m\mu$ they are notably reduced by the transmittance of an ordinary glass bulb. The spectral transmittance curve (continuous line) of such a bulb is shown.

FIGURE 8. Spectral transmittance curves of a didymium glass recorded on a General Electric photoelectric spectrophotometer at the National Bureau of Standards with nominal slit widths equivalent to 4 and 8 $m\mu$ of spectrum, as shown.

For the two slit widths, note (1) shifts of the wavelengths of maximum absorption for the 480 and 513 $m\mu$ bands and (2) differences in recorded transmittances at all regions of sharp curvature.

descent lamps used on spectrophotometers operate within or close to this range. The data plotted are those from Planck's equation for the theoretical black body at these temperatures [44, 45, 46], but it is known that they closely represent the relative distributions of lamps at these color temperatures [47, 48]. Below 350 $m\mu$, however, absorption of the ordinary glass bulb rapidly reduces the energy to zero near 300 $m\mu$. This is apparent from the spectral transmittance curve shown in figure 9 for a piece of glass cut from a 200-watt incandescent lamp bulb.

The quartz mercury lamp is useful not merely for wavelength calibration of spectrophotometers; at certain wavelengths the radiant intensity is sufficiently great that it may also be used as the source for photometric measurements. The advantages of such use are that the results cannot be affected by wavelength or slit-width errors, as is possible with an incandescent lamp as source.

The Hg lines that have been found useful for such purpose at the National Bureau of Standards are (1) visually, on the König-Martens spectrophotometer, those at 404.7, 435.8, 546.1, and 578.0 (576.9+579.0) $m\mu$, and (2) photoelectrically, on the Beckman spectrophotometer, those at 365.0, 334.2, 302.2 and 253.6 $m\mu$ in addition to those listed above in the visible spectrum. The relative intensities among various of these lines vary considerably from one type of lamp to another. One such set of intensities is shown in figure 10. In this figure the intensities below 400 $m\mu$ are taken without change from table XVII of reference [49]. Above 400 $m\mu$, however, they have been adjusted to fit the individual lines. The data of figure 10

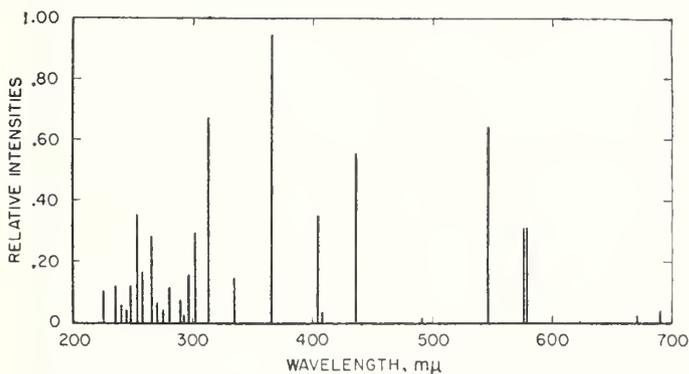


FIGURE 10. *Relative intensities of the various spectral "lines" in a quartz mercury arc.*

Such a source may be used for the wavelength calibration of spectrophotometers, and the stronger lines may be used also for photometric measurements. The relative intensities differ with the operating conditions and types of arcs. Those shown are for a "hot" quartz arc. For a low-pressure "cold-cathode" arc the major portion of the intensity is concentrated in the 253.6 $m\mu$ line. Wavelengths are given in table 1. The intensities shown are based on data by Luckiesh [49] (copyright by D. Van Nostrand Company, Inc.) and are reproduced by permission.

are for a "hot" quartz lamp. The relative intensities for a low-pressure, "cold-cathode" mercury lamp are much different, the intensity of the line at 253.6 $m\mu$ exceeding all the others.

The most commonly used source of radiant energy for spectrophotometry in the ultraviolet

spectral region is probably now the hydrogen arc. Under certain operating conditions of pressure and current a nearly continuous spectrum can be obtained, of adequate intensity for use over the range from 200 to above 400 $m\mu$. Hydrogen arcs are suitable for use in both photoelectric and photographic spectrophotometers.

Other sources widely used in photographic spectrophotometry, but too unsteady for the usual type of photoelectric spectrophotometer, are (1) the under-water high-voltage spark discharge and (2) various arcs or sparks where the electrodes have been especially selected to yield a great number of closely spaced spectral lines. The under-water spark spectrum has long been used at the National Bureau of Standards and its appearance is shown later in figure 16. The spectrum is perfectly continuous without superposed lines and covers the range from 500 to 220 $m\mu$ and perhaps beyond. Examples of arc or spark spectra in air, as used widely by various investigators in ultraviolet absorption spectra, may be found in various publications [3, 11].

The detectors commonly used with spectrophotometric equipment over some part of the range from 200 to 1,000 $m\mu$ are (1) the eye, (2) the photographic plate, (3) the photoelectric cell (the vacuum or gas-filled electron-emission type, or the barrier-layer type) and (4) the thermopile or other form of radiometric device. Only the first three types of detectors will be considered here, since the thermopile and other radiometers are mostly used in infrared work.

The relative sensitivity (luminosity) curve of the human eye—that is, the photometric response of the eye to an equal-energy spectrum—has been determined by various investigators. It varies considerably from one individual to another and is widely different at low luminances (scotopic vision) [50] and at high luminances (photopic vision) [51]. In figure 11, curves A and B, are

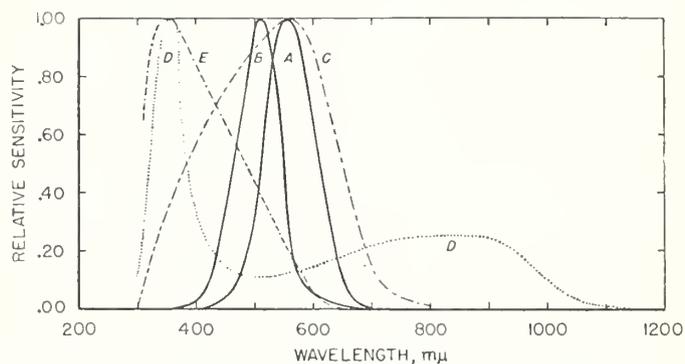


FIGURE 11. *Relative sensitivities of various detectors used in spectrophotometry.*

A and B are the photopic (light adapted) and scotopic (dark adapted) average luminosity curves of the human eye. C is more or less representative of barrier-layer photocells, D and E of certain "red sensitive" and "blue sensitive" electron-emission phototubes. The sensitivity of E continues adequate to at least 200 $m\mu$. Differing greatly from these curves are the response curves of the respective detectors at the exit slits of spectrophotometers resulting from energy distribution of source, prism dispersion (if not compensated by varying slits), and instrumental transmittance.

shown the average photopic and scotopic luminosity curves plotted to unit maxima. The shift of the curve towards the shorter wavelengths with decreasing brightness is known as the Purkinje effect. In most visual spectrophotometry the eye of the observer is probably in varying states of adaptation between the two extremes, depending on the part of the spectrum in which measurements are being made and the room luminance within his field of view.

The spectral responses of various types of photocells, including various types of sensitive surfaces, vary enormously [6, 52]. Even for photocells of a given type and surface the responses may vary considerably, somewhat similarly to the luminosity curves of different individuals. In figure 11 are shown spectral sensitivity curves of certain types of photoelectric cells that have been commonly used in spectrophotometers. These are plotted relative to unit maxima and give no indication of the absolute sensitivity of the cell. They indicate relative response to an equal-energy spectrum.

Curve C is more or less typical of the barrier-layer cells used in the Cenco-Sheard and Coleman spectrophotometers. Curve D represents more or less the so-called "red sensitive" electron-emission type of phototube used in the General Electric recording spectrophotometer, the Coleman universal spectrophotometer, and the Beckman quartz spectrophotometer. Curve E represents the "blue sensitive" phototube also used in the Beckman instrument.

It should be remembered that curves C, D, and E of figure 11 do not represent the relative responses of these photoelectric cells at the exit slits of actual instruments. The response will be considerably affected by the spectral energy distribution of the source used, by the spectral dispersion (for prism instruments) unless compensated by varying slit widths, and by the spectral transmittance of the spectrophotometer.

Similarly the luminosity curves, A and B, of figure 11 do not represent the relative field luminances in a visual spectrophotometer, nor does the plate sensitivity curve of a photographic plate represent the relative spectral densities obtained in any photographic spectrophotometer. In both cases the response curves (luminance or density) depend on the instrumental dispersion and transmission and the energy distribution of the source as well as on the spectral sensitivity of the detector.

The photographic plate has rarely been used for spectrophotometry above $500\text{ m}\mu$, this being the effective limit for unsensitized emulsions. Absorption of the gelatine prevents adequate sensitivity below about $220\text{ m}\mu$. If it is desired to use photographic methods below this wavelength, special plates must be used. These may use fluorescent dyes in the emulsion or may use non-

gelatine suspensions [4, 6, 11]. A recent publication [54, fig. 34] gives relative spectral sensitivity curves of four typical photographic materials.

2. Photometric Principles and Devices

The design of the photometric part of a spectrophotometer depends directly upon the detector or receptor used. It may be recalled that spectrophotometry consists always of measuring the ratio of two radiant energies (or radiant fluxes) at a specified frequency or wavelength, this measurement being repeated at other frequencies or wavelengths as often as desired over the spectral range of interest. With a photoelectric cell this ratio may be determined by measuring the ratio of currents evoked by the two radiant fluxes, provided a closely linear relation can be established between the irradiance on the photocell and the current evoked in the cell and indicated in the galvanometer or other measuring device. In such cases the photometric system is mostly electrical.

A similar direct measurement of the spectrophotometric ratio by the photographic plate is difficult and by the eye impossible. In nearly all photographic spectrophotometers and in all visual spectrophotometers, therefore, this ratio is measured by some auxiliary optical system, often incorporated in the instrument, the setting being indicated by equality of luminance or of photographic density in a 2-part photometric field.

(a) Visual Photometry

While the eye can estimate only roughly the absolute magnitudes of photometric quantities, it can determine with considerable precision when the two properly juxtaposed parts of a photometric field are equally bright—provided, of course, that the two parts of the field are of the same chromaticity, as is usually the case in visual spectrophotometry.

In a visual spectrophotometer, therefore, the photometric part of the instrument includes (1) means for securing a uniformly bright 2-part photometric field and (2) means for varying the luminance of one or both parts of this field in a continuous, easily adjustable, and known manner, so that when the eye indicates equality of match the desired spectrophotometric ratio can be determined.

Various types of photometric field have been used; these are shown in figure 12, taken from the O. S. A. Spectrophotometry Committee report [1]. In type I of this figure the two parts of the field consist of two juxtaposed spectra formed at the exit slit of the instrument and viewed by a magnifying eyepiece; the other fields illustrated in figure 12, type II, are formed or projected in or between the telescope and collimator lenses and

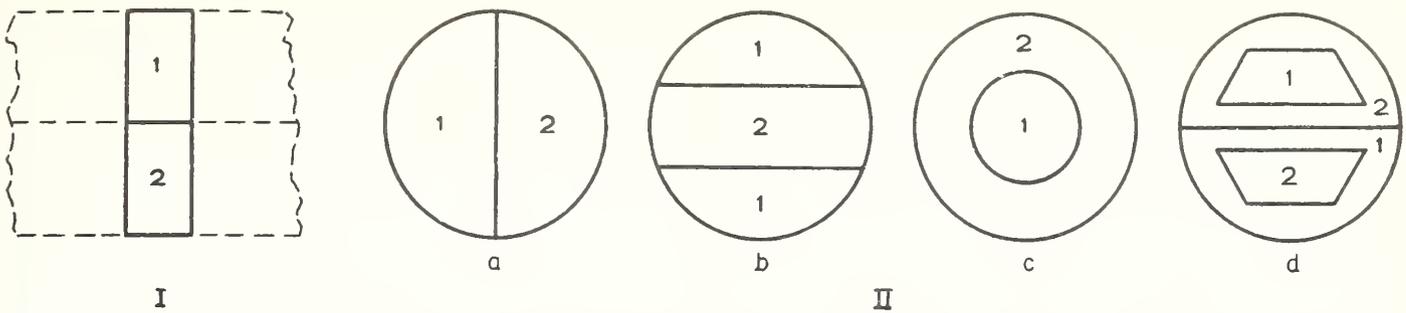


FIGURE 12. Various types of photometric fields used on visual spectrophotometers. Type I, juxtaposed spectra; type II, monochromatic field.

The eye can equate two properly juxtaposed brightnesses with considerable precision.

are viewed by the eye placed (usually) close to the exit slit. The trapezoids of type II-d are usually about 8 percent darker than their surroundings (being produced by reflection from clear glass plates), and equality of contrast rather than equality of brightness is the criterion for photometric match.

Various methods have been used to vary the luminance in one or both parts of the field. (1) When two collimators are used the slit width of one may be varied and the luminance is taken as proportional to the width. The method conserves light but its accuracy is limited. (2) Rapidly rotating sectors have been used, having variable aperture while rotating. (3) Polarization methods have often been used, probably more than other means. An instrument using this method of luminance variation is illustrated in section V-1.

Still other methods are possible and have occasionally been employed. However, the fundamental inverse-square relation has been little used in visual spectrophotometry because of the resulting low luminances. For further information regarding various phases of visual photometry or spectrophotometry reference may be made to section V-1 below or to some of the publications already listed [1, 5, 35].

When properly used, a visual spectrophotometer can give results of high accuracy. Under the best conditions the uncertainty of the mean of several settings is some fraction of 1 percent of the value. However, the necessity of making repeated settings to obtain adequate reliability makes the method tedious and time-consuming. As an independent method in measuring standards of spectral transmittance and for certain other special problems, the König-Martens visual spectrophotometer at the National Bureau of Standards is still of great utility, but for most transmission measurements and for practically all reflection measurements the instrument and method have been superseded by more rapid photoelectric spectrophotometers.

(b) Photographic Photometry

When a photographic plate is exposed to radiant energy and developed, the well known blackening

occurs, and the relation between the photographic (optical) density of the developed plate and the exposure to which the plate is subjected is more or less as shown in figure 13 [6]. Density and exposure are terms widely used in photographic photometry, the former being defined usually as the negative logarithm of the transmittancy (ratio of luminous flux transmitted by the silver deposit to that incident upon it) of the developed photographic material, the latter representing the logarithm of the product of illuminance (light units) and the time of exposure. Photographic or optical density is thus analogous to absorbancy as defined in section II-2, above.

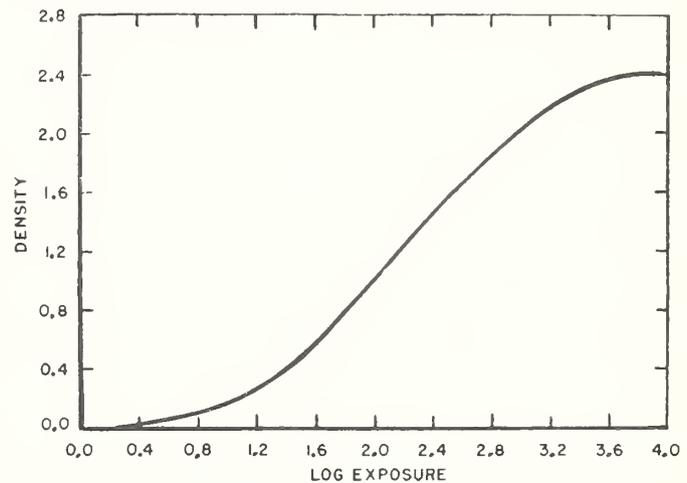


FIGURE 13. Typical density-exposure curve of photographic emulsions.

Photographic spectrophotometers are usually designed in such a way as not to be dependent on this and certain other characteristics of photographic materials. This curve is derived from figure 164 of reference [6] (copyright by McGraw-Hill Book Company, Inc.) and is reproduced by permission.

The relation shown in figure 13 is the basis for much photographic photometry. However, the many variables involved in the preparation, exposure, and development of photographic emulsions render accurate photographic spectrophotometry by this relation very difficult. Accordingly in such work it has become almost universal to ignore the characteristic relation shown in figure 13, and instead to design the photometric system such that two juxtaposed spectra are simultaneously photographed for each exposure and the

only photometric observation required is the determination of points of density match along the juxtaposed spectra. Thus all worries regarding type of emulsion, factors of development, and uniformity of the plate, are eliminated.

In one of the two juxtaposed spectra the radiant energy has passed through the sample (for example, the solution), and in the other spectrum it has passed through the comparison sample (for example, the solvent) and been reduced in known amount by the photometric system. In general, a series of juxtaposed spectra is taken on each plate, the reduction of the comparison beam being made in known steps in the respective exposures.

Measurement of the developed plate then consists in determining the precise frequencies or wavelengths at which the match-points occur, these being the frequencies or wavelengths at which the transmittancy of the solution has the predetermined value at which the photometer was set for that particular pair of spectra. The match points may be determined visually or photoelectrically. Further details are given in section V-2.

Perhaps the principal advantage of the photographic method of spectrophotometry is the permanent record obtained, and for a long time it was the only method commercially available for the ultraviolet.

(c) Photoelectric Photometry

As already noted, a photoelectric cell, with appropriate meter, contains within itself the possibility of direct spectrophotometric measurement, something wholly untrue of the eye and practically unfeasible with the photographic plate. If there be adequate sensitivity, the question of primary importance in direct photoelectric photometry is whether there is a linear relation between the irradiance on the photoelectric cell and the resulting indication on the meter or other device by which the photoelectric current is indicated. Lacking such linearity there must be adequate calibration or resulting error.

For a long time such linearity for the electron-emission type of cell, now usually called a phototube, appeared to be the exception rather than the rule [52]. For many modern phototubes, however, particularly the vacuum type, and at irradiances of the order of those used in spectrophotometry, the linear relation is closely if not exactly followed. Some of the photoelectric spectrophotometers now on the market are based on the linearity of the irradiance-current relation. In another type of spectrophotometer this

linear relation is of no importance; the phototube is merely a balancing indicator and the photometric system is based on polarization principles similar to those in certain visual spectrophotometers.

The barrier-layer photocell, not requiring external voltage, has also been used in spectrophotometry. The question of linearity is likewise of importance with this photocell, and here as always the reliability of the photometric scale should be checked in one way or another if results of the highest accuracy are desired.

(d) Sample Holders

Four of the five principal constituents of every spectrophotometer—namely the source, the spectrometer, the photometer, and the detector—have been considered above. There remains only the sample container and the holder, carrier, or other device by which the sample may be moved in or out of the beam or from one beam to another, or interchanged with the standard or comparison sample, or merely held in place. Space is lacking to illustrate the numerous devices that have been designed for this purpose, these designs varying in turn with the type of measurement to be made, whether it be the transmittance of a glass, the transmittancy of a solution, or the directional reflectance of a textile, powder, or other type of material.

Glass or quartz cells of varying types for holding solutions and solvents are described in detail in many of the publications listed [3, 4, 11, 35, 53]. Several important considerations relate to the use of such cells. The end plates used in the solution and solvent cells should be identical in every respect; the internal dimensions of the cells should be accurately known; and all parts of the cells with which the solution and solvent are in contact should be capable of being thoroughly cleaned.

Another important factor in both transmission and reflection measurements relates to the mechanical stability of the sample holder or carrier, and the precise reproducibility of positioning when the sample is replaced by the standard, or when sample and standard are interchanged. Closely related to this in reflection measurements is the problem of the positioning of a sample such as a textile which is apt to "bulge" so that it is not in the same plane as the standard or comparison sample. Putting both samples under a cover glass may remedy this, but may introduce multiple reflection errors [55].

V. Typical Instruments and Methods

1. Visual

It is believed that the only visual spectrophotometer commercially available at present in this country is that manufactured and sold by the Gaertner Scientific Corporation [56]. Since this is somewhat similar to the visual spectrophotometers sold by Adam Hilger, Ltd. [3] and formerly sold by the Bausch and Lomb Optical Company [57], and also is similar in principle to the König-Martens spectrophotometer [35] in use at the National Bureau of Standards, it has been chosen for illustration. Where the other instruments have special points of difference, these will be noted.

The photometric part of the Gaertner spectrophotometer is outlined in figure 14, which is based on advertising circulars of the company. In this instrument, as shown, and in the Hilger and Bausch and Lomb instruments, the photometer is a separate unit from the spectrometer, whereas in the König-Martens spectrophotometer the two parts are incorporated into one instrument.

The source of radiant energy shown in the illustration is one designed for both transmission and reflection measurements. (A source designed solely for transmission measurements can be obtained if desired.) It is a white-lined sphere, 10 inches in diameter, in which 16 incandescent 6-volt lamps are mounted around the vertical equator.

For reflection measurements the two samples being compared, *A* and *B*, are held against openings in the sphere as shown in the upper part of figure 14.

Specially designed diffusing screens near each lamp shield the sample and standard from direct irradiation without casting shadows. Multiple reflections from the walls of the sphere then combine to produce diffuse irradiation. This irradiation fails to be perfectly diffuse principally because of the sphere opening through which the reflected energy is taken to the instrument. Collimating lenses are placed at *L*. The type of directional reflectance measured is designated as $R_{d,0}$ (section II-3).

For transmittancy measurements a single diffusing plate, *C*, is used, as shown in the lower part of figure 14. The radiant energy reflected from this plate is split into two beams by the pair of rhombs, *R*, and led through the solution and solvent cells, *T* and *T'*, into the photometer.

For both reflection and transmission measurements the two beams are passed by suitable optical means through the photometer and the spectrometer, and the eye placed at the ocular slit of the spectrometer sees a photometric field of type IIa, figure 12, with dividing line horizontal.

The photometer *P* is the polarization type known as the Martens photometer [58], although

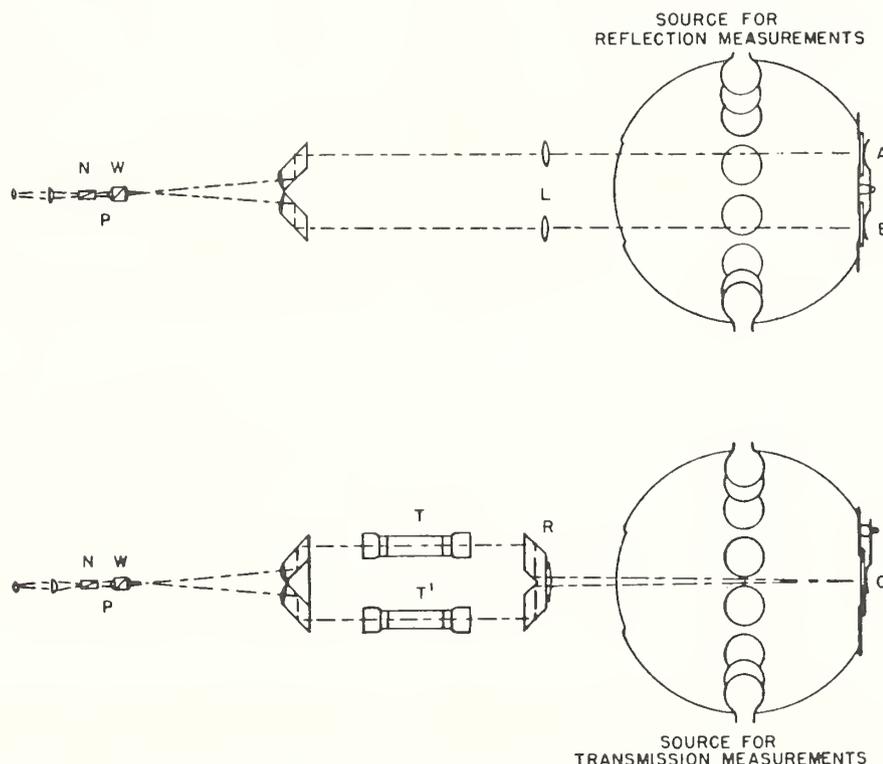


FIGURE 14. Optics of the Gaertner visual spectrophotometer.

The spectrometer is not shown. With this equipment the samples are irradiated before the radiant energy is dispersed in the spectrometer.

the internal optics of the German and American designs are not identical. The two beams entering the instrument are led to form the two halves of the photometric field, the dividing line of which is the edge of a biprism. The light in the two halves of the field is polarized in mutually perpendicular planes by means of a wollaston prism, W , the planes of polarization being respectively parallel to and at right angles to the dividing line of the field (the edge of the biprism). A nicol prism, N , is mounted at the left of the wollaston prism, and by turning this nicol the two halves of the field may be precisely matched in brightness.

If the transmittancy of a solution is being measured, let α_0 be the angle of match with both beams blank (or containing identical solvent cells), and α_1 be the angle of match when the respective cells containing solution and solvent are placed in the beams. Then

$$T_s = \frac{\tan^2 \alpha_1}{\tan^2 \alpha_0}$$

The derivation of this formula and many other things of interest in this connection may be found in the papers by Martens [58] and McNicholas [35]. If the solution and solvent are interchanged, a similar relation is obtained. That is,

$$T_s = \frac{\cot^2 \alpha_2}{\cot^2 \alpha_0}$$

By combining these two relations it follows that

$$T_s^2 = \frac{\tan^2 \alpha_1 \cot^2 \alpha_2}{\tan^2 \alpha_0 \cot^2 \alpha_0}$$

or
$$T_s = \tan \alpha_1 \cot \alpha_2$$

The \tan^2 relation is useful if one wishes to put direct-reading scales on the photometer and this has been done by both Gaertner and Bausch and Lomb. In this case it is essential (unless correction be made) that α_0 be exactly 45° (so that $\tan^2 \alpha_0 = 1.000$).

It may easily be overlooked in this connection that a substitution or interchange method should always be used in spectrophotometry. In a substitution method the test sample and its reference standard should be compared in the same beam by way of a third or comparison sample in the second beam. This is true in both transmission and reflection measurements. It is often convenient to have the comparison sample identical with the reference standard but except in direct-reading instruments this is not essential.

On the König-Martens spectrophotometer at the National Bureau of Standards the interchange method is used and T_s is computed from the $\tan \times \cot$ relation. This is equivalent to a substitution

method, but the value of α_0 is immaterial and no measurement is made of it. A reference standard is necessary but no comparison sample.

On the Bausch and Lomb spectrophotometer means were provided for passing the beam through solution and solvent cells in which the thickness of absorbing layer could readily be varied without removing the cells.

On the Hilger Industrial and Research Spectrophotometer a similar $\tan^4 \alpha$ relation is used [59, 3], resulting in a more open scale at high absorbancies.

Auxiliary equipment designed and constructed at the National Bureau of Standards [35] for use with the König-Martens spectrophotometer includes the following features:

1. Hemispherical irradiation of the sample and standard for reflection measurements, by means of which the irradiation is in effect almost perfectly diffused. The type of directional reflectance measured is thus in effect $R_{D,0}$, which is equal to R_0 by the reciprocal relation.

2. Installation of mercury and helium sources which can be readily interchanged with the incandescent source and are valuable for use in transmission measurements as well as in wavelength calibration.

3. The installation of accurately calibrated rotating sectors by means of which (1) the photometric scale can be readily checked over the range from $T=0.80$ to $T=0.00883$ and (2) the transmission scale can be greatly extended at low transmissions. This extension is effected by placing the sector ($T=0.09945$ or 0.00883) in the blank beam and measuring the transmittance of the sample relative to that of the sector.

Many other types of visual spectrophotometers have been designed and used during the past 100 years. These are described in other publications [1, 3, 5, 6, 11] or in papers to which these publications refer.

2. Photographic

Major contributions to the science of photographic spectrophotometry have been made by Adam Hilger, Ltd., of London. Their rotating sector photometer introduced about 1910 for use with suitable source and spectrograph was apparently the first commercially available equipment for reliable ultraviolet spectrophotometry, and such equipment was shortly thereafter in use in the United States [60].

The National Bureau of Standards equipment is described in B. S. Sci. Paper 440 [53], and the optics of the apparatus are outlined in figure 15. Radiant energy from the source, E , passes through cells T and T' , containing solution and solvent placed in a constant-temperature inclosure. The respective beams are deviated and focused by the wedge lenses at L , then further redirected by the biprism, B , in front of the spectrograph slit;

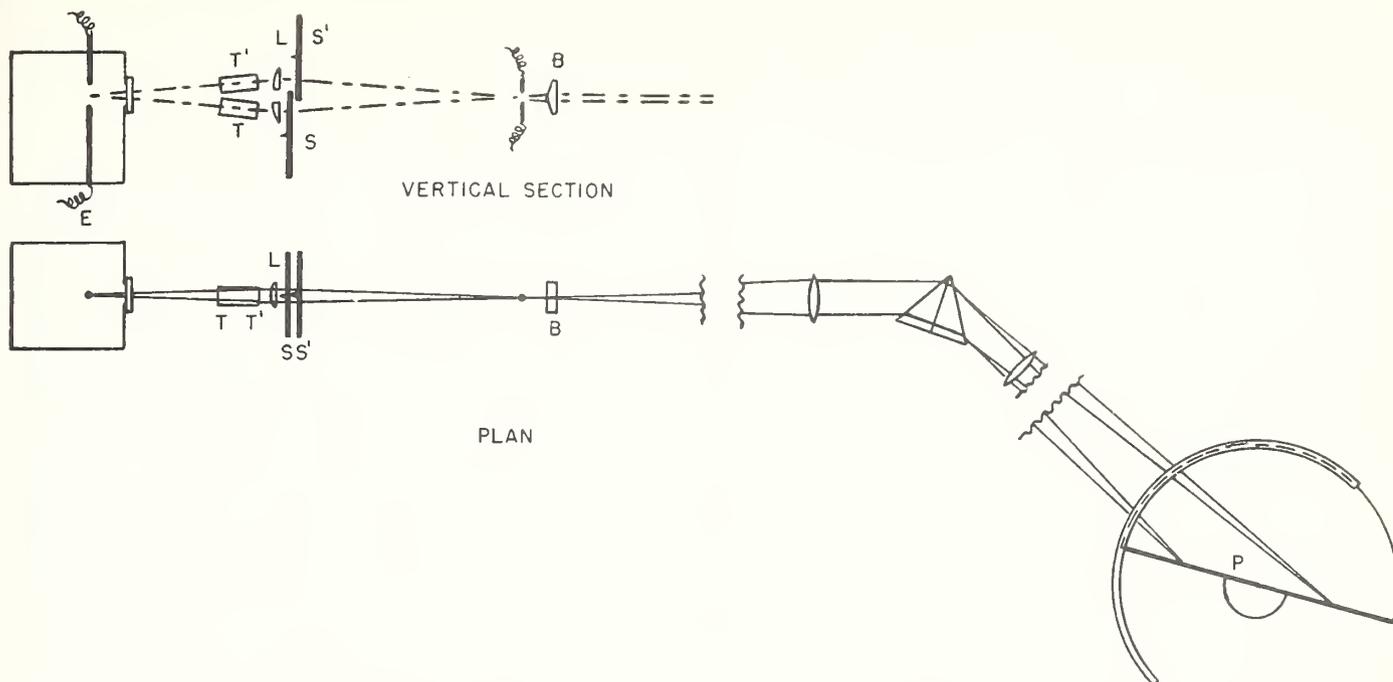


FIGURE 15. Optics of the Hilger sector-photometer, photographic spectrophotometer as installed at the National Bureau of Standards.

With this equipment the samples are irradiated before the radiant energy is dispersed in the spectrograph.

finally the two beams are made to form a pair of juxtaposed spectra on the photographic plate at *P*, the dividing line between the two spectra being the spectral image of the horizontal edge of the biprism, *B*. All optical parts are of crystalline quartz. Rotating sectors are placed at *S* and *S'*. The sector in the solution beam is kept at maximum opening, designated as 1.000 on the transmittancy scale; the sector in the solvent beam may be set at any transmittancy value between 1.000 and 0.000, this value being different for each exposure.

There results, when the plate is developed, a series of exposures as shown in figure 16. By determining for each exposure the frequencies or wavelengths at which the two juxtaposed spectra are of equal density, the transmittancy or absorbancy curve may be derived. On the plate at top and bottom may be seen the aluminum spark spectra used for wavelength calibration (the lines at the shorter wavelengths do not show well in figure 16) and the comparison spectra taken with both beams blank and both sectors set at 1.000. On this plate it will also be noted that

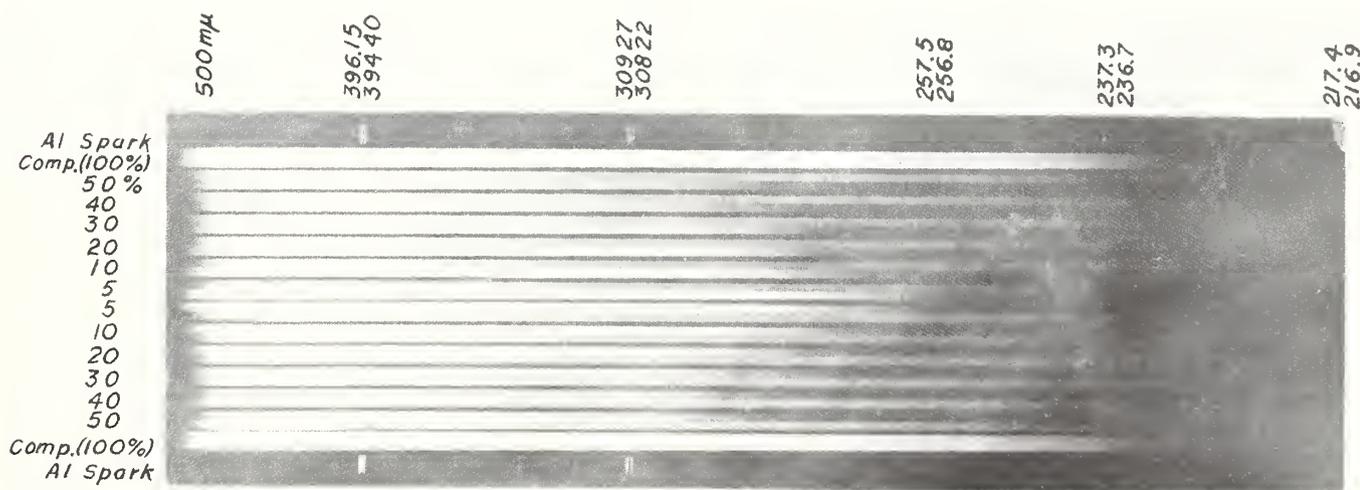


FIGURE 16. Typical set of exposures made with equipment shown in figure 15.

Note continuous spectrum extending from 500 to 220 $m\mu$ produced by high-voltage Tesla-coil spark in distilled water. For details see text.

the spectra in the lower half are a reverse repetition of the spectra taken in the upper half. On the National Bureau of Standards instrument both sectors were made adjustable and scales attached. To eliminate the effect of possible slight lack of match in the two halves of the comparison or 100 percent spectra, the solution and solvent cells are interchanged in the two beams at the middle of the plate and the reverse set of exposures made. Averaging the wavelengths obtained from the respective pairs of exposures tends to eliminate error from this cause.

The soundness of the principle involved in this method of photographic photometry is open to question, although at the Bureau of Standards consistent deviations in results from those by other methods were never noted [53]. The matter has been considered by various investigators but it is sufficient here to refer to the chapter by Jones in *Measurement of Radiant Energy* [6]. He discusses in detail the various factors involved in photographic photometry by various methods and concludes in this instance "that results are valid provided the sector speeds are sufficiently high." He further states, "It is impossible, of course, to make a general statement as to how fast sectors should be run to eliminate reciprocity-intermittency errors, but under the majority of conditions 30 interruptions per second are adequate." Since in the Hilger sector photometer both beams are interrupted by rotating sectors it is probable that error from this cause is not appreciable even at somewhat slower speeds.

However, to eliminate this uncertainty the Hilger Company put on the market another photographic photometer designated as the "Spekker" photometer [61, 3]. This design eliminated the rotating sectors and changed the optics to avoid the vertical dispersion introduced by the wedge-lenses and biprism (a more concentrated source can thus be used) and to produce a more nearly collimated beam through the solution and solvent. Instead of the rotating sectors, rectangular diaphragms are placed at appropriate places in the beams and the aperture in the solvent beam can be reduced by known amounts to compensate for the absorption of the solution in the other beam. Of course, for accurate work, this requires uniformity of radiant flux per unit area across the diaphragm. A similar photometer was previously designed by Lewis [62], except that vanes were used in place of the diaphragms. The methods are otherwise similar to the sector-photometer method.

In this country sector photometers are made and sold by the Bausch & Lomb Optical Co. [63] and the Gaertner Scientific Corp. [64] for use with certain of their spectrographic equipment. The principle in each case is the same as that of the Hilger sector photometer, but the sectors are synchronized so that variations in intensity of the

source are much more certainly eliminated than with the Hilger sector equipment. Absorption tubes up to 70- or 100-mm length can be accommodated.

Perhaps the principal defect of the above methods is the time involved in the measurements. The series of exposures usually takes a half hour or more, to which must be added the time involved in development and reading of the plate. Various schemes have been devised to speed up this process, the principal objective being to obtain the whole series of calibrated spectra at one exposure, so that unstable solutions can be recorded in a very short time interval. In a design by O'Brien [65, 11] a Lummer-Brodhun cube with up to 20 silvered strips alternating with the same number of clear regions is combined with a rotating logarithmic step sector, all placed just in front of the spectrograph slit and with dimensions determined by the length of the slit. Juxtaposed spectra are obtained, absorption in the solution being balanced by the reduced apertures in the step sector. In this manner the spectrogram obtained provides a direct plot of photographic density against wavelength with any value of exposure.

In another design by Twyman, a pair of Hilger echelon cells [3, 61] having variable internal thicknesses are combined to give a series of juxtaposed spectra on the photographic plate. Absorption in the solution is balanced by a suitable rotating sector. Again the dimensions are determined by the length of the spectrograph slit.

3. Photoelectric

Numerous photoelectric spectrophotometers have been designed by various investigators but not until the 1930's did any type become commercially available and have extensive usage. The two photoelectric spectrophotometers that have been chosen for illustration are the General Electric recording spectrophotometer and the Beckman quartz spectrophotometer. Spectral reflection as well as spectral transmission measurements can be made on both of these, and they are both commercially available.

(a) General Electric Recording Spectrophotometer

The present model of the General Electric recording spectrophotometer [42] originally designed by Hardy [40, 41], gave great impetus to colorimetric analysis and specification because (1) there was great increase in speed over previous nonrecording spectrophotometers, (2) it could make spectral reflection measurements just as readily as spectral transmission measurements, and (3) its precision and reliability were adequate for most types of measurement. The recording

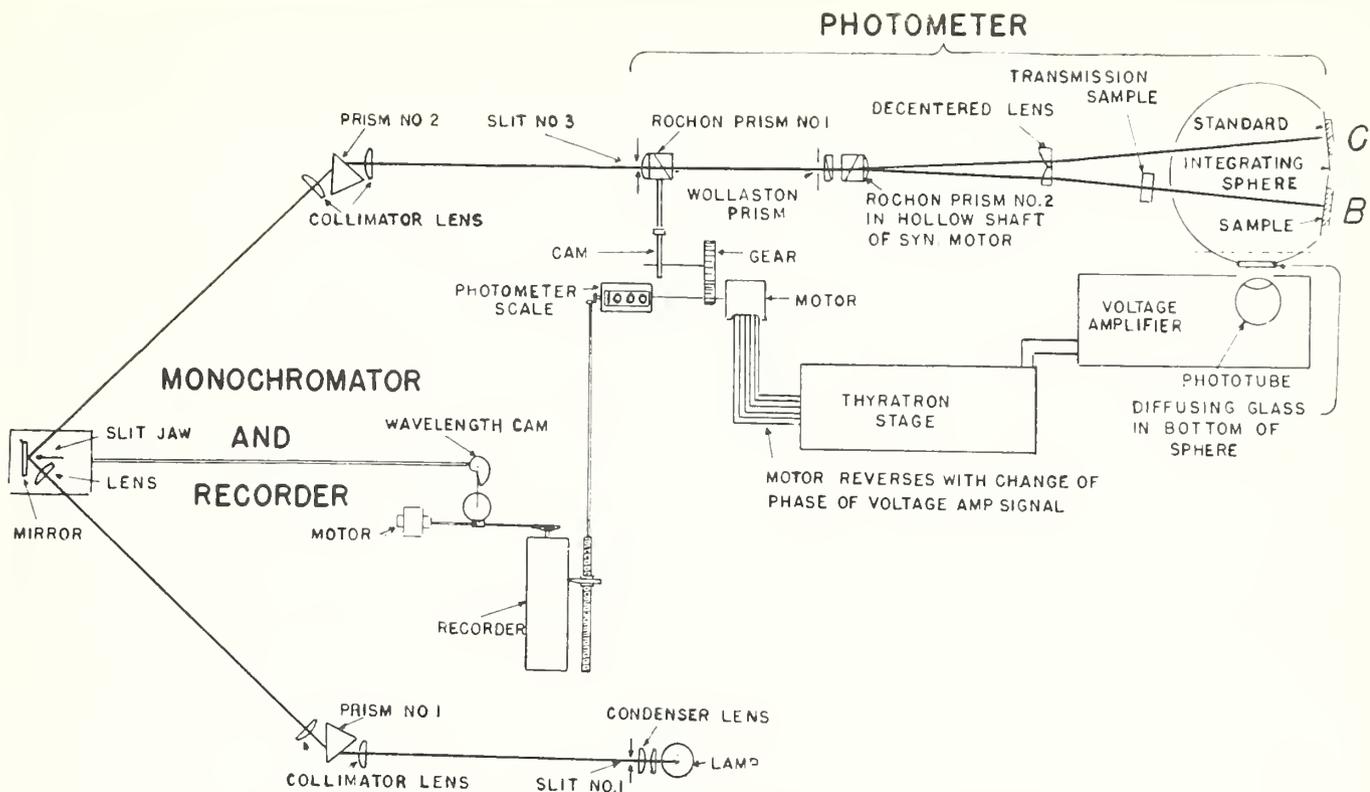


FIGURE 17. Schematic diagram of General Electric photoelectric recording spectrophotometer. On this instrument the radiant energy is spectrally dispersed before incidence upon the samples.

feature is also of great importance in many kinds of work.

A schematic diagram of the instrument is shown in figure 17 which is a reproduction (with slight changes) of figure 1 of Michaelson's paper [42]. Radiant energy from the lamp forms a prismatic spectrum in the plane of the mirror at the left of the figure. A second slit here isolates a small part of the spectrum and passes it on through prism No. 2. This second dispersion spreads the stray energy into a secondary spectrum in the plane of slit No. 3. This third slit obstructs most of this stray energy but transmits most of the spectral band isolated by the second slit. Thus there passes into the photometer a spectral band of width determined by the slits used, and almost wholly free of stray energy from other spectral regions. As the spectrum is traversed the mechanical widths of the slits are continually changed by cams so as to transmit a constant amount of spectrum expressed in terms of millimicrons. The usual nominal width is 10 $m\mu$ over the range from 400 to 700 $m\mu$ but instruments with 4, 5, and 8 $m\mu$ widths have been made and the wavelength range has occasionally been extended to 750 $m\mu$ (and with wider slits to above 1,000 $m\mu$), as on the National Bureau of Standards instruments.

The radiant energy transmitted by slit No. 3 passes in turn through (1) the balancing rochon prism No. 1, (2) the wollaston prism, where it is split into two beams polarized at right angles to each other, (3) the rotating rochon prism No. 2, mounted in the hollow shaft of a synchronous motor, (4) the separating decentered lenses, (5) the transmission compartment and (6) the sphere openings, and is finally incident upon the reflecting samples at *B* and *C*.

As rochon No. 2 rotates, the respective radiant energies incident upon *B* and *C* alternately go through successive maxima and minima, each maximum on *B* coinciding in time with the minimum on *C*. The instantaneous values of these alternating components are given in the following expressions [40]:

$$I = I_1 R_1 + I_2 R_2 = A^2 (R_1 \sin^2 \alpha \sin^2 \omega t + R_2 \cos^2 \alpha \cos^2 \omega t)$$

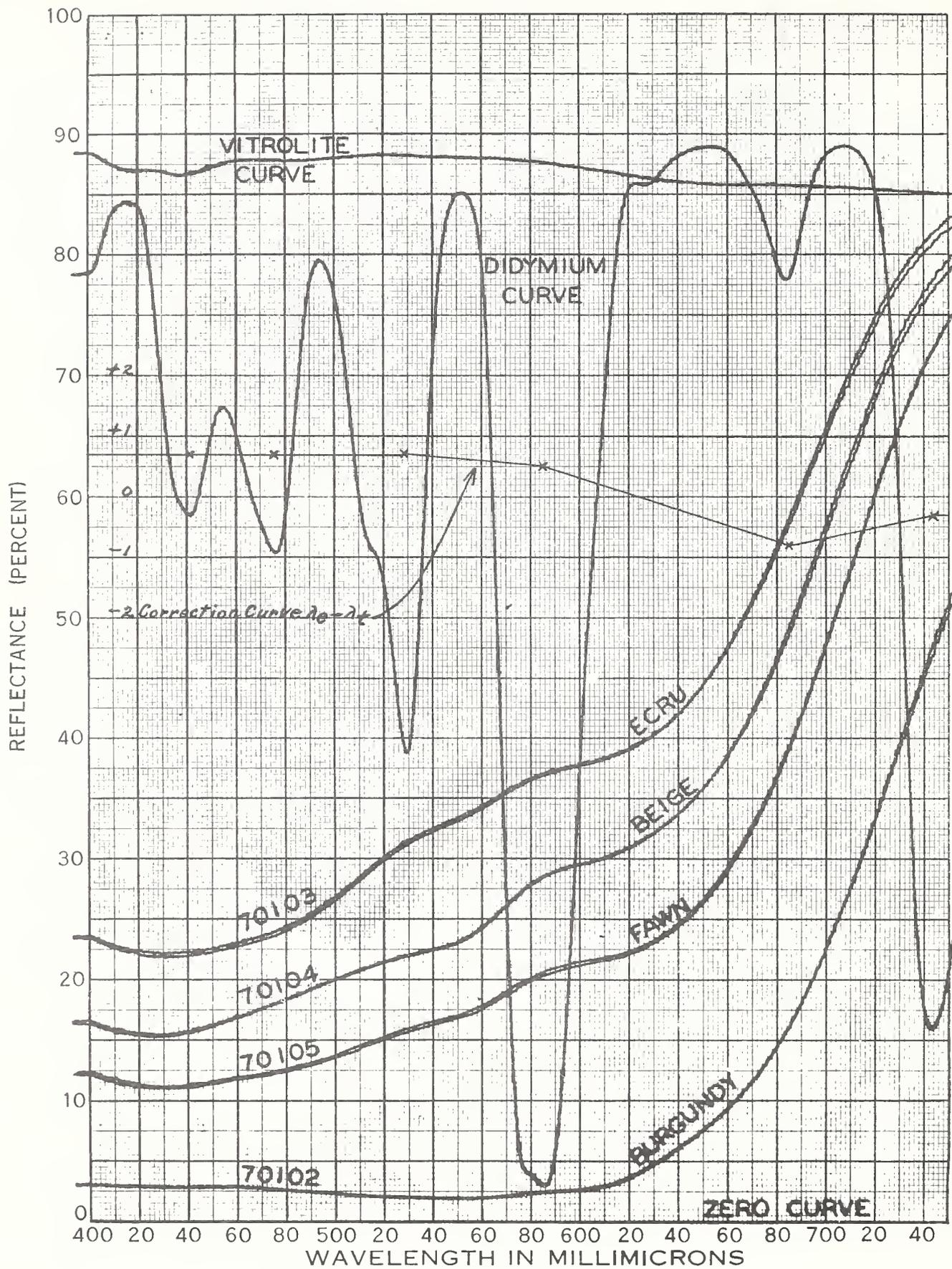
Let $A^2 = 2$, for convenience; then

$$I = (R_1 \sin^2 \alpha + R_2 \cos^2 \alpha) - (R_1 \sin^2 \alpha - R_2 \cos^2 \alpha) \cos^2 \omega t.$$

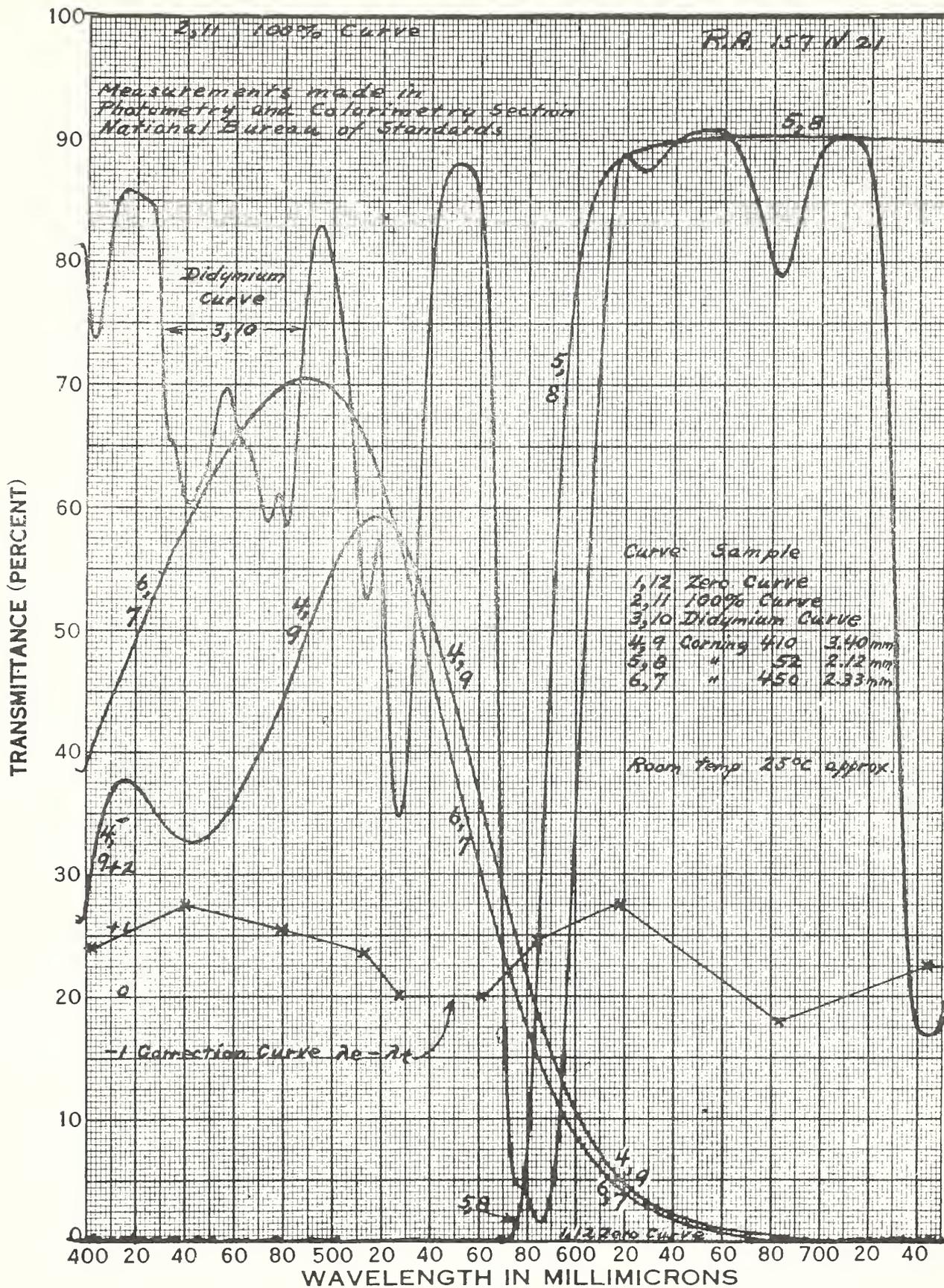
"Since the photoelectric current is a linear function of the light intensity, at least over a small range, this equation, when multiplied by a

FIGURE 18. Typical spectral transmittance curves recorded on a General Electric spectrophotometer at the National Bureau of Standards.

Zero, 100%, and didymium calibration curves are also shown. The didymium glass transmittance curve is used for wavelength calibration of the sheet, and the wavelength correction curve is shown plotted. Nominal slit widths used are equivalent to 4 $m\mu$ of spectrum.



(Legend on page 27)



(Legend on page 24)

suitable proportionality constant, represents the instantaneous value of the cell current. It will be seen that the cell current consists of a steady component proportional to $R_1 \sin^2 \alpha + R_2 \cos^2 \alpha$ and an alternating component of frequency $2f$ ($=\omega/\pi$) and amplitude proportional to $R_1 \sin^2 \alpha - R_2 \cos^2 \alpha$. Since the prism (rochon prism No. 2) rotates at 1,800 rpm the frequency of the alternating component is 60 cycles per second" [40].

A photoelectric cell of the cesium-oxide type is placed just beneath a ground-glass window in the bottom of the sphere. This phototube is connected, through an a-c voltage amplifier and a "thyatron stage", so that any alternating current from the photocell will in effect drive the balance motor, the direction of turn of the motor depending on the phase of the current. This phase will, in turn, depend on which of the two radiant energies reflected from *B* and *C* is the greater.

The motor is geared through a cam arrangement so that, as the armature turns, it turns rochon prism No. 1 and always in the direction that will balance the two radiant energies reflected from *B* and *C*. When these two reflected radiant energies are equal, there is no torque on the motor and the motor and rochon are at rest.

In operation, another motor (at the left) runs steadily; through cams it changes the wavelength of the radiant energy passing slit No. 3 by moving the second slit through the spectrum, and at the same time it turns the wavelength drum on which the recording paper is placed. The final connection is made by translating through levers and cams the amount that rochon No. 1 turns so as to operate a recording pen that moves up and down on the paper. This pen moves linearly with the transmittance or reflectance of the sample and thus the respective curve is steadily traced through the spectrum. In addition to the normal cam, cams giving " $\times 5$," "log," and "loglog" curves can be used.

Typical sheets of such recordings as regularly run at the National Bureau of Standards are shown in figures 18 and 19. Figure 18 is the record of three transmittance curves run for certain glasses as indicated. The slits for this recording were equivalent to approximately $4 \text{ m}\mu$ of spectrum. In this figure will be noted first the three calibration curves. (1) The "zero-curve" is run with the sample beam blocked off. (2) The "100-percent curve" is run with no sample in either beam. (3) The "didymium curve" is run with a calibrated didymium glass in the sample beam; this curve is for wavelength calibration of the sheet as explained in section III-2. After the

three calibration curves, the curves for the samples tested are successively run; then the whole series of sample curves and calibration curves are repeated in reverse order. This repetition insures against undetected erratic or gradual changes in conditions during the run. Each curve takes $2\frac{1}{2}$ or 5 minutes, at the choice of the operator, depending on the selectivity.

Figure 19 shows a similar graph sheet for directional reflectance measurements made on the other recording spectrophotometer at the National Bureau of Standards, this instrument having slits equivalent to approximately $10 \text{ m}\mu$ of spectrum. (For reflectance measurements the "transmission sample" is of course removed.) The order and purpose of the respective curves is the same as in figure 19, except that a "Vitrolite curve" is run instead of the 100-percent curve. The purpose of the Vitrolite curve is to enable all of the reflectance curves to be corrected so as to be truly relative to freshly prepared MgO, the accepted standard for such work; see section VII-3. This is done as follows.

In transmission measurements it does not matter what the reflecting materials *B* and *C* (fig. 17) are, provided they will give a "100-percent" curve entirely on the network part of the sheet. (The cams are reliable for extrapolation above the network to only about 100.5 percent.) They can be fresh or old MgO, MgCO_3 , white glass, white paper, etc. However, it is a matter of importance that the reflectance be high, to avoid loss of sensitivity; and it is a matter of convenience that *B* and *C* be essentially identical, so that the 100-percent curve will lie closely if not exactly on the 100-percent coordinate line. The substitution principle is fully complied with when at any wavelength the transmittance is computed from the ratio of ordinates of the transmission sample curve to the 100-percent curve (correcting each for the zero curve if necessary).

For spectral reflection measurements (transmission sample removed), if one is willing to prepare identical fresh MgO standard surfaces each day, the operation is similar to that for transmission measurements, except that by the substitution principle the MgO surface at *B* is the standard surface; the other MgO at *C*, identical or not, is merely the comparison sample. Because of the slight decrease in reflectance of the MgO (see section VII-3), it is necessary that each MgO sample be newly prepared each day. If *B* is not newly prepared it is no longer standard; if *C* is not newly prepared (and *B* is), the 100-percent curve will rise off the network below $500 \text{ m}\mu$. Of course if it is known by experience that the 100-percent

FIGURE 19. Typical spectral directional-reflectance curves recorded on a General Electric spectrophotometer at the National Bureau of Standards.

Zero, Vitrolite, and didymium calibration curves are also shown. The didymium glass serves for wavelength calibration as in figure 18, but the shapes of the correction curves on the two instruments are different. Nominal slit widths equivalent to $10 \text{ m}\mu$ of spectrum are used on this second instrument. The Vitrolite curve replaces the 100% curve and enables the reflectance data to be reduced relative to freshly prepared magnesium oxide.

curve is "on the line" and one wishes to take the chance, he can omit the 100-percent curve and simply prepare sample *C* each morning for the day's operation.²

This is risky, however, for certain kinds of work, for experience has indicated that newly prepared MgO samples may very slightly (0.1 or 0.2 percent) for unknown reasons. At the National Bureau of Standards it has been considered better to calibrate a working standard of directional reflectance (placed at *B*), by comparison with several different new preparations of MgO and to repeat this check occasionally to detect any possible change in the working standard.

The "Vitrolite curve" in figure 19 represents such a calibrated standard of directional reflectance [43]. By its use, comparison sample *C* can

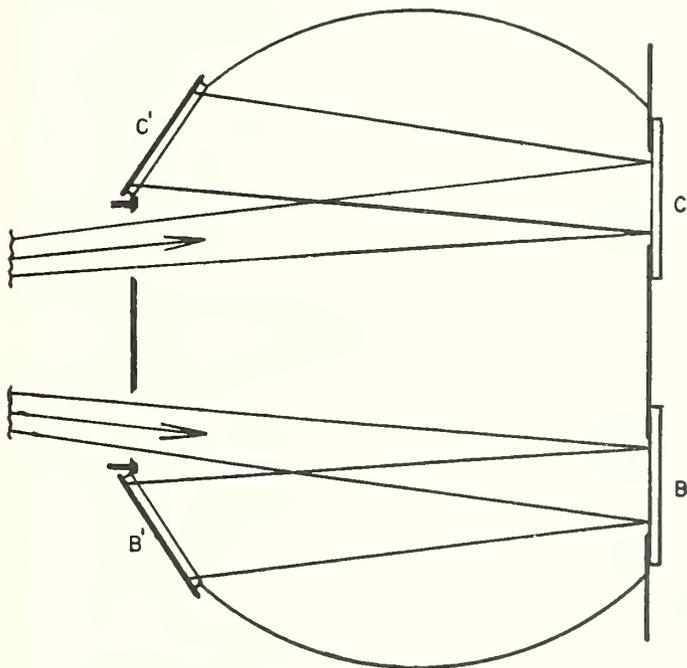


FIGURE 20. Design of sphere used on newer models of General Electric spectrophotometer.

The energy is incident at 6° and if the surface of *B* is plane and polished the specular component of the reflected energy is received at *B'* as shown. *B'* may be a "light trap" to absorb and thus exclude this specular component from the measurements, or it may be an MgO surface to reflect and thus include this component. In the older design of sphere, shown in figure 17, the energy is incident at right angles to *B* and part, but not all, of the specular component is reflected out through the entrance aperture.

be any sample of high enough reflectance to keep the recording within the network; however, in order to keep the recordings close to their true values (which has several advantages), it is well for sample *C* to be a reasonably fresh surface of MgO. At any wavelength, the correct value of the directional reflectance of the sample (placed

at *B*), relative to that of fresh MgO, is obtained by dividing the recorded ordinate for the sample by the ratio of the recorded ordinate of the Vitrolite to the correct value for the Vitrolite relative to fresh MgO.

The type of directional reflectance measured is $R_{0,a}$ with the type of sphere illustrated in figure 17; part of the specular component of the energy reflected from a plane polished surface at *B* goes back through the entrance aperture. With the newer design of sphere, shown in figure 20, the radiant energy is incident upon samples *B* and *C* at approximately 6° and the specular component of the reflected energy is returned to the sphere apertures at *B'* and *C'* as shown. If the surface of the test sample placed at *B* is plane and polished, this specular component can be wholly excluded from or included in the measurements by either placing a completely absorbing "light trap" at *B'* or filling the aperture with a sample of fresh MgO. The respective directional reflectances may be designated as $R_{6,a}$ and $R_{6,D}(=R_6)$. However, two things of importance should be noted in this connection:

1. *Specular component included ($R_{6,D}$).* If the sample at *B* is 96 percent diffusing and only 4 percent specularly reflecting (approximated by a plane, polished white glass), the error involved (in $R_{6,D}$) if the reflectance of *B'* does not quite equal that of fresh MgO is of second order, a small fraction of the 4 percent. However, if the sample at *B* is a polished mirror, so that the whole of the reflected beam is first incident upon *B'* before being diffused throughout the sphere, the error is of first-order importance and should be carefully considered.

2. *Specular component excluded ($R_{6,a}$).* If the surface of the sample is not plane but is mottled or irregular in flatness, such, for example, as a piece of enameled iron may be, some of the specularly reflected energy may not be caught in the light trap at *B'*, and the specular component is thereby not completely excluded from the measurements.

The wavelength correction curves for the graphs of figures 18 and 19 are shown in the graphs. The method of applying this correction is simply to read the values of transmittance or directional reflectance at $\lambda + (\lambda_e - \lambda_i)$ instead of at λ . The most effective method of reading such values is usually decided for himself by each reader; equipment facilitating the curve reading has been designed by Reimann [66].

Many other details relating to the General Electric or similar recording spectrophotometers may be found in papers issued from the National Bureau of Standards [38, 10, 67] and in papers published by Pineo [68], Stearns [69], and Buc and Stearns [70, 39, 71]. Discussion of the error possible with fluorescent samples is given in section VI-5.

² It has been the Bureau's experience that the MgO may decrease perceptibly in the violet in a day or so after preparation. Others seem to have found somewhat greater stability, perhaps due to different ways of preparing the surface. How often one might need to prepare new surfaces will depend on his experience in the matter and on the accuracy of the work he is attempting to do. See also section VII-3.

(b) Beckman Quartz (Nonrecording) Spectrophotometer

The Beckman quartz photoelectric spectrophotometer, model DU, has several features not found in other commercial spectrophotometers: (1) It covers the wavelength range from 210 to 1,000 $m\mu$ approximately, (2) transmission measurements in the ultraviolet from 210 to 400 $m\mu$ are made as readily as in the visible spectrum, (3) directional reflectance measurements over the range from 350 to 1,000 $m\mu$ can be made under conditions closely equivalent to those recommended by the International Commission on Illumination in 1931 for colorimetric work.

The optics of the apparatus for transmission measurements are as shown in figure 21, taken from the paper by Cary and Beckman [37]. Radiant energy from an incandescent lamp or other source, *A*, is focused on the slit, *D*, by means of the concave mirror, *B*, and the plane mirror, *C*. The beam entering the slit is collimated by the mirror, *E*, and passes through the quartz prism to the reflecting surface, *F*. After reflection at *F* the beam returns along nearly the same path to the same slit, *D*, where it emerges slightly above the entrance beam and the mirror, *C*. After passage through the sample or sample compartment, *G*, the beam is incident on the phototube, *H*.

Two phototubes are supplied installed in the instrument, either of which may be inserted in the beam at will. One is a "cesium oxide" phototube ("red sensitive") for use primarily above 620 $m\mu$. The other is a special "cesium antimony" phototube ("blue sensitive") for use primarily below 620 $m\mu$. If incandescent lamp and common glass phototube are used, this range terminates at about 320 $m\mu$. If the tube has an envelope of Corning 9740 glass, and if a hydrogen lamp with a window of the same type of glass is used as source (all supplied with the instrument when desired), the transmission range extends to nearly 200 $m\mu$.

The photometric scale is based on electrical rather than optical principles. The photoelectric current from the irradiated phototube produces across the load resistor a voltage drop that is balanced by a potentiometer. While this null

setting is being made, any imbalance is amplified electronically and is indicated by a millimeter on the instrument. Accuracy of the photometric scale depends among other things on conformity of the phototube load resistor to ohm's law and on the linearity of the irradiance-current relationship. Since vacuum phototubes are used, the relation should hold closely. Linearity is still further assured by the use of a frame-type anode in the phototube.

Various details regarding the instrument and its operation in spectral transmission measurements may be found in a recent paper from the National Bureau of Standards [33] as well as in the original paper. The following points may be noted here:

1. The slits are narrower than usual on commercial spectrophotometers, normally isolating spectral regions of only 0.5 to 1.5 $m\mu$ within the range from 220 to 950 $m\mu$. This feature of the instrument serves three useful purposes. It enables the spectral transmittance of samples having high selectivity to be determined with an accuracy unattainable with instruments having wider slits. It makes possible the precise and accurate comparison of nearly identical samples, even those having high absorption, without the use of slits unduly wide. It also makes possible the reliable determination of very low transmittances by a "step-down" procedure using samples of intermediate transmittance.

2. No error was detected in the photometric scale on an instrument obtained at the National Bureau of Standards in 1945. These tests were made by measuring and comparing the spectral transmittances of certain glass standards on the Beckman, the König-Martens, and the Gibson spectrophotometers, and together with many other details are described in the recent publication [33].

3. Residual wavelength errors found on two Beckman spectrophotometers are shown later in figure 27, curves 1 and 2. A screw adjustment provides for bringing any desired wavelength into perfect alinement. The mercury wavelength 546.1 $m\mu$ has been found suitable for this purpose.

4. The quartz mercury arc supplied with the instrument serves not only for wavelength calibration but can be used at certain wavelengths for photometric measurements (section IV-1).

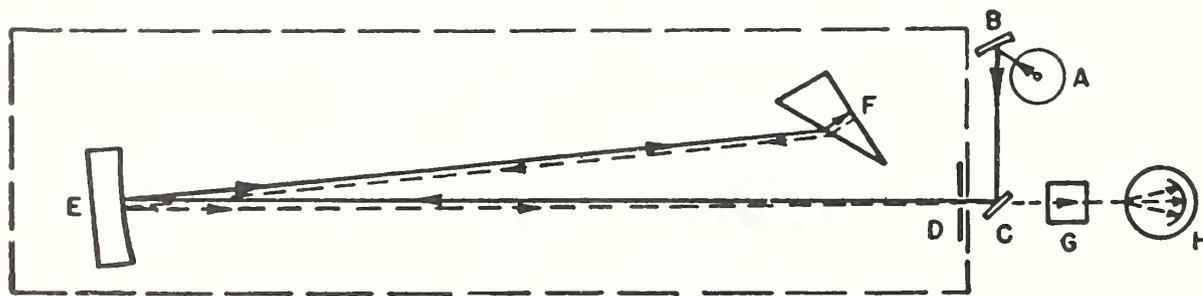


FIGURE 21. Optics of the Beckman quartz nonrecording spectrophotometer.

In this instrument the energy is spectrally dispersed before incidence on the sample.

5. The hydrogen arc supplied with the instrument gives a sufficiently continuous spectrum that transmission measurements can be made at any wavelength from 210 to 400 $m\mu$.

6. Trouble has been experienced in the measurement of polarizing materials if the quartz plate over the entrance slit is not cut with faces perpendicular to the axis [33]. At the National Bureau of Standards this plate has been removed.

7. The instrument has the usual amount of stray energy present for single-dispersion spectrophotometers. The purple filter supplied with the instrument should always be used with incandescent source below 400 $m\mu$, and other stray-energy filters should be obtained and used under certain circumstances (section VI-3).

8. Any deviation of the beam by the sample (if wedge-shaped or lens-shaped, for example) may cause error in the measurements.

Directional reflectance measurements can readily be made on the Beckman spectrophotometer by means of special attachments obtained from the manufacturer. The optics of such measurements are shown in figure 22. The radiant energy

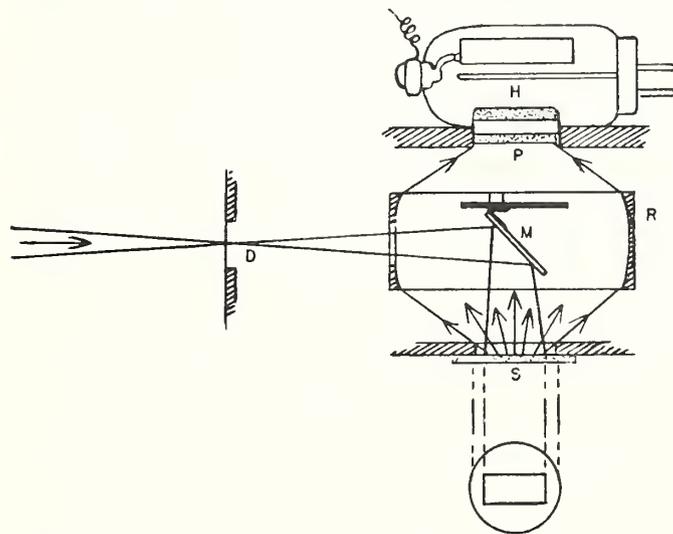


FIGURE 22. Optics of the reflectance attachment to the Beckman quartz spectrophotometer.

The incident energy from the exit slit, D , is directed by the mirror, M , so as to be incident at right angles on the sample or standard placed at S . The energy reflected from S at approximately 45° in all directions is focused by the ellipsoidal mirror ring, R , onto the diffusing glass, P , immediately under the phototube, H .

transmitted through the exit slit, D , is directed downward by means of the mirror, M , and is incident approximately at right angles upon the sample or standard at S . The irradiated area is about 8×17 mm. A carrier enables either the sample or the reflectance standard with which it is being compared to be placed in turn in the beam.

The energy reflected from the sample or standard at approximately 45° in all directions is reflected and focused by the ellipsoidal metallic mirror ring at R onto the ground-glass plate at P .

The phototube compartment is mounted on top of the attachment so that the phototube, H , is just above the diffusing glass.

The operation of the instrument for reflection measurements is similar in all respects to that for transmission measurements. The following points may be noted, however:

1. The slit widths necessary for normal operation are much wider than for transmission measurements. They vary from 3 to 4 $m\mu$ in the region near 530 $m\mu$ to about 8 $m\mu$ at 350 $m\mu$, and 15 $m\mu$ at 650 $m\mu$. These widths may be made to vary considerably by adjustment of the sensitivity dial, however, or by operation solely at the ".1" position of the selector switch.

2. While the axis of the reflected energy is closely at 45° , the angular spread of the beam is approximately 20° (35° to 55°). The directional reflectance, $R_{0,45}$, thus measured, is numerically equivalent to that, $R_{45,0}$, recommended by The International Commission on Illumination, as noted above, except that the ICI recommendation implies that the spread of the reflected beam should be zero.

3. In reflection measurements of fluorescent materials the values obtained are liable to the same sort of error as with the General Electric spectrophotometer (section VI-5).

(c) Other Instruments and Methods

In addition to the General Electric and the Beckman spectrophotometers, there are many other photoelectric spectrophotometers of various designs that may be noted here. Some of these are commercial instruments: (1) The Cenco-Sheard spectrophotometer, 325 to 750 $m\mu$, using concave reflection grating and barrier-layer photocell [72]; (2) the Coleman "double monochromator" spectrophotometer, 350 to 1,000 $m\mu$, using transmission gratings instead of prisms [73]; (3) the Coleman "universal" spectrophotometer, 300 to 800 $m\mu$, using single grating with barrier-layer cell, and designed primarily for chemical analysis in the industrial laboratory [73]; (4) the Coleman "junior clinical" spectrophotometer, 400 to 700 $m\mu$, designed solely for clinical analysis [73]; (5) a Gaertner instrument using two spectrometers and a Martens photometer, 450 to 820 $m\mu$ [74].

Two new instruments now appearing on the market are the Cary recording quartz spectrophotometer, 200 to 700 $m\mu$ [75] and the Beckman model B spectrophotometer, 320 to 1,000 $m\mu$ [76].

Among those designed and used by various investigators since 1930, but so far as known not put on the market (those before 1930 are listed in reference [2]), may be noted instruments or equipment by Preston and Cuckow [77], Hogness, Zscheile, and Sidwell [78], Schlaer [79], Zworykin [80], Harrison and Bentley [81], Brode and Jones [82], Gillod [83], Drabkin [84],

Little [85], Studer [86], Hardy [87], and Zscheile [88].

Some of these papers treat the subject of photoelectric spectrophotometry in considerable detail, describing methods of wavelength calibration, slit-width and stray-energy errors, absorption cells, treatment of data, and other matters of interest and importance. Examination of them should be of great benefit to those who wish to study the numerous possibilities in the design of such instruments and the use and reliability of the data obtained.

4. Filter Photometers

For certain kinds of work it has been found adequate to isolate desired spectral regions by means of optical filters, each of which will transmit a more or less narrow region of the spectrum. Since the selection of spectral regions is dependent upon the filters at hand, the method has sometimes been referred to as abridged spectrophotometry [89]. Instruments incorporating such means of spectrum variation are known as filter photometers, chemical photometers, or chemical colorimeters, and by various trade names. The present section deals primarily with the filters available for such instruments.

In general, of course, what is desired is a filter that will transmit freely over a narrow range of wavelengths and absorb or reflect completely at all other wavelengths at which (1) the source emits radiant energy in important amounts, and

(2) the detector gives appreciable response. If either (1) or (2) is negligible over a given spectral range the spectral transmittance of the filter is unimportant over the same range.

The most suitable filter, or series of filters, for use in any particular instrument or type of work depends, therefore, not only on the spectral range of direct interest but also on the spectral characteristics of the source, detector, and filter over a much wider range. Filters that might be adequate for use with a mercury arc might be wholly unsuitable with an incandescent source. Filters that might be satisfactory for photographic or visual use might give highly inaccurate results if used with a phototube or thermopile as detector.

Several types of filters are available for general use. These are (1) glasses, usually two or more in combination, (2) two or more dyes in solution or incorporated into gelatine or other medium, (3) interference filters, and (4) miscellaneous types.

General information regarding the spectral transmission characteristics of such filters, or of the individual components or selectively absorbing constituents of such filters, may be found in various publications from the National Bureau of Standards [90, 91, 92, 93], in advertising circulars of the Corning Glass Works [94], the Eastman Kodak Company (the Wratten dyed gelatine filters) [95], and the Farrand Optical Company and Baird Associates (the interference filters) [96, 97], and in various other publications [98, 99, 100]. Special sets of filters are also often supplied with the various instruments on the market, and information

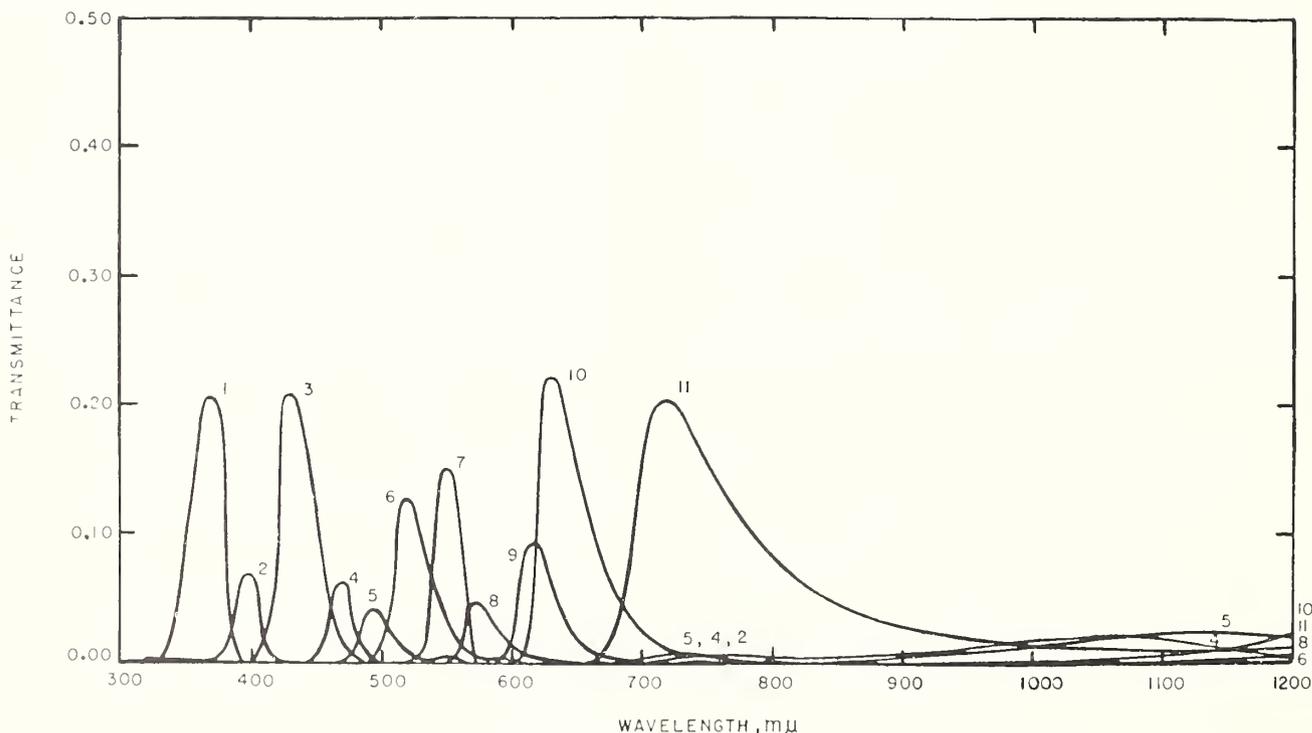


FIGURE 23. Spectral transmittance curves of Corning glass filters (or filter combinations) which may be used for wavelength isolation in filter photometers.

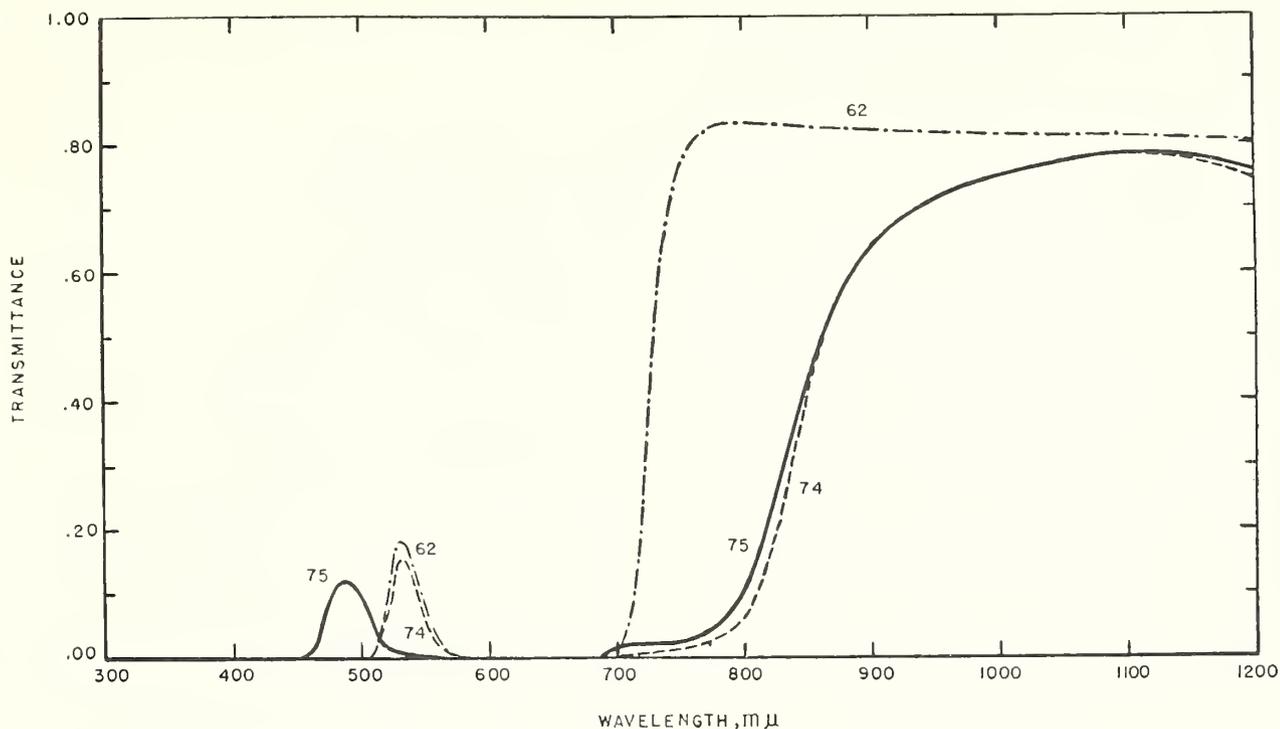


FIGURE 24. Spectral transmittance curves of Eastman Wratten (dyed gelatine) filters which may be used for wavelength isolation in filter photometers.

regarding the spectral transmittances of such filters may doubtless be obtained from the makers or sellers of the instruments.

In figure 23 are shown certain typical curves obtainable with glass filters sold by the Corning

Glass Works, in figure 24 similar curves of certain Wratten filters, in figure 25 curves for certain interference filters, and in figure 26 curves for miscellaneous filters. All of the data for the curves of these four figures were obtained at the National Bureau

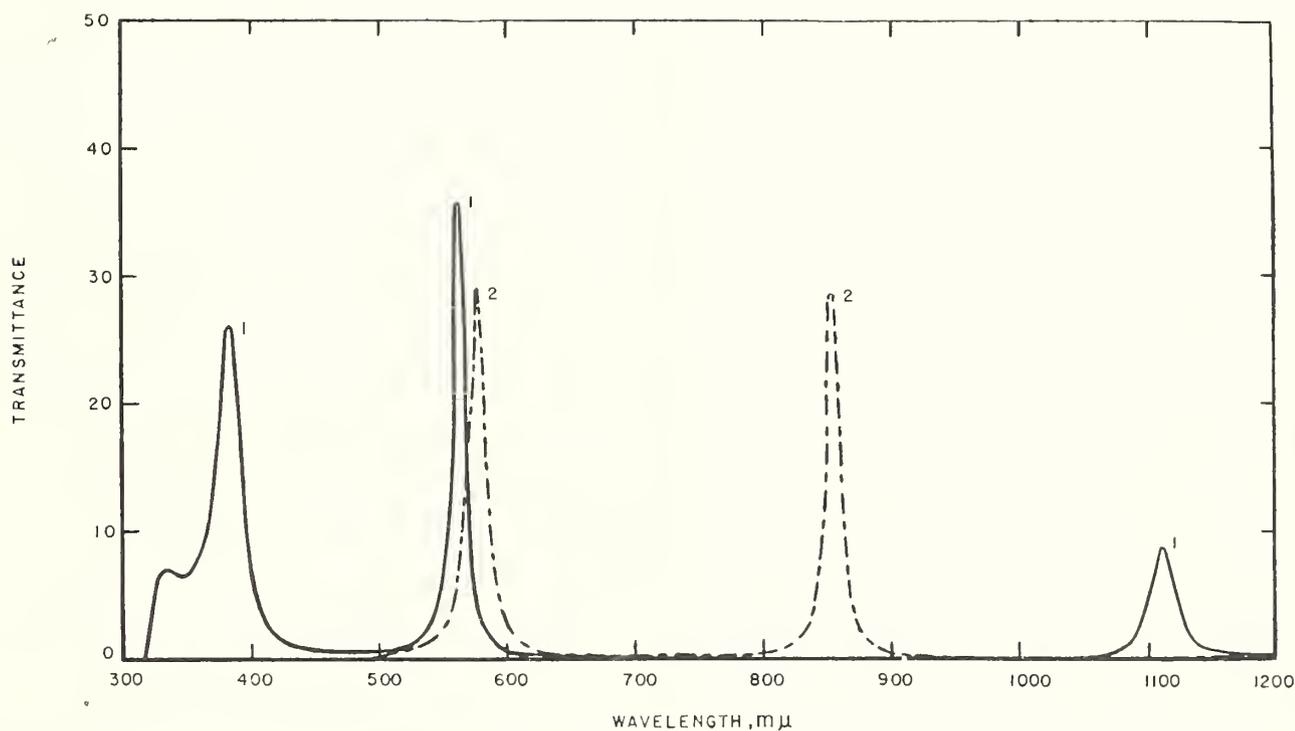


FIGURE 25. Typical spectral transmittance curves of Baird (curve 1) and Farrand (curve 2) interference filters which may be used for wavelength isolation in filter photometers.

Filters are obtainable with transmittance bands at nearly any desired wavelengths.

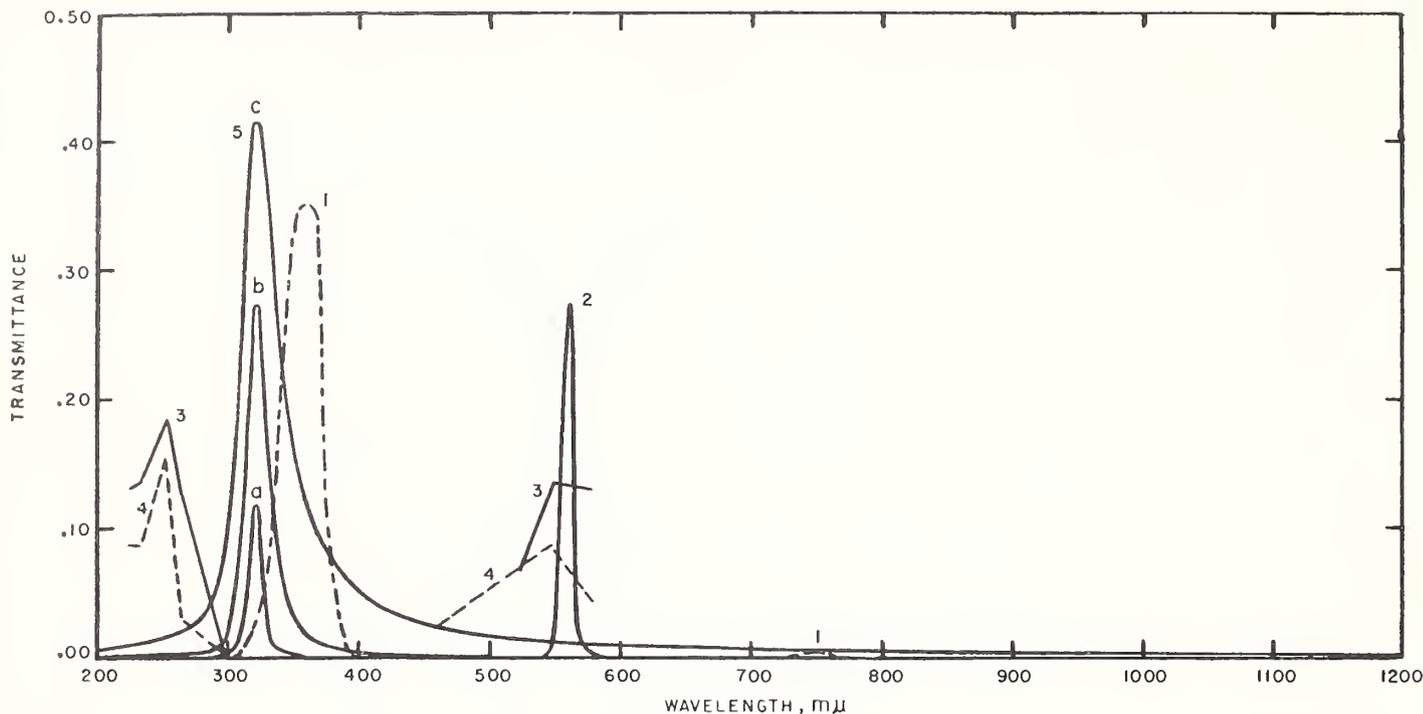


FIGURE 26. Spectral transmittance curves of miscellaneous filters which may be used for wavelength isolation in filter photometers.

1. Corning 5860 glass. 2. Gibson 4-component glass filter. 3. Quartz cell containing mixture of chlorine and bromine gas. 4. Quartz cell containing chlorine gas. 5. Fused quartz plates carrying silver films of the following thicknesses: (a) 0.0552 micron, (b) 0.0326 micron, (c) 0.0217 micron.

of Standards on samples purchased from the manufacturers, with the following exceptions: (1) The silver films of figure 26 were prepared on fused quartz by L. W. Scott of the National Bureau of Standards; (2) the data of curves 3 and 4 of figure 26 were taken from reference [92] originating with Peskov, *J. Phys. Chem.* **21**, 382 (1917), and these papers should be consulted for further details. Both the Farrand Optical Company and the Baird Associates advertise filters similar to those shown over the range from 400 to 700 $\mu\mu$; in this range with these filters, there is no inherent limitation on the wavelength region that may be isolated. It is understood that similar interference filters for use in the ultraviolet or infrared are in process of development.

Two things of importance should be considered in connection with the selection and use of filters for abridged spectrophotometry, in addition to factors discussed elsewhere. One of these is the wavelength to be assigned to the photometric value obtained by use of the filter with incandescent source; the other is an expression for the effectiveness of such filters; with respect to their transmittance at the nominal or desired wavelength and their exclusion of radiant energy at all other wavelengths. In this connection the shape of the transmittance curves shown in figures 23 to 26 may be compared with the shape of the slit-width "curves" of figure 6. In effect, the use of

filters is more or less equivalent to the use of wide slits on a spectrophotometer, although the generality and precision of wavelength settings are missing with the filters.

If the photometric values obtained with a series of filters are to be used to plot a transmittance or reflectance curve, the correct wavelength at which to plot any particular value is, of course, the one for which the same value would be obtained in accurate spectrophotometry.

The photometric value obtained by use of the filter is

$$R = \frac{\int_0^{\infty} S_{\lambda} I_{\lambda} T_{\lambda} R_{\lambda} d\lambda}{\int_0^{\infty} S_{\lambda} I_{\lambda} T_{\lambda} d\lambda},$$

where

- S_{λ} = spectral response of the detector (eye, phototube, thermopile, etc.) for an "equal-energy" spectrum,
- I_{λ} = relative spectral energy of the illuminant,
- T_{λ} = spectral transmittance of the filter,
- R_{λ} = spectral distribution of the measured quantity, and
- λ = wavelength.

Since, in general, R_{λ} is unknown, this relation is not useful. As a first approximation to the correct wavelength at which to plot R , it is customary

to use the spectral centroid, λ_c , as defined in the following relations:

$$\lambda_c = \frac{\int_0^{\infty} S_\lambda I_\lambda T_\lambda d\lambda}{\int_0^{\infty} S_\lambda I_\lambda T_\lambda d\lambda}$$

or

$$\lambda_c = \frac{\int_0^{\infty} S_\lambda I_\lambda T_\lambda R_\lambda d\lambda}{\int_0^{\infty} S_\lambda I_\lambda T_\lambda R_\lambda d\lambda}$$

Values of λ_c by the second equation need be used only for highly selective materials (R_λ varying widely over the range of T_λ), and for such cases the values of λ_c by the other equation must first be obtained and R plotted as a function of λ_c yielding approximate values of R_λ . For nearly achromatic materials such as white or near-white enamels, papers, etc., the variation in R_λ is usually so small as to be negligible for the purpose, and values of λ_c by the first equation may be used. In general, it is much more important to know the values of T_λ accurately than the values of S_λ and I_λ ; and the narrower is the spectral region transmitted by the filter, that is, the more restricted is the range of T_λ , the less accurately will the values of S_λ and I_λ need to be known. The integrations are usually made by summations at every 10 $m\mu$; but if the filter is very restrictive, 5 $m\mu$ intervals are necessary.

The effectiveness of a filter for use in abridged spectrophotometry is often given in terms of its maximum transmittance and its "half-width." By the latter term is meant the wavelength interval between the two sides of the transmittance curve, taken at a transmittance equal to half the maximum. These specifications are good as far as they go, but they ignore the radiant energy transmitted at wavelengths near the base of the transmittance curve or at some other wavelength region perhaps far removed from λ_c .

An expression for the effectiveness, E , of a filter for transmitting and isolating radiant energy at any specified wavelength, λ' , has been suggested [93]:

$$E_{\lambda'} = T_{\lambda'} \frac{\sum S_\lambda I_\lambda T_\lambda |\lambda - \lambda'| \Delta\lambda}{\sum S_\lambda I_\lambda |\lambda - \lambda'| \Delta\lambda}$$

where the symbols have the same meanings as above and the absolute value signs ($|\ |$) indicate that the differences are to be taken all greater than zero. Other relations for expressing the various characteristics of a filter are given in the same publication.

If, instead of an incandescent source, a mercury

are or other source is used having intense lines at suitably spaced wavelengths, the filter photometer can give photometric values at these wavelengths with all the reliability and certainty of a true spectrophotometer. The problem is simpler than with the incandescent source in that the wavelength isolated by the filter is more definite, and in many cases the exclusion of unwanted energy is less difficult. Filters designed specifically for use with the mercury arc in abridged spectrophotometry have been developed by various investigators [91, 94, 95, 96, 97, 101, 102, 103].

Numerous designs of filter photometers are on the market. Examination of such instruments will show that they combine all of the essential parts of a spectrophotometer except that the series of filters replaces the more costly prism (or grating), lenses (or mirrors), and slits of the conventional instrument.

All of the types of errors that are listed in sections VI-1 to VI-7 may be present in measurements made with filter photometers. The wavelengths to be assigned to measurements with the filters were considered above. Reliability of the photometric scale is dependent on the same principles as in a true spectrophotometer; on many of the photoelectric instruments the linearity of the irradiance-current relationship must be carefully considered, and on visual instruments the soundness of the photometric device should be established in one way or another. Stray energy depends directly upon the spectral transmittance of the respective filters over the whole range of sensitivity, as already explained. Temperature effects must be carefully considered not only from the standpoint of the temperature of the sample, but also because the response of a barrier-layer cell varies so markedly with temperature. The magnitudes of errors resulting from fluorescence of the sample will depend on whether the radiant energy passes first through the sample or through the filters; but will always be less important in transmission measurements of nondiffusing materials than in reflection measurements of diffusing materials (see section VI-5). Slit-width errors can be very large because of the width of spectrum band passed by most of the filters; in some cases the error might be interpreted as a wavelength error; in either case the magnitude will depend importantly on the second derivative of the spectral transmission curve across the spectral range isolated by the particular filter being used (see section VI-6). Other errors, such as deviation of the beam when the sample is inserted, the effects of multiple reflections, electrical or mechanical instability, and so on, all must be carefully considered if the most reliable work is to be done with such instruments.

In this connection it may be noted that the types of filters desired for filter photometers in abridged spectrophotometry, such as here con-

sidered and illustrated in figures 23 to 26, are radically different from those necessary when the filter photometer is used for tristimulus colorimetry. In this colorimetric use, filters are de-

sired that adequately duplicate the tristimulus functions of the human eye. Such filters cover a wide spectral range and would give only the crudest sort of spectrophotometric analysis.

VI. Errors—Discussion and Elimination

Numerous types of errors may be present in spectrophotometric measurements, and only the most careful calibration and operation of the instrument will render such errors negligible if the highest all-around reliability is desired. However, the importance of spectrophotometric errors varies greatly with the purpose of the measurements. One general statement is always true: The greater is the deviation between the spectral distributions of sample and standard, the more likely is the measured ratio of sample to standard to be in error.

Sometimes these errors may result from a fundamental defect in design or operation of the instrument; sometimes they result directly from carelessness or lack of training of the operator; sometimes they result from uncontrolled variables such as temperature and humidity. Whatever the cause, it is repeated here for emphasis that the importance of spectrophotometric errors depends on the purpose of the measurements. This is true for each type of error to be discussed but will not be noted further. Rather, what will be considered will be the causes of the errors and their magnitudes.

1. Wavelength

Two errors are to be distinguished, the error in the wavelength scale or calibration and the

error thereby produced in the measured ratio. For any given wavelength error, the error induced in the ratio will depend directly on the spectral selectivity of the sample relative to the standard. This is fairly obvious; if the sample is nonselective at any wavelength or wavelengths, a wavelength error at those wavelengths induces no error in the measured ratio, but if the sample is highly selective, such as a selenium or didymium glass at certain wavelengths, an error of even 0.1 $m\mu$ has a measurable effect on the transmittance obtained. It is a simple matter for each investigator to determine the magnitude of this effect on the sample being measured.

It is believed that all commercially available spectrophotometers now have direct-reading wavelength scales. It is perhaps a matter of interest to note the residual errors found on various of the direct-reading wavelength scales of spectrophotometers in the Photometry and Colorimetry Section of the National Bureau of Standards. These are shown in figure 27, and give an idea of what to expect in a direct-reading instrument when put into such adjustment that the sum of these residual errors is kept close to a minimum. The deviations seldom exceed $\pm 1.0 m\mu$. It is to be emphasized, however, that wavelength errors as small as shown are dependent on optimum adjustment of the instrument. It is easily possible for an instrument to get badly out of adjustment, and continual check is strongly advised.

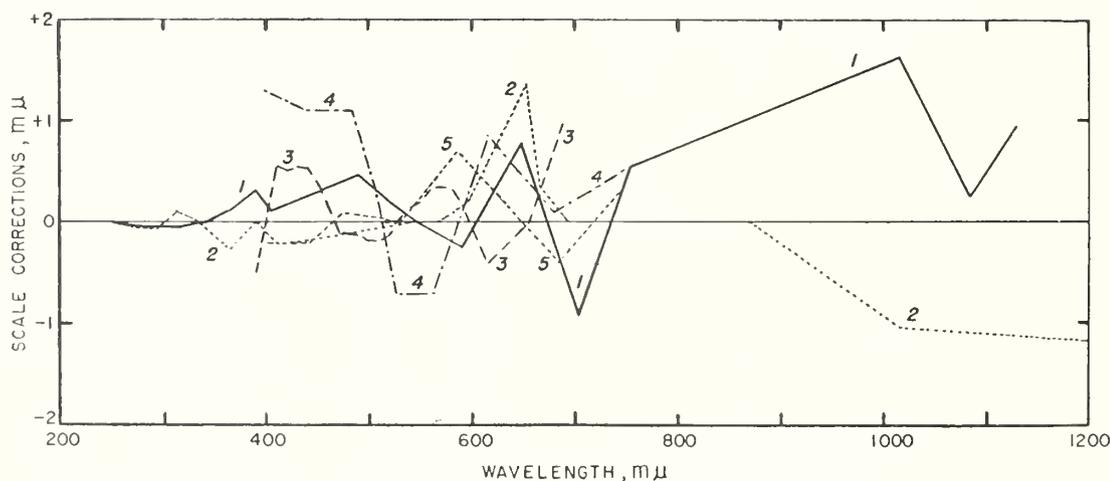


FIGURE 27. Wavelength correction curves of various spectrophotometers in the Photometry and Colorimetry Section of the National Bureau of Standards.

All of the instruments are direct-reading in wavelength and are adjusted so that corrections are close to minimum. Curves 1 and 2 are for Beckman spectrophotometers, curve 3 for a Hilger spectrometer used in the Gibson spectrophotometer, curves 4 and 5 from typical recordings for General Electric spectrophotometers.

2. Photometric Scale

A check of the photometric scale of a spectrophotometer independent of all other sources of error is difficult or impossible to make on most spectrophotometers. Useful for this purpose would be a series of samples whose respective transmittances do not vary with wavelength, which do not reflect highly, which will not displace the beam, and for which the transmittances can be independently determined with high accuracy. No such glasses or other suitable materials are available, however. Reflection samples are not good for such purpose because the directional reflectances are affected by the geometrical irradiating and receiving conditions. The use of thin metallic films does not seem advisable from the standpoints of permanence and effect of multiple reflections.

However, on visual spectrophotometers such a check is possible by means of rapidly rotating sectors. If such sectors are properly made, the angle of the total opening relative to 360° —that is, the effective transmittance of the sector—can be measured on a circular dividing engine with uncertainties only in the fifth decimal place. Of course, the use of such rotating sectors is based on the validity of Talbot's law. Throughout the spectrum, no certain deviations from this relation are known, however. In addition to published data bearing on this [104, 35] are extensive unpublished data at the National Bureau of Standards obtained over a period of many years on the König-Martens spectrophotometer. These data, obtained with sectors having transmittances over the range from 0.80 to 0.009, approximately, but principally at 0.50 and 0.10, prove both the validity of Talbot's law at the various wavelengths and the reliability of the instrument over most of the photometric scale, or else there is a remarkable balancing of errors. (The only consistent deviations are near the extinction points and are caused by stray unpolarized light, as explained in the next section.)

On photoelectric and photographic spectrophotometers similar checking of the photometric scale by rotating sectors is in general not feasible for one reason or another. Accordingly, shortly after the advent of commercial photoelectric spectrophotometers, the National Bureau of Standards instituted the service of issuing glass standards of spectral transmittance [105, 33]. To date, over 140 of these filters have been issued with accompanying certificates.

The spectral transmittances of these filters are approximately as shown in figure 28. A single filter thus covers a considerable part of the transmittance scale. In one sense these may be considered inferior to strictly neutral filters in that a deviation from the true value may be due to causes other than inaccuracy of the photo-

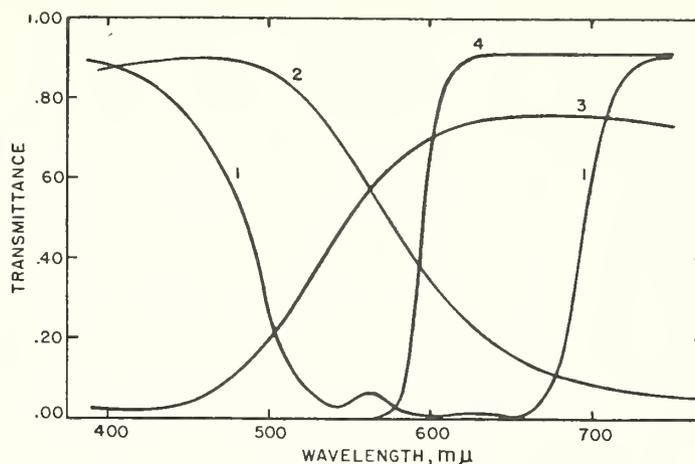


FIGURE 28. Typical spectral transmittance curves of glass filters issued by the National Bureau of Standards for use in checking the photometric scale of spectrophotometers.

Curves: 1—cobalt blue; 2—copper green; 3—carbon yellow; 4—selenium orange.

metric scale. On the other hand, they are superior to the neutral filters in detecting errors resulting from stray energy, wide slits, and wavelength inaccuracies.

A similar check of the photometric scale can be made by means of the standard solutions, whose spectral transmittancies are given in section VII-2. An additional means of checking the scale is afforded by measuring the transmittancy of solutions in different thicknesses or, if Beer's law is known to be followed, at different concentrations in the same thickness. The relation, from the equations of section II-2, is

$$-\log_{10} T_s/bc = a_s$$

If, at each wavelength tested, a_s is found to be constant at the various thicknesses and/or concentrations, in all probability the photometric scale of the instrument is accurate.

3. Stray Energy

Stray energy is often a source of important error in spectrophotometry and is produced by a variety of causes. Some of these causes are as follows:

1. The stray energy emerging from the exit slit along with the spectral energy may be a general admixture of energy of all wavelengths emitted by the source and transmitted by the spectrophotometer. It may be caused by dust, scratches, or other optical imperfections on the prism, lenses, mirrors, or other optical parts of the instrument.

2. The stray energy may be reflected from the insides of collimator tubes, the edges of diaphragms, or from other places. This may be a general admixture of all wavelengths or may be

more or less localized in some other region of the spectrum than that being measured.

3. On a polarization spectrophotometer the stray energy may be manifested as unpolarized energy, and may therefore not be affected by rotation of the nicol or other device by means of which the photometric value is obtained. This may produce relatively large errors near the extinction points.

4. In a grating spectrophotometer, stray energy may come from orders of the spectrum other than that nominally used.

5. If the instrument or detector is not properly shielded, stray radiant energy from the room in which the spectrophotometer is used may cause error.

The spectrophotometric ratio measured is, in general, given by the following relations:

$$T_{\lambda+x} = \frac{mI_{\lambda} + nI_x}{mI_{o\lambda} + nI_{ox}}$$

where I and I_o have the usual significance, λ and x refer to the spectral and the stray energy, respectively, and m and n are weighting factors dependent on the spectral sensitivity of the detector and the spectral energy of the source. Also let

$$T_{\lambda} = I_{\lambda}/I_{o\lambda}$$

and

$$T_x = I_x/I_{ox}$$

Then the above equation can be rearranged to give

$$T_{\lambda} = T_{\lambda+x} + \frac{nI_{ox}(T_{\lambda} - T_x)}{mI_{o\lambda} + nI_{ox}}$$

This shows that the correct transmittance, T_{λ} , may be greater or less than the measured ratio, depending on whether the transmittance of the sample for the spectral energy is greater or less than its transmittance for the stray energy. The error will be zero if $T_{\lambda} = T_x$ or if n or I_{ox} is zero.

The proportion of nI_{ox} to $mI_{o\lambda} + nI_{ox}$ has been evaluated in certain cases [1, 78]. On a visual spectrophotometer it increases rapidly in the end regions of the spectrum and becomes 100 percent in the ultraviolet and infrared. On the Beckman spectrophotometer with present type of phototube, it becomes nearly or wholly 100 percent above 1,200 $m\mu$. The most difficult part of the ratio to evaluate is often the quantity T_x , that is, the fraction of the stray energy transmitted by the particular filter being measured.

However, a little study of the relation will usually give one an idea of whether or not the stray energy is of importance with any particular sample and wavelength. For example, suppose

that a filter of the selenium-orange type, figure 28, is being measured at 560 $m\mu$ (where T_{λ} is in reality zero) with a photoelectric single-dispersion spectrophotometer containing a phototube highly sensitive to the red and infrared where the transmittance of the filter is about 0.9. The value of $nI_{ox}/(mI_{o\lambda} + nI_{ox})$ at 560 $m\mu$ might well be equal to 0.02. By inserting values in the above equations, $T_{\lambda+x}$ is found to be of the order of 0.018, an apparent transmittance of 1.8 percent. This type of error often occurs at various wavelengths in measuring this type of glass.

Two methods are commonly used to eliminate stray energy from causes 1, 2, and 4 above.

1. The stray energy transmitted through the exit slit may be dispersed by means of a second spectrometer so that if the first instrument transmitted 2 percent of stray energy at any particular wavelength, the second similar instrument will probably transmit about 0.02×0.02 , or roughly only 0.04 percent. This scheme has been used in certain photoelectric spectrophotometers, as noted in section V-3.

2. With a single-dispersion instrument the stray energy may be reduced by an appropriate filter placed at some suitable location in the optical path, which will transmit freely at the wavelengths at which it is used but absorb strongly at all other wavelengths to which the detector is highly sensitive. They are usually not needed in the regions of high response of the receiver but should usually be used when the spectral response becomes less than 10 or 15 percent of the maximum. Even in regions of fairly high response they may be needed when measuring the low transmittance of a filter that has high transmittance in other spectral regions. The subject is considered in greater detail in various publications [1, 5, 6, 78].

In the use of stray-energy filters, care should be taken to avoid the introduction of multiple-reflection errors. This can usually be effected by mounting the filter so that its surfaces are not quite at right angles to the axis of the beam.

The effect of stray unpolarized light, cause 3 above, is avoided on the König-Martens visual spectrophotometer by not taking measurements too near the extinction points (0° and 90°). When low transmittances are measured, rotating sectors are placed in the blank beam, as already explained, bringing the match points back towards the center of the scale.

On the Hilger Industrial and Research Spectrophotometer [3, 59], stray unpolarized light is reduced to negligible proportions by the second pair of nicols.

The effect of unpolarized energy on the General Electric recording spectrophotometer has been discussed by Pineo [68].

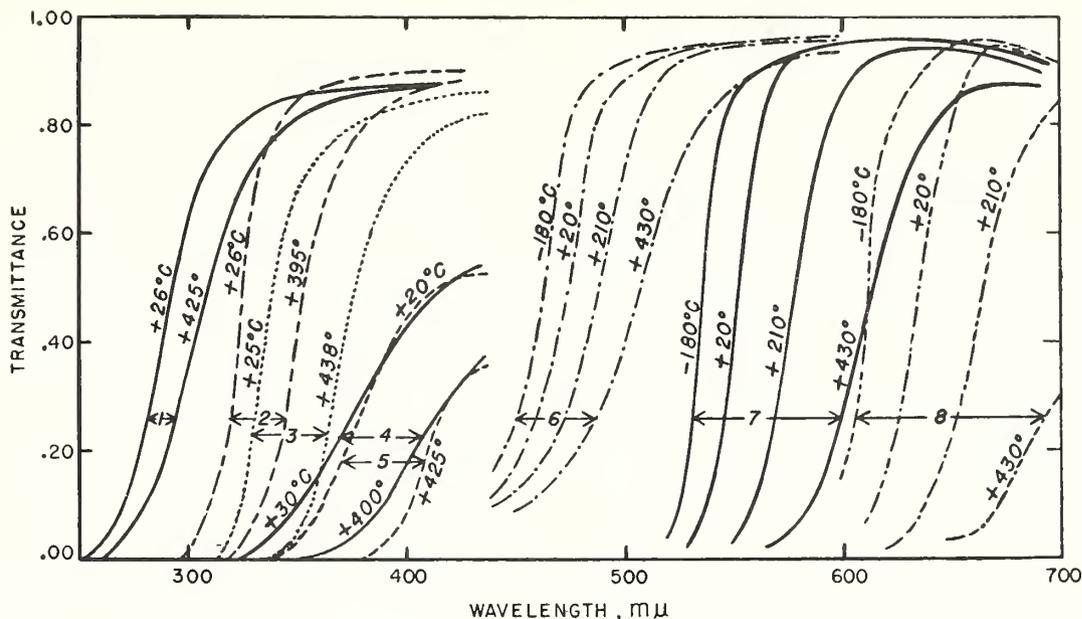


FIGURE 29. *Effect of temperature on the spectral transmittance (or internal transmittance) of certain glasses.*

Curves 1, 2, and 3 are for certain clear glasses, curves 4 and 5 for certain amber glasses, curves 6 for a Noviol yellow glass, curves 7 and 8 for selenium orange and red glasses. Curves 1 to 5 from Meyer [109], curves 6 to 8 from Gibson [107].

4. Temperature Effects

The effects of temperature on the spectral transmission characteristics of many kinds of glasses and other materials have often been studied and are in many cases well-known [106, 107, 108, 46, 109, 110]. Some typical examples are given in figures 29 and 30.

It can readily be seen that with certain samples at certain wavelengths the effect is so great that the transmittance is measurably affected by room temperature variations or by heating of the sample

in the instrument. On the same samples at other wavelengths and on other samples, the effect is immeasurably small unless studied over large temperature ranges. The effect is usually, though not always, small in the infrared, at least for glasses.

Some generalizations can be made. The transmittance at any wavelength usually decreases as the temperature is increased, provided the spectral transmittance is increasing with wavelength; and the greater the slope of the transmittance curve, the greater the temperature effect usually is. If

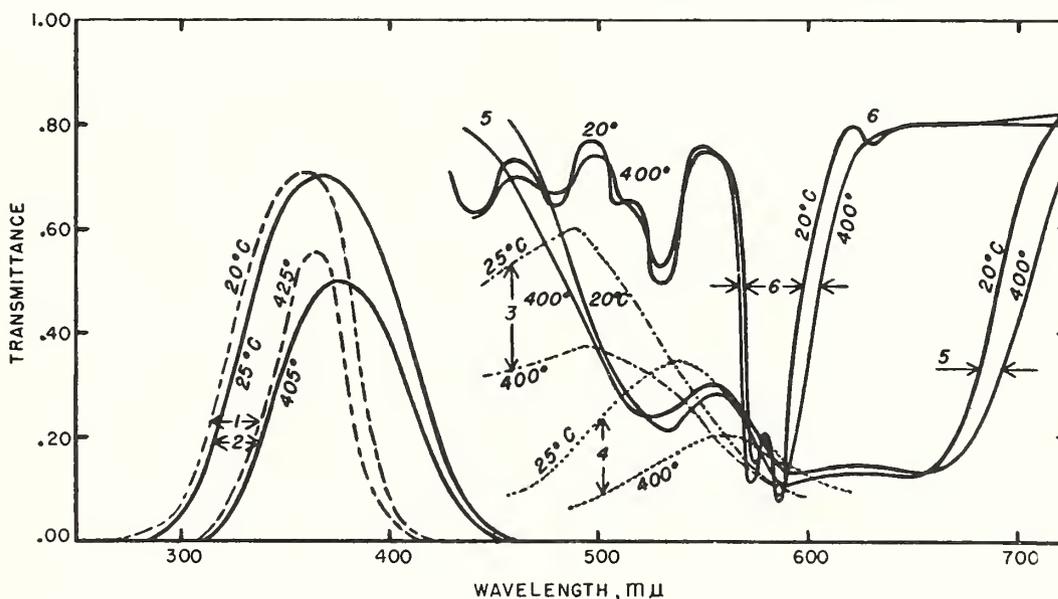


FIGURE 30. *Effect of temperature on the spectral transmittance of certain glasses.*

Curves 1 and 2 are for certain violet or blue-violet glasses, curves 3 for a copper green glass, curves 4 for a chrome green glass, curves 5 for a cobalt blue glass, curves 6 for a didymium glass. Curves 1 and 2 from Meyer [109], curves 3 and 4 from Gibbs [106], curves 5 and 6 from Holland and Turner [110].

the spectral transmittance curve is not varying much with wavelength the temperature effect is usually very small. If the spectral transmittance is decreasing from shorter to longer wavelengths, the effect is usually small and often of opposite sign.

The effects just described are elastic in that the sample always returns to a specified transmittance at a specified temperature. Corresponding changes in the spectral reflectance curves of certain samples have been observed at the National Bureau of Standards, but no quantitative study of the effect has been made.

Differing from the above are the permanent changes in the spectral reflectances of various materials (papers, dyed textiles, etc.) under strong and continued irradiation and the "solarization" of glasses when exposed to intense ultraviolet radiant flux [111], but such effects are not within the scope of this paper.

5. Fluorescence

The accurate measurement of the spectral transmittance or reflectance of strongly fluorescent materials has not yet been satisfactorily treated from the standpoint of instrument design, especially for reflecting materials. In the various types of instruments illustrated above, it may have been noted that, in some designs, the samples were mounted between the source of radiant energy and the spectrophotometer. In other designs, the samples are mounted between the instrument and the receptor. In the first type, the full flood of undispersed radiant energy is incident upon the sample and may cause temporary or permanent changes in the sample during the measurements. In the second design, the radiant energy is dispersed in the instrument and energy from only the small spectral region isolated by the exit slit falls upon the sample, thus, in general, avoiding

the excessive temperature or fading effects of designs of the first type. For nonfluorescent samples the two designs are inherently equivalent and will give the same results under otherwise identical conditions.

With strongly fluorescent samples this may not be true, however. Both methods may produce erroneous results, but the second type of instrument is subject to much the greater error. In evaluating this error, one must define what is to be understood as the correct result. Where the effect has been most notable are in instances in which it was desired to derive colorimetric specifications of strongly fluorescent reflecting materials from the spectrophotometric data [112]. In such cases the correct result is considered to be a spectral curve or set of data consisting of (1) the true spectral directional reflectance, to which has been added (2) the spectral distribution of the fluorescent energy when excited by a source having the same spectral distribution of energy (ultraviolet, visible, infrared), as is used in the specified visual examination of the sample. This latter restriction is essential because the amount and distribution of the fluorescent energy may vary considerably with different irradiants.

If the colorimetric specification of the fluorescent sample is desired for an incandescent irradiant, a spectrophotometer of the first type (such as the Gaertner visual) can give a correct result. If it is desired for artificial daylight a correct result can be obtained if the artificial daylight is used as source in place of the bare incandescent light. If it is desired for natural daylight, it cannot be accurately obtained unless the illumination sphere be discarded and the sample and standard be actually irradiated by natural daylight. (Natural daylight and artificial daylight may differ considerably in the ultraviolet region.)

On spectrophotometers of the second type the fluorescent sample is irradiated by the continually

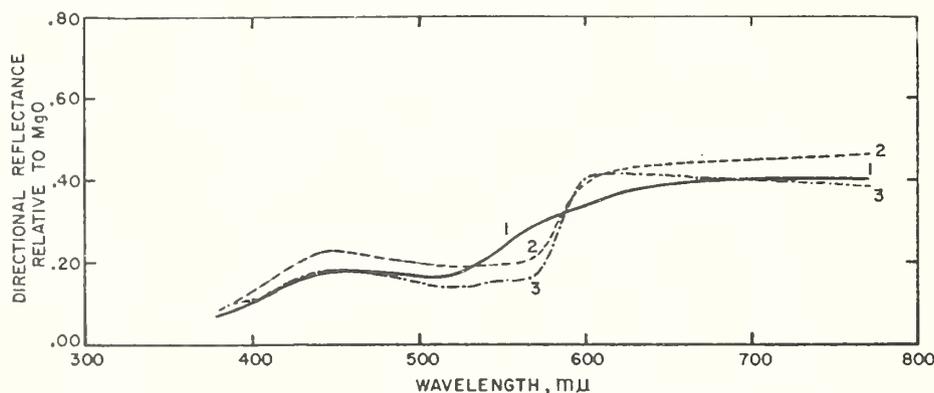


FIGURE 31. Effect of fluorescence on the measured values of spectral directional reflectance.

The sample was a pink phenolformaldehyde that fluoresced strongly to green and yellow energy but weakly or none at all to blue, violet, and ultraviolet energy. Curve 1 ($R_{\theta, d}$) was obtained on a photoelectric spectrophotometer in which the energy was spectrally dispersed before incidence on the sample; the high values from 500 to 580 $m\mu$ result from the reddish fluorescence excited by the green and yellow energy. Curves 2 ($R_{\theta, e}$) and 3 ($R_{45, e}$) were obtained with visual spectrophotometers (data extrapolated at ends) in which the sample is irradiated directly by the source, and the fluorescence excited is spectrally dispersed before measurement. Colorimetric comparison of the sample with nonfluorescent standards of nearly the same color showed that colorimetric values derived from curve 1 were much more in error than those derived from curves 2 or 3.

changing region of the spectrum isolated by the exit slit, and is therefore subject to widely varying irradiances during the measurements, probably none of which are of any particular interest in the actual use of the fluorescent sample. The magnitude of such an error is shown in figure 31, these curves relating to the sample for which the effect was first noticed [113].

Explanation of the effect is readily seen. The sample in figure 31 exhibited a strong reddish fluorescence when excited by energy in the green and yellow-green parts of the spectrum. The sample was run on the General Electric recording spectrophotometer, which uses a phototube that responds freely to the red and near infrared. When the sample is irradiated by energy in the green and yellow parts of the spectrum (500 to 580 $m\mu$), the strong reddish fluorescent energy evoked falls directly (undispersed) upon the red-sensitive phototube and is added to that truly reflected at the respective wavelengths in the green or yellow. The resulting reflectance curve, No. 1 of figure 31, is thus erroneously high in the green region. The same type of error occurs on the Beckman spectrophotometer, although the choice of two phototubes for the measurement can reduce the error somewhat in some cases.

The effect is much greater in reflection measurements of diffusing materials than in transmission measurements of nondiffusing samples. In the latter the fluorescent energy is emitted in all directions, only a small fraction being incident upon the phototube, and this can be reduced by moving the sample as far as possible from the phototube, or by a judicious use of filters. In reflection measurements of diffusing specimens, the proportion (geometrically) of the fluorescent energy accepted for measurement is the same as that of the diffusely reflected energy.

6. Slit Width

The magnitudes of slit-width errors depend primarily upon two things: (1) the effective width of the entrance and exit slits with a continuous spectrum source, and (2) the curvature (not the slope) of the spectral distributions under consideration. The principle can readily be seen from the simple illustrations in figure 32. Let a and b be the edges of a slit of width, $2\Delta\lambda$, and let λ be the nominal wavelength at which the spectrophotometer is set. Furthermore, let c and d represent two energy distributions that are being evaluated for wavelength, λ , by a nonselective detector such as a thermopile. This will integrate the energy transmitted by the slit over the range $2\Delta\lambda$, and the value obtained will be taken as for λ . It is apparent that for distribution c a value of c' is obtained, which is the correct value. For distribution d , on the other hand, a

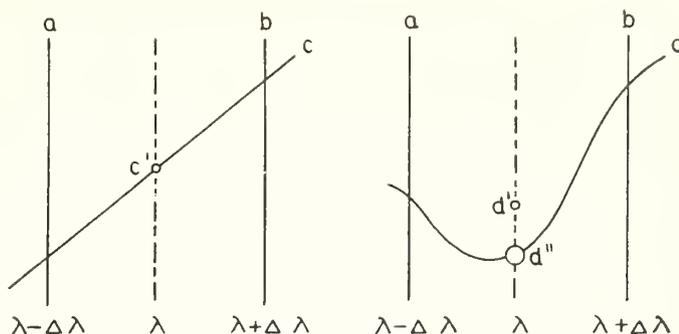


FIGURE 32. Showing that slit-width errors depend on the curvature, not the slope, of the distribution curve across the slit.

The slit width extends from a to b , $\lambda \pm \Delta\lambda$. At the left the measured and true values coincide at c' . At the right the measured value is at d' , the true value at d'' . See also the effect of slit width in figure 8.

value of d' is obtained, which is different from the true value d'' .

The effect of slit widths is easily shown on a recording spectrophotometer with a didymium glass. Two such curves are recorded in figure 8, one for 4- $m\mu$ slits and one for 8- $m\mu$ slits. Note that the differences are greatest where the curvatures are sharpest, and that at certain places on the steep sides of the bands (where the curves cross) the differences are zero.

Mathematical expressions to evaluate slit-width errors have been derived by various investigators. Among the first were Runge [114], Paschen [115], and Hyde [116]; these are discussed on pages 182 to 188 (by Weniger) and 349 to 351 of Measurement of Radiant Energy [6]. Equation 80, page 349, from that book is useful for spectrophotometry, namely,

$$T = \frac{\phi_T(\theta)}{\phi(\theta)} = \frac{F_T(\theta) - K\Delta^2 F_T(\theta) + L\Delta^4 F_T(\theta) + \dots}{F(\theta) - K\Delta^2 F(\theta) + L\Delta^4 F(\theta) + \dots},$$

where T is the correct transmittance or reflectance of the sample at the wavelength under consideration, θ is the angle of deviation at this wavelength, $\phi_T(\theta)$ and $\phi(\theta)$ are the relative intensities of the pure spectra with and without the sample in the beam, $F_T(\theta)$ and $F(\theta)$ represent the relative intensities, as evaluated by the detector, of the corresponding impure spectra resulting from the use of finite slit widths, the Δ^2 and Δ^4 functions relate to the second and fourth derivatives of the spectral distributions, and K and L are constants, L much smaller than K .

Note the absence of functions dependent on the first derivatives and the major importance of the second derivatives in this relation, consistent with figures 8 and 32. Hyde's paper [116] should be consulted for a more complete presentation of the relations, including the definitions of the Δ^2 and Δ^4 functions and indications of how to apply the corrections.

For a given sum of entrance and exit slits, the

errors or corrections are least when the two slit widths are equal. For this case $K=1/12$ and $L=1/90$.

The obvious difficulty or bother of applying this slit-width correction, which at best is only an approximation, illustrates the desirability of keeping the slits sufficiently narrow that correction is unnecessary for the purpose at hand. It also illustrates the value of having auxiliary sources, such as the Hg arc, for use on a spectrophotometer, eliminating any possibility for slit-width errors at the wavelengths available.

More recent investigators of slit-width errors include Hogness, Zehele, and Sidwell [78], Van den Akker [117], Buc and Stearns [70], and Hardy and Young [118]. The most general case of a nonsymmetrical "pass band" is treated in this last paper, and the authors derive a formula including a first-order correction term that may be considered as a wavelength correction and that becomes of importance with wide slits and an unsymmetrical pass band, as illustrated by Van den Akker. Hardy and Young also suggest a novel procedure for determining the second-order correction, and propose a definition for the effective slit width of a spectrophotometer.

7. Miscellaneous

Various other errors are possible in spectrophotometric work, some related to particular types of measurements, some related to particular instruments.

One that is often present is a multiple-reflection error, occurring especially when the transmittance of a sample is measured relative to the blank beam. The introduction of the sample being measured in effect changes the value of I_1 in

the defining relation

$$T = I_2/I_1$$

This can happen in various ways. The energy reflected from the sample, which should not reach the detector, may have a small part of it redirected toward the detector by another optical part. The energy reflected from a shiny slit head may have a small part of it redirected by the sample into the slit (sample between source and slit) or the energy reflected from a phototube may have a small part of it redirected by the sample back onto the phototube (sample between slit and phototube). The error can usually be discovered, if present, by measuring the transmittance of a thin piece of crown glass placed first in its normal position at right angles to the beam and then at some small angle from this position. In this second position the reflected energy is directed out of the beam without affecting the true transmittance appreciably. The remedy will then depend on circumstances but should be such that the sample being measured can be inserted at right angles to the beam without introducing the error.

In polarization instruments the extinction points should be carefully checked and adjusted if in error. In direct-reading instruments the 100-percent point or curve should be checked (and evaluated if not precisely 100.0) to insure that the substitution method is being in effect employed.

If the transmittance sample is wedge-shaped, lens-shaped, or merely thick (in a converging or diverging beam), its introduction into the beam may in effect change the I_1 reading. The possibility and magnitude of this error depends on the design of the instrument.

VII. Standard Data

Standards of spectral transmittance or transmittancy are of importance in checking the photometric scale of spectrophotometers, as already explained, and such standards are useful whether the instruments are used for spectral transmission measurements or for measurements of spectral directional reflectance or spectral energy distribution. In general, no further standards are needed for transmission measurements, since practically all measurements are made on nondiffusing materials relative to the blank beam or to a solvent cell.

Reference or "100-percent" standards are needed, however, for spectral reflection or spectral energy measurements. While it might be possible to design an "absolute" spectroreflectometer for diffusely reflecting materials, such has not been done, and a diffusely reflecting sample or standard of known spectral directional reflectance is accord-

ingly necessary. For spectral energy measurements a standard of known spectral distribution is likewise necessary if values for the unknown source are to be evaluated from the spectrophotometric ratio.

1. Spectral Energy Standards

A suitable standard of spectral energy distribution through the visible spectrum is the incandescent-filament electric lamp operated at a specified color temperature. The color temperature of such a source is defined as the temperature at which the Planckian radiator or "black body" would have to be operated to give the same color as the incandescent-filament lamp. When the incandescent lamp is thus operated at a specified color temperature, the relative spectral energy distribution through the visible spectrum is given

by Planck's equation to a considerable degree of accuracy. This relation is [6]

$$I_{\lambda} = \frac{AC_1\lambda^{-5}}{C_2 e^{\lambda\theta} - 1}$$

in which

I_{λ} = spectral radiant intensity (or some quantity proportional thereto),
 e = base of natural logs,
 θ = absolute temperature in degrees Kelvin,

A , C_1 , and C_2 = constants.

In the visible spectrum differences between the measured relative energy distribution from an incandescent-filament lamp operated at a specified color temperature and the relative energy distribution derived from Planck's equation are so slight [47, 48] as to border on the uncertainties of measurement, and it has become customary therefore to accept incandescent-filament lamps as adequate standards of spectral energy distribution through the visible spectrum. Numerous publications have been issued in which these distributions have been computed and tabulated so that one does not need to derive the values for himself [44, 45, 46].

In table 3 is shown the relative energy distribution of the Planckian radiator at 2,848° K ($C_2 = 14,350$ micron degrees). The same distribution is given by 2,842° K ($C_2 = 14,320$) on the former international temperature scale, and by 2,854° K ($C_2 = 14,380$) on the new international tempera-

TABLE 3. *Relative energy distribution of Planckian (black body) radiator at 2,848° K ($C_2 = 14,350$ micron degrees), ICI Standard Illuminant A*

The same distribution is given by 2,842° K ($C_2 = 14,320$) on the former international temperature scale, and by 2,854° K ($C_2 = 14,380$) on the new international temperature scale

Wavelength	Relative energy distribution	Wavelength	Relative energy distribution
380.....	9.79	600.....	129.04
90.....	12.09	10.....	136.34
400.....	14.71	20.....	143.62
10.....	17.68	30.....	150.83
20.....	21.00	40.....	157.98
30.....	24.67	650.....	165.03
40.....	28.79	60.....	171.96
450.....	33.09	70.....	178.77
60.....	37.82	80.....	185.43
70.....	42.87	90.....	191.93
80.....	48.25	700.....	198.26
90.....	53.91	10.....	204.41
500.....	59.86	20.....	210.36
10.....	66.05	30.....	216.12
20.....	72.50	40.....	221.66
30.....	79.13	750.....	227.00
40.....	85.95	60.....	232.11
550.....	92.91	70.....	237.01
60.....	100.00	80.....	241.67
70.....	107.18		
80.....	114.44		
90.....	121.73		

ture scale. This energy distribution was recommended in 1931 by the International Commission on Illumination as one of three standard illuminants for colorimetry and has come to be known as ICI standard illuminant A. It is perhaps as suitable as any other as a standard of spectral energy distribution. At lower color temperatures the radiant intensities become too low, particularly at the shorter wavelengths, to serve as a suitable standard. At higher color temperatures the lamp ages more and more rapidly, and the assumed spectral energy distribution may become inaccurate.

Incandescent-filament lamp standards of color temperature are issued by the National Bureau of Standards at 2,848° K and at other color temperatures.

2. Spectral Transmission Standards

Reference has already been made (section VI-2 and fig. 28) to the glass standards of spectral transmittance issued by the National Bureau of Standards for use in checking the photometric scale of spectrophotometers. These standards have a high degree of permanence if the surfaces are kept undamaged and if they are not exposed to sunlight or other excessive radiant flux. However, they are not precisely reproducible from specification and each glass must be independently calibrated before issuance. Accordingly, no transmittance values for the glasses are tabulated.

However, the spectral transmittancies of certain solutions have been measured with sufficient care, and the solutions themselves are sufficiently stable, that they may be accepted as suitable standards for checking the photometric scale of spectrophotometers. These are:

1. The aqueous solutions of copper sulfate and cobalt ammonium sulfate used in the series of filters developed at the National Bureau of Standards for reproducing the colors of sunlight and daylight and for the determination of color temperatures [46].

The published values for absorbancy and transmittancy are given in tables 4 and 5, together with the composition and certain other pertinent information. Many additional details are given in M114 [46] including the changes in absorbancy with temperature. Both solutions obey Beer's law over a considerable range. The values given for the eight Hg and He wavelengths are considered the most reliable, with an uncertainty in A , not exceeding 0.001 for the particular chemicals used. Spectrophotometric reproducibility of the chemicals is also considered in the paper. By increasing the thickness or concentration, a wide range of the photometric scale can be covered, except at the shorter wavelengths.

TABLE 4. Values of spectral absorbancy, A_s , and transmittancy, T_s , for standard copper sulfate solution as specified, unfiltered, thickness 10.00 mm, temperature 25° C, having the following composition:

Copper sulfate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$)^a 20.000 g
 Sulphuric acid (specific gravity 1.835) 10.0 ml
 Water (distilled) to make 1,000 ml

Wavelength	A_s	T_s ^b	Wavelength	A_s	T_s ^b
$m\mu$			$m\mu$		
350	0.0090	0.979	600	0.0680	0.855
60	.0063	.986	10	.0855	.816
70	.0046	.989	20	.1125	.772
80	.0035	.992	30	.143	.719
90	.0028	.994	40	.180	.661
400	.0023	.995	650	.224	.597
10	.0019	.996	60	.274	.532
20	.0016	.996	70	.332	.466
30	.0014	.997	80	.392	.406
40	.0012	.997	90	.459	.348
450	.0011	.997	700	.527	.297
60	.0011	.997	10	.592	.256
70	.0012	.997	20	.656	.221
80	.0014	.997	30	.715	.193
90	.0018	.996	40	.768	.171
500	.0026	.994	750	.817	.152
10	.0038	.991	Hg 404.7	.0021	.995
20	.0055	.987	Hg 435.8	.0013	.997
30	.0079	.982	Hg 491.6	.0019	.996
40	.0111	.975	He 501.6	.0028	.994
550	.0155	.965	Hg 546.1	.0135	.969
60	.0216	.951	Hg 578.0	.0368	.919
70	.0292	.935	He 587.6	.0487	.894
80	.0390	.914	He 667.8	.319	.450
90	.0518	.888			

^a Analysis showed the copper sulfate to have 99.7 percent of the theoretical copper content.

^b Values of T_s are derived from values of A_s . $A_s = -\log_{10} T_s$.

2. An aqueous solution of potassium chromate, K_2CrO_4 (0.04g/l) in 0.05N KOH. This solution has been studied by many investigators here and abroad, for example, Hogness, Zscheile, and Sidwell [78], and is considered one of the most suitable as a standard of spectral transmittancy and absorbancy in the ultraviolet. Its absorption in the violet is also useful because the copper and cobalt solutions have too little absorption in this region to be of much value. Accordingly, an extensive series of measurements was initiated in 1940 by Geraldine Walker Haupt of the Photometry and Colorimetry Section of the National Bureau of Standards to obtain standard spectral transmittancy and absorbancy data for a specified potassium chromate solution of known stability. This work was interrupted before completion, but sufficient data had been obtained that a tentative set of spectral transmittancy values could be adopted, and Mrs. Haupt has made these tentative values available for use herein. They are given in table 6. It is expected that this work will be completed and final values published. Pending this, the following details are of interest and importance in connection with the use of table 6.

TABLE 5. Values of spectral absorbancy, A_s , and transmittancy, T_s , for standard cobalt ammonium sulphate solution, unfiltered, thickness 10.00 mm, temperature 25° C, having the following composition:^a

Cobalt ammonium sulphate ($\text{CoSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$)^b 14.481 g
 Sulphuric acid (specific gravity 1.835) 10.0 ml
 Water (distilled) to make 1,000 ml

Wavelength	A_s	T_s	Wavelength	A_s	T_s
$m\mu$			$m\mu$		
350	0.0038	0.991	630	0.0137	0.969
60	.0040	.991	10	.0124	.972
70	.0050	.989	20	.0115	.974
80	.0065	.985	30	.0112	.975
90	.0088	.980	40	.0110	.975
400	.0125	.972	650	.0105	.976
10	.0168	.962	60	.0097	.978
20	.0224	.950	70	.0087	.980
30	.0340	.925	80	.0076	.983
40	.0522	.887	90	.0066	.985
450	.0773	.837	700	.0054	.988
60	.1031	.789	10	.0046	.989
70	.1213	.756	20	.0038	.991
80	.1349	.733	30	.0032	.993
90	.1472	.713	40	.0030	.993
500	.1635	.686	750	.0028	.994
10	.1742	.670	Hg 404.7	.0144	.967
20	.1689	.678	Hg 435.8	.0437	.904
30	.1452	.716	Hg 491.6	.1497	.708
40	.1113	.774	He 501.6	.1661	.682
550	.0775	.837	Hg 546.1	.0901	.813
60	.0496	.892	Hg 578.0	.0219	.951
70	.0308	.932	He 587.6	.0167	.962
80	.0207	.953	He 667.8	.0089	.980
90	.0158	.964			

^a These data apply accurately also from 400 to 750 $m\mu$ to a similar solution made up with 10.3 g of cobalt sulphate ($\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$).

^b Chemical analysis showed a cobalt (plus nickel) content of 100.0 percent of the theoretical, the ratio of nickel to cobalt (metals) being 1 to 200.

All of the potassium chromate solutions were prepared by S. Claibough of the National Bureau of Standards, Chemistry Division, under the direction of E. Wichers. The data of table 6 were obtained from solutions prepared in the following ways:

- (1) A solution of K_2CrO_4 stock material, reagent grade, 0.0400 g per liter, in 0.05N KOH.
- (2) A solution of K_2CrO_4 of the same concentration and alkalinity as (1) but prepared from 0.0303 g of $\text{K}_2\text{Cr}_2\text{O}_7$ which when neutralized gave 0.0400 g of K_2CrO_4 per liter.

Both solutions were found to remain stable (in transmittancy) for the two years during which the measurements were being made, if stored in ordinary storeroom glass bottles. "Flaking" may occur during this time and any sediment should be allowed to settle to the bottom of the bottle. It is recommended that alkali-resistant ware, now available, be used for storing the solutions.

In the values of T_s given in table 6 there is considerable uncertainty in the third decimal where three significant figures are used. Where but two significant figures are used there is some uncertainty in the last figure.

TABLE 6. Tentative values of spectral transmittancy, T_s , and absorbancy, A_s , for standard potassium chromate solution, unfiltered, thickness 10.00 mm, room temperatures (usually close to 25° C), having the following composition:

0.0400 g per liter of potassium chromate (K_2CrO_4) in 0.05 normal potassium hydroxide solution ^a

Wavelength <i>mμ</i>	T_s ^b	A_s ^c	Wavelength <i>mμ</i>	T_s ^b	A_s ^c
210	0.000		350	.28	.5528
15	.037	1.4318	55	.202	.6946
20	.35	.4559	60	.148	.8297
225	.60	.2218	65	.115	.9393
30	.68	.1675	70	.102	.9914
35	.62	.2076	375	.103	.9872
40	.509	.2933	80	.118	.9281
45	.408	.3893	85	.152	.8182
250	.319	.4962	90	.207	.6840
55	.268	.5719	95	.30	.5229
60	.232	.6345	400	.41	.3872
65	.201	.6968	404.7	.52	.2840
70	.180	.7447	410	.635	.1972
275	.173	.7620	20	.748	.1261
80	.189	.7235	30	.824	.0841
85	.254	.5952	435.8	.861	.0650
90	.372	.4295	40	.884	.0535
95	.527	.2782	450	.928	.0325
300	.705	.1518	60	.961	.0173
05	.83	.0809	70	.981	.0083
10	.90	.0458	80	.992	.0035
15	.905	.0434	90	.998	.0009
20	.867	.0520	560	1.000	.0000
325	.810	.0915			
30	.715	.1457			
35	.605	.2182			
40	.485	.3143			
45	.38	.4202			

^a This solution of potassium hydroxide can be prepared with sufficient accuracy by dissolving 3.3 g of potassium hydroxide sticks (85% KOH) of reagent quality in sufficient distilled water to make 1 liter.

^b Tentative values. See text.

^c These values of A_s derived from the values of T_s .

3. Spectral Reflection Standards

As a fundamental standard of spectral directional reflectance nothing has as yet been found

more suitable than freshly prepared magnesium oxide. Its (total) luminous reflectance is high, 0.97 or 0.98, and nothing has been found of certainly higher reflectance. Its directional luminous reflectance, $R_{0,45}$, is 1.00, and its spectral selectivity throughout the visible spectrum appears to be less than 1 percent. These data are based on work by Priest [119], McNicholas [24], and Preston [120], and are summarized in National Bureau of Standards Letter Circular LC547 [121]. In recent publications, Benford and others [122, 123] give the spectral reflectance of MgO and $MgCO_3$ in the ultraviolet and visible spectrum obtaining a value of about 0.99 for the luminous reflectance of MgO, with a selectivity of about 2 percent in the visible spectrum.

Other agencies besides the Bureau have also recommended MgO for the same or similar purpose. In 1931, the International Commission on Illumination adopted a resolution which may be translated as follows: "For the colorimetric measurement of opaque materials the luminance of the specimen studied ought to be expressed as a function of the luminance of a surface of the oxide of magnesium considered under the same conditions of illumination and observation" [124]. In 1944, this method was incorporated in ASTM Standard Method of test for spectral characteristics and color of objects and materials [125].

While these characteristics make fresh MgO an excellent fundamental standard, it has other characteristics that are undesirable and that make the calibration and use of a secondary working standard a very advisable procedure. An MgO surface is extremely fragile and thus is not very suitable for continued handling. A more serious defect is that its spectral reflectance may change perceptibly within a short time after preparation.

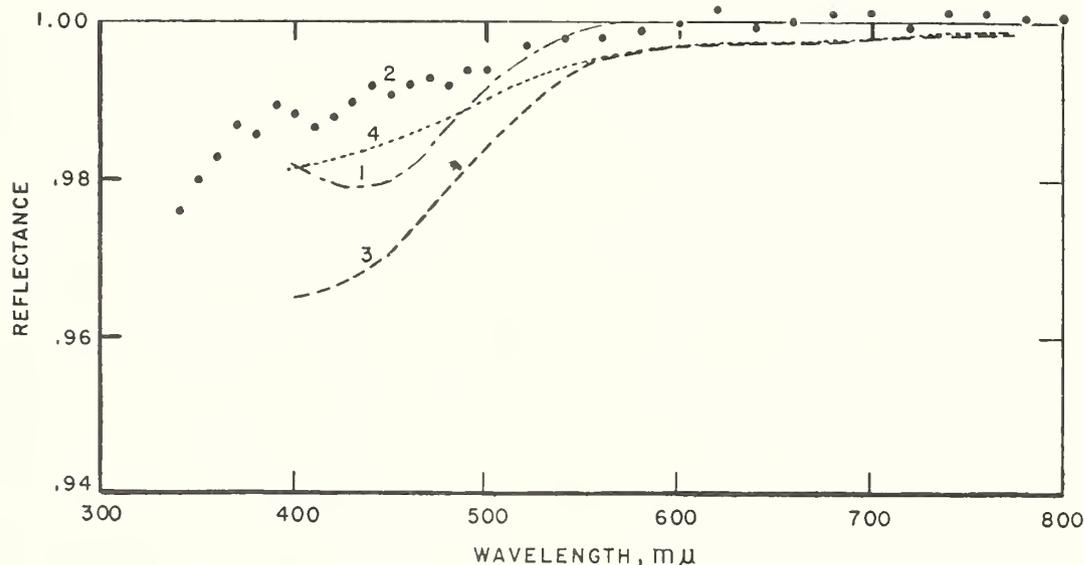


FIGURE 33. Effect of age, and probably other factors, on the spectral reflectance of magnesium oxide.

Values are plotted relative to the reflectance of freshly prepared MgO. Curves 1, 3, and 4 were obtained with the General Electric spectrophotometer, curve 2 with the Beckman spectrophotometer. The ages of the samples were: curve 1, 2½ weeks; curve 2, 5 months; curve 3, 20 months; curve 4, 2 years.

The nature of the change is shown in figure 33; the reflectance decreases below 550 $m\mu$ as shown. Furthermore the nature and extent of the changes are variable. A third reason for use of a working standard is that slight variations in reflectance (0.1 or 0.2 percent) have been noted for different preparations of freshly prepared MgO. By calibrating the working standard against several different MgO preparations, a more representative standard is obtained than would be any single MgO surface by itself.

White structural glass by the name of Vitrolite, with polished surface, has proved the most suitable for working standards of spectral directional reflectance, although the material is not uniformly good for this purpose and must be selected with care. A considerable supply of suitable Vitrolite has been obtained by the National Bureau of Standards, and standards are now calibrated and

issued for either the General Electric recording spectrophotometer or the Beckman quartz spectrophotometer. Further information regarding these is given in NBS Letter Circular LC929, Standards for checking the calibration of spectrophotometers (200 to 1,000 $m\mu$) [126].

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STANDARD CONDITIONS FOR PRECISE PRISM REFRACTOMETRY

By Leroy W. Tilton

ABSTRACT

Air as a standard reference medium for precise refractive-index measurements is discussed with respect to the precision necessary in the control and measurement of its temperature, pressure, humidity, and carbon-dioxide content. Particular attention is given to the selection of provisional values for α , the optical temperature coefficient for air; and the preparation of accurate correction tables for the reduction of refractive-index observations to standard conditions of reference is described and exemplified.

The approximate range of temperature effects on absolute refractive index is indicated; also the range of pressure effects on liquids. A new (empirical) relation between index and density is derived from Pockels' optical data on elastically deformed glass and a basis for quantitative treatment of permissible stress-birefringence is given. For glasses, the character of annealing and the permissible degree of striation are considered; and for several media the requisite constancy in wave length of light source is determined.

Tolerance equations are given for all requirements that are quantitatively discussed and it is concluded that both precision and accuracy to within ± 2 or 3×10^{-6} can be attained in determining refractive index by the classical method of minimum deviation.

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I. INTRODUCTION

In the practice of precise refractometry a desirable degree of approximation to the ideal physical and chemical conditions of measurement is seldom realized. Although the necessity for control of some of the physical conditions is more or less well known, only a comparatively small amount of the published index data is accompanied by adequate references to the conditions under which the measurements were actually made or to the conditions which are considered as standard.¹

The tolerances for variations in air temperature, pressure, and humidity have not been explicitly and precisely evaluated, and the effects of temperature and pressure on the absolute indices of various media are not adequately known. Moreover, the question of the effect of heterogeneities in the sample has not hitherto been discussed in connection with refractive-index measurements. On the other hand, it appears that no attempt has been made to justify or to disprove the commonly accepted opinion that even a moderate amount of birefringence in a medium may make precise index measurements useless.

Certain refractive-index measurements of high precision, that are being made in the refractometric laboratory at the National Bureau of Standards, have made it necessary to determine the precision with which all of the working conditions must be controlled or, in some instances, quantitatively observed. It has seemed advisable to differentiate clearly between the effects of such conditions on the reference medium and on the sample to be measured and, wherever possible, to specify on a quantitative basis the requirements and tolerances relating to these working conditions; also, to set forth in detail a system of accurate corrections to standard reference conditions. However, when considering such matters as heterogeneity of the sample and the permissible degree of its birefringence, the treatment is necessarily limited to simple approximations.

For convenience of reference, the definitions of various symbols that are used and explained in this paper are summarized here as follows:

α \equiv temperature coefficient of expansion of air at constant pressure; also (negative) temperature coefficient of refractivity;

β \equiv pressure coefficient of volume change at constant temperature (often called compressibility), that is

$\beta = -\frac{1}{V_0} \left(\frac{dV}{dP} \right)$; also, for air, the pressure coefficient of refractivity;

C \equiv total (volume) proportion of CO_2 in atmosphere;

C', C'', C''' \equiv constants of proportionality in various equations expressing index-density relationship;

c \equiv excess of CO_2 above the normal proportion, that is, $c = C - 0.00035$;

d \equiv density;

¹ Brief historical references have been made in a former paper, BS J. Research 2, 912-915 (1929) RP64.

- E \equiv Young's modulus of elasticity;
 $\epsilon = 179 \times 10^{-6} / (\mu_0 - 1)$;
 $\gamma = 41 \times 10^{-6} / [760(\mu_0 - 1)]$;
 K \equiv modulus of volume elasticity or "bulk modulus",
 that is, $K = 1/\beta$;
 λ \equiv wave length of light;
 m \equiv mass;
 μ \equiv index of refraction of air, the general subscripts t ,
 p , v , and c referring to the temperature, pres-
 sure, humidity, and carbon-dioxide content,
 respectively, while the subscript 0 indicates
 standard conditions of 0° C and 760 mm
 pressure and D specifies a wave length of
 5893 Å;
 n \equiv relative (to air) index of refraction of a medium;
 \bar{n} \equiv absolute (or vacuum) index of refraction of a
 medium;
 P \equiv pressure of air;
 p \equiv excess in mm of Hg over 1 atmosphere of air
 pressure, that is, $p = (P - 760)$;
 (p'/v') and (q'/v') \equiv the Pockels experimental constants for certain
 glasses, v' being the velocity of light in the
 unstressed glass;
 s \equiv proportion by volume of striae in glass;
 σ \equiv Poisson's ratio;
 T and t \equiv temperatures on absolute and centigrade scales,
 respectively;
 $T_{\Delta t_a}$ \equiv tolerance in air temperature control and measure-
 ment that corresponds to an error of $\pm 1 \times 10^{-6}$
 in (relative) refractive index;
 $T_{\Delta p_a}$ \equiv tolerance in air-pressure control and measure-
 ment that corresponds to an error of $\pm 1 \times 10^{-6}$
 in (relative) refractive index;
 $T_{\Delta v}$ \equiv tolerance in absolute humidity that corresponds
 to an error of $\pm 1 \times 10^{-6}$ in (relative) refrac-
 tive index;
 $T_{\Delta c}$ \equiv tolerance in abnormal proportion (by volume) of
 CO₂ in the atmosphere that corresponds to an
 error of $\pm 1 \times 10^{-6}$ in (relative) refractive
 index;
 $T_{\Delta P}$ \equiv tolerance in hydrostatic-pressure variations cor-
 responding to $\pm 1 \times 10^{-6}$ in refractive index of
 liquid and solid media;
 $T_{\Delta n_b}$ \equiv tolerance in absolute stress-birefringence corre-
 sponding to an error of $\pm 1 \times 10^{-6}$ in refractive
 index (for the transverse ray if $n < 1.8+$);
 $T_{\Delta \lambda}$ \equiv tolerance in wave length of source corresponding
 to an error of $\pm 1 \times 10^{-6}$ in refractive index;
 V \equiv volume; and,
 v \equiv pressure of water vapor in the atmosphere in
 mm of Hg.

II. AIR AS A STANDARD MEDIUM OF REFERENCE

The great majority of all measurements involving light velocities are made while using the earth's atmosphere as the practical standard medium of immersion or reference. Since the velocity of light in air is a function of chemical composition and also of the temperature and pressure of air, it is necessary to specify CO₂, moisture content, and other factors with appropriate precision in order that the measurements may have definite value.

The composition of CO₂-free dry air has been discussed in a former paper.² Although this composition appears constant to a high degree when air is chemically analyzed, it has long been estimated that variations in the normal density of air are as large as 5 or 10 parts in 10,000 and for this reason air is no longer considered a suitable reference medium for accurate specific gravities of gases. Statistical analysis of existing data indicates, likewise, that the refractive index of air at standard conditions is not constant but varies over a range perhaps as large as $\pm 12 \times 10^{-7}$ from a general average value of 1.0002925 for $\lambda = 5893$ Å. Consequently all relative indices of refraction may be subject to an inherent uncertainty which can be estimated as

$$\Delta n = \pm 12n \times 10^{-7} \quad (1)$$

unless accompanied by reliable measurements on air itself.

In reducing relative indices to absolute values when direct measurements on air are lacking, it is probable that this uncertainty is statistically diminished by using for μ , the index of air, a value properly computed³ from dispersion data and from an equation that gives the sodium-lines index of air for 0° C and 760 mm pressure as

$$(\mu_D - 1) \times 10^7 = \mu_{S=0} - kS \quad (2)$$

where S is the relative annual sunspot number (for the year in which the relative indices were measured) as given in the Wolf-Wolfer series of sunspot observations. For use in this equation the parameters $\mu_{S=0}$ and k should be determined from the best available data on the refractive index of air for the period in question. Provisional values obtained by the writer for the whole interval 1857 to 1934 are, respectively, 2932.2 and 0.148; whereas for the limited interval 1912 to 1934, during which a number of very important observations have been made, the corresponding values of the parameters are 2927.5 and 0.079. With either of these sets of values for the parameters, however, the probable errors of estimates of index interpolated by use of equation 2 are large, namely ± 5 and $\pm 3 \times 10^{-7}$, respectively, and it is readily seen that for certain years within the interval 1912 to 1934 two estimates can actually differ by 5×10^{-7} . Extrapolations will probably be even less reliable and consequently it appears (see equation 1) that uncertainties of ± 1 or 2 units of the sixth decimal place of index can not be entirely excluded when refractivities of optically dense media are measured in CO₂-free dry air. From evidence at present available it does not seem likely, however, that such differences can occur over periods of time measured in days or weeks but rather that such possibilities should be remembered when comparing absolute determinations separated by or extending over a period of years.

² L. W. Tilton, BS J. Research 13, 111 (1934) RF695.

³ See equation 22 of this paper.

Obviously, it is seldom convenient to use as a reference medium pure air having a standard temperature and pressure, and free from CO_2 . Usually it is advisable to allow these conditions to vary continuously and to keep adequate records during index determinations. In investigating the relationships involved, a unit error of 1×10^{-6} will be considered as was done in former papers⁴ discussing the geometrical aspects of prism refractometry.

1. TOLERANCES IN MEASUREMENT AND CONTROL OF REFERENCE MEDIUM

The most obviously important effects of varying air conditions on measured relative indices are those arising from the changes in air density through changes in pressure, P , and in air temperature, T (absolute). The relationship between the index of air and its density, d , is usually assumed to be that constancy expressed by Gladstone and Dale's law as $(\mu - 1) = Cd$. Then, in accordance with the ideal gas law, $PV = RT$, it is customary to write for any given wave length

$$(\mu - 1)_{TP} = (\mu_0 - 1) \left(\frac{273}{T} \right) \left(\frac{P}{760} \right) \quad (3)$$

where μ_0 is a value for standard conditions of 0°C and 1 atmosphere pressure, and P is measured in mm of Hg; or, defining p as the excess in mm of Hg over 1 atmosphere of pressure, $p = (P - 760)$ and

$$(\mu - 1)_{tp} = (\mu_0 - 1) \frac{(1 + \beta p)}{(1 + \alpha t)} \quad (4)$$

where t is the temperature on the centigrade scale, β is the pressure coefficient of volume change of a gas at constant temperature (namely $1/760$ for a 1 mm change at a pressure of one atmosphere) and α is the temperature coefficient of expansion of air at constant pressure. The value of α would be $0.00366 = 1/273$ for a change of 1°C , at a temperature of 0°C , if air behaved like a perfect gas, but 0.00367 if the results of experiments on the expansion of air are considered. The only published references to computed reductions or corrections for indices measured in air at other than standard conditions have been based on these or on similar assumptions.

Since at constant temperature the proportionality between $(\mu - 1)$ and d has been found to hold without serious discrepancy over a pressure range of many atmospheres, it is safe to assume that the pressure coefficient of refractivity is identical with β for such small variations in atmospheric pressure as those which occur during index measurements and also, for variations which exist between various places on the earth's surface. Concerning α , however, the similar assumption does not seem equally reliable. According to generally accepted theories of dispersion, a change in the temperature of a medium affects the resonance frequency as well as the mass per unit volume. As a result there is a change or "shift" of the effective wave length of the absorption band, and for each given wave length a different secondary temperature influence on index is superposed on that which is directly ascribed to the change in density. Thus it is

⁴ L. W. Tilton, BS J. Research 6, 59 (1931) RP262; 11, 25 (1933) RP575.

not necessarily true that values of $\partial \mu / \partial t$ can be accurately obtained by differentiating equation 4 with respect to t and solving for

$$\frac{\partial \mu}{\partial t} = - \frac{\alpha(\mu - 1)_{t,p}}{1 + \alpha t} \quad (5)$$

with a constant value of α determined from the expansion of air or even from index data for a single wave length. Instead of such a constant "optical temperature coefficient",⁵ the measurements on air by W. F. Meggers and C. G. Peters⁶ yield an α varying with wave length from 0.00367₄ at 7500 Å to 0.00373₈ at 3500 Å.

Table 1 summarizes the results of direct determinations of this constant for air. The Gylden value was deduced from astronomical observations. Mascart's value should not be considered because of a systematic inconsistency in results obtainable from his data.⁷ Scheel's

TABLE 1.—Optical temperature coefficient for air
(Visible range of frequencies)

Observer	Reference	Temperature interval on Centigrade scale	$\alpha \times 10^4$
Gylden.....	Mém.acad.St.Petersburg 10, no. 1 (1866); 12, no. 4 (1868)	Seasonal range..	369
L. Lorentz.....	K.Danske Videnskab.Selskabs Skrifter [5] 10, 485 (1875)	1 to 17°, 100°.....	367
Von Lang.....	Pogg. Ann. 153, 463 (1874). (This α is given as computed by Benoit).	2 to 95°.....	303
Mascart.....	Compt. Rend. 78, 617-679 (1874); Ann.Sci.École Normale Supérieure [2] 6, 9-78 (1877).	5 to 38°.....	382
Benoit.....	Travaux et Mémoires Bur. Int. Poids et Mesures 6, 102 (1888).	1 to 80°.....	367
Walker.....	Trans.Roy.Soc. (London) A201, 454 (1903).....	10 to 100°.....	360
Scheel.....	Verhandl.deut.physik.Ges. 9, 24-36 (1907).....	-192 to 0°.....	373
Barus.....	Carnegie Inst.Wash.Pub.no. 249-I, page 137 (1916).....	22 to 100°.....	361
Meggers and Peters.....	See page 735 Bul BS 14, (1918) S327.....	0 to 30°.....	368.8
Péiard.....	J.phys.radium [6] 6, 223 (1925).....	0 to 100°.....	371.8
Cheney.....	Phys.Rev. [2] 29, 292-298 (1927).....	25 to 300°.....	367

α is very high, but his temperature interval is much lower than that used by the others. He selected $\alpha=0.00367_4$ for reducing his own index of air from 16 to 0° C. Cheney's temperature interval is much higher than that used by the others. A close analysis of his published numerical data shows that a low value of $\alpha=0.00364$ fits as well or better than the conventional value which he considered verified. Von Lang's α is too low for serious consideration and no great accuracy was claimed for the Barus data.⁸

Considering the amount of data taken, particularly the number of temperatures used, the results by Benoit and by Péiard are probably entitled to special consideration. They used the same method (Fizeau dilatometer) and to some extent the same apparatus. If their values are averaged one has $\alpha=0.00369$ which agrees closely with the average value of $\alpha=0.00368_8$ that is obtained from the Meggers

⁵ From inspection of equation 5 one finds that α can be regarded as the (negative) temperature coefficient of index of refraction of air per unit of "excess refractivity", as determined for $t=0^\circ$ C and $p=0$.

⁶ Bul. BS 14, 697 (1918) S327.

⁷ If from the experiments which he conducted when investigating temperature effects one discards those for temperatures below the middle group, which averages 14.4° C for t , the writer finds that $\alpha=0.00395$ is indicated. On the other hand, when the observations at temperatures above those of the middle group are discarded an $\alpha=0.00356$ is obtained.

⁸ It is of interest to note that Barus' qualitative conclusion concerning the decrease of α for high temperatures is consistent with the trend which has been mentioned for some of the data of table 1, particularly the high value which Scheel found in the very low temperature interval that he used, and the low value which the writer would recommend from Cheney's data.

and Peters data for those particular wave lengths⁹ which Pérard used. Thus it seems certain that the optical temperature coefficient is appreciably larger than the coefficient of expansion which has been so generally used. Since the Meggers and Peters values of α are based on data for such a large number of wave lengths, and since their average for the visible region is in excellent agreement with the average α from the Benoit and the Pérard data, the Meggers and Peters values are therefore recommended for all intercomparisons between precise relative-index data taken at markedly different temperatures.

The wave-length variation in α is of importance chiefly in the ultra-violet region; but even for the visible region, especially for the shorter wave lengths, it is in some cases preferable to use the variation in α when reducing six- and seven-decimal-place indices to certain reference conditions. In particular, when relative indices are used as a basis for computing temperature variations in the absolute index of a medium, it is desirable to consider the most accurate data available on air.

(a) TEMPERATURE OF AIR

Whether α is regarded as constant or as varying with wave length, a formal expression for the error in relative index caused by air-temperature variations is readily written after considering the definitive relation

$$n \equiv \frac{\bar{n}}{\mu} \quad (6)$$

which exists between the relative index, n , of a medium and its absolute index, \bar{n} . Since the latter is necessarily independent of air temperatures and μ^2 is approximately unity

$$\frac{dn}{dt} = -\bar{n} \frac{d\mu}{dt} \quad (7)$$

and consequently, using equation 5, it is found that changes in air temperature result in index variations of like sign according to the equation

$$\Delta n = \frac{\bar{n}\alpha(\mu-1)}{(1+\alpha t)} \Delta t_a \quad (8)$$

from which precise index corrections may be computed for the whole wave-length interval 2218 to 9000 Å by using the Meggers and Peters values¹⁰ of α , which they approximately expressed by the equation

$$\alpha = 0.00367 + \frac{3 \times 10^{-6}}{\lambda^3} \quad (9)$$

where λ is to be used in microns.¹¹

For the purpose of establishing practical tolerance limits in temperature control and measurements, \bar{n} is essentially equivalent to n , and α for the visible region is approximately the ratio 1/270. Moreover, it happens that the refractivity of air at temperatures between 0 and 50° C varies only from about 24 to 30 $\times 10^{-5}$ over the range of

⁹ Pérard used only five wave lengths, all within the visible region, and the data do not seem sufficiently concordant either to confirm conclusively or to disprove the variation of α with wave length.

¹⁰ See page 735 *Bul. BS* 14 (1918) S327.

¹¹ For precise refractometry the angstrom is generally favored by the writer as the appropriate unit of wave length, but the micron is often more convenient in the writing and using of dispersion equations.

the visible spectrum, and ordinary atmospheric-pressure changes of $p = \pm 30$ mm do not materially increase this variation. Therefore, with an accu. acy of approximately ± 10 percent, the tolerance corresponding to $\pm 1 \times 10^{-6}$ in refractive-index measurement is

$$T_{\Delta t_a} = \pm n^{-1}(1 + \alpha t) \text{ degrees C} \quad (10)$$

or, for work on media having indices of 1.9 and 1.3, respectively, the air-temperature changes of $\pm 0.53^\circ(1 + \alpha t)$ and $\pm 0.77^\circ(1 + \alpha t)$ are equivalent to errors of $\pm 1 \times 10^{-6}$ in the measured relative indices. Thus it is evident that a neglect of seasonal changes in air temperatures may easily affect the fifth decimal place of relative-index work on any medium, even though its temperature coefficient of absolute index is negligibly small.

(b) PRESSURE OF AIR

For the purpose of arriving at similar correction and tolerance equations for air pressures, one differentiates equations 4 and 6 with respect to p and finds that an increase in air pressure results in a decreased value of the measured relative index of other media according to the equation

$$\Delta n = -\frac{\bar{n}\beta(\mu-1)_p}{(1+\beta p)} \Delta p \quad (11)$$

from which precise corrections to measured indices can be satisfactorily computed.

Again approximating, as was done with equation 8, equation 11 reduces to the practical working tolerance in air pressures

$$T_{\Delta p_a} = \pm 2.8n^{-1}(1 + \beta p) \text{ mm} \quad (12)$$

which is written for an error of $\pm 1 \times 10^{-6}$ in refractive index and, obviously, for the usual small values of p only one term is needed. Thus for media having indices of 1.9 and 1.3, respectively, the air-pressure changes of 1.5 and 2.2 mm are equivalent to variations of unity in the sixth decimal place of measured relative index of refraction. For many localities, therefore, it is apparent that the fluctuations in barometric pressure within a single working day should not be neglected in precise measurements of relative index, and it is certain that the air-pressure variations that occur over somewhat longer periods affect the fifth decimal of such indices.

(c) HUMIDITY OF AIR

The proportion of water vapor in air is such a variable factor, depending on locality and weather conditions, that it is a difficult matter to decide upon any definite proportion as a satisfactory normal moisture content of the atmosphere. Consequently it is probably better to treat moisture as an impurity and refer all refractive-index measurements to dry air as a standard reference medium.

L. Lorenz¹² gives, for sodium light, a value of $\mu_D = 1.0002500$ as the index of refraction of water vapor at a concentration of 1 mol in 22.4 liters. Comparing this with his index of dry air for equivalent conditions, he gives a correction of $\Delta\mu_v = -0.000041 v/760$ to be applied to his results on dry air when it is desired to obtain indices for moist

¹² Ann. Physik [3] 11, 91 (1880).

air containing water vapor of v mm pressure. The Cuthbertsons¹³ and others¹⁴ have confirmed the magnitude of this correction and, for the region 4779 to 6708 Å, have shown that the dispersion of water vapor is closely comparable with that of air. It seems, in fact, from experimental evidence, that the coefficient of this Lorenz correction, namely, 41×10^{-6} , should not vary more than $\pm 1 \times 10^{-6}$ throughout the visible spectrum.

Since the pressure of water vapor in the air seldom exceeds a small fraction of an atmosphere, it seems permissible to assume the equivalence of optical temperature coefficients for water vapor¹⁵ and for air, and to use the Lorenz humidity correction for all ordinary temperatures by writing for the whole visible range of frequencies

$$(\mu_t - 1)_v = (\mu_t - 1)_{v=0}(1 - \gamma v) \quad (13)$$

where $\gamma = 185 \times 10^{-6}$ is used to replace $41 \times 10^{-6} / [760 (\mu_0 - 1)]$. This equation is written for a pressure of 760 mm, but is applicable at all ordinary atmospheric pressures. Differentiating equation 13 with respect to v gives

$$\frac{d\mu}{dv} = -\gamma(\mu_t - 1) \quad (14)$$

and, referring to a similar differentiation of equation 6, it is evident that an increase in the moisture content of the air results in an increased relative index of a medium which is being measured therein, the error equation being

$$\Delta n = +\bar{n}\gamma(\mu_t - 1)\Delta v \quad (15)$$

for use in precise computations of refractive index of the medium which is measured in air.

For an error of unity in the sixth decimal of index, and with sufficient accuracy for many purposes, equation 15 yields

$$T_{\Delta n} = \pm 20n^{-1}\text{mm} \quad (16)$$

which shows that the unit tolerances in absolute humidity vary from approximately ± 10 to ± 15 mm of vapor pressure. These figures apply to measurements of relative index on media having indices of 1.9 and 1.3, respectively. Vapor pressures of 10 and 15 mm correspond at 50° C to 10 and 15 percent in relative humidity and at 30° C to 30 and 45 percent, respectively. Thus it is evident that humidity corrections are not negligible in sixth-decimal-place refractometry, especially when working in air at temperatures somewhat higher than those of ordinary room conditions or whenever there is danger of humidities approaching the saturation point (because, for example, of leaks in water-jacketed housings used for temperature control).

¹³ C. and M. Cuthbertson, *Trans. Roy. Soc. (London)* A213, 16 (1913).

¹⁴ J. Wüst and H. Reindel, *Z. physik. Chem.* B24, 176 (1934). See, also, P. Hölemann and H. Goldschmidt, page 204 of same volume.

¹⁵ From the Hölemann and Goldschmidt data (see footnote 14) on water vapor, considered together with published densities of steam, one may deduce a tentative value $\alpha = 0.0035$ to compare with 0.0037 for air.

(d) CARBON-DIOXIDE CONTENT OF AIR

After water vapor the next atmospheric constituent of recognized importance, so far as its variability is concerned, is carbon dioxide. Although the range in its fluctuations is known to be much smaller than in the case of water vapor, there is a prevalent opinion that when using air as a reference medium for measurements of highest precision one should eliminate either the CO₂ itself or the effects of its variations. While there are on record some presumably reliable analyses of air showing a carbon-dioxide content as high as 0.3 percent by volume, such samples were taken under exceptional circumstances, such as in very crowded theaters of an obsolete and most unfavorable type, or in rooms with no ventilation, and they have no interest in the present connection. Boothby and Sandiford¹⁶ reported that outdoor air sampled under favorable conditions varies in its CO₂ content by only a few thousandths of one percent from a normal value of 0.036 percent. Similarly, F. G. Benedict¹⁷ found that outdoor air does not vary in this respect more than a few thousandths from an average value of 0.031 percent, but Hann¹⁸ states that the percentage of CO₂ varies from 0.033 to 0.043 in the air of cities. Consequently, in view of these small variations, it seems permissible and somewhat preferable to adopt some value, say 0.00035, as the normal volume proportion of CO₂ in air rather than to favor the more stringent requirement that a standard reference medium must be entirely free from carbon dioxide.

Precise index measurements are, of course, seldom made in outdoor air, but in any well ventilated modern laboratory it is unlikely that the carbon-dioxide content ever exceeds two or three times the normal value. In air from the Boston and New York subways, sampled just after the rush hours, Benedict found only twice the normal CO₂ content. Moreover he found that an increase in CO₂ is accompanied by an equal volume decrease in O₂, and consequently it is the difference in the refractivities of carbon dioxide and of oxygen which must be considered in making corrections to indices measured in air having an abnormal CO₂ content.

The averages of a number of published¹⁹ values of the refractive indices of CO₂ and O₂ are 1.000450 and 1.000271, respectively, for sodium light, a temperature of 0° C and a pressure of 760 mm. Hence a correction of

$$\Delta\mu_c = +0.000179(C - 0.00035) \quad (17)$$

should be applied to the measured index of normal air (containing the proportion 0.00035 of CO₂) at 0° C and 760 mm pressure when it is desired to obtain the index of air containing the total proportion C of carbon dioxide.

A comparison of the dispersions of oxygen and of carbon dioxide shows that the correction given by equation 17 varies only 1 percent or less for the visible-spectrum region. Proceeding as with the water-vapor correction (part c of this section) and writing $c = C - 0.00035$ to represent excess CO₂, one finds for all ordinary temperatures

$$(\mu_t - 1)_C = (\mu_t - 1)_{c=0}(1 + \epsilon c) \quad (18)$$

¹⁶ Am. J. Physiol. 55, 295 (1921).

¹⁷ The Composition of the Atmosphere, Carnegie Inst., Wash., Pub. no. 166., pages 110-114 (1912).

¹⁸ J. von Hann's Lehrbuch der Meteorologie, page 5 (1915).

¹⁹ See Landolt-Börnstein Tabellen.

where $\epsilon = 0.612$ replaces $179 \times 10^{-6} / (\mu_0 - 1)$. Differentiating equation 18 with respect to c gives

$$\frac{d\mu}{dc} = +\epsilon(\mu_t - 1) \quad (19)$$

whence, again using equation 6, it is apparent that the presence in laboratory air of CO_2 in abnormally high proportions results according to the equation

$$\Delta n = -\bar{n}\epsilon(\mu_t - 1)\Delta c \quad (20)$$

in decreased values for such relative indices as are measured in the laboratory.

For unit error in the sixth decimal place of refractive index the corresponding tolerance in volume proportion of abnormal CO_2 content of air is

$$T_{\Delta c} = \pm 0.006n^{-1} \quad (21)$$

with sufficient accuracy for all purposes. Evidently no consideration whatever need be given to CO_2 content of air in sixth-decimal-place refractometry. Even for the extreme case when a medium of index 1.9 is being measured, the excess proportion of CO_2 must reach the high value of 0.003, or nine times the proportion normally present in fresh air, in order to produce an error of -1×10^{-6} in relative index. For seventh-decimal-place index work, however, it may be ascertained from these results that the normal CO_2 content of fresh air causes a lowering of approximately 1×10^{-7} in the measured relative indices of very dense media as compared with the use of a standard reference air containing no carbon dioxide. Consequently, it may be safely inferred that, in general, indices of laboratory air, or relative indices measured in such air, are not likely to have any precision whatever in the eighth decimal place unless simultaneous CO_2 determinations are made.

2. CORRECTIONS TO STANDARD REFERENCE CONDITIONS

If the working conditions affecting the reference medium are not maintained constant at the desired standard values or controlled within the tolerance limits as specified above, then it becomes necessary to observe the existing conditions and make the proper corrections to the results actually obtained. When it is necessary to correct sixth-decimal-place indices to standard conditions, or to convert them to absolute indices, the use of slide-rule corrections is often lacking in adequate precision and correction tables for these purposes are advisable. Their preparation in detail is a profitable preliminary whenever numerous data are to be reduced for a limited number of spectral lines. For highest precision such tables are especially desirable because certain refinements in precision of correction need be considered explicitly only during their original preparation.

The writer recommends the use of logarithms in the computation of indices, and the evaluation of all corrections in terms of $\log n$. One of the first requirements is a double-entry table of barometer corrections with barometer temperature and barometer reading as arguments. With the temperature reductions²⁰ to be listed in this table the barometer calibration corrections (for a given instrument)

²⁰ Temperature reductions for use with a Fortin-type barometer and brass scale are given in tables 46 and 47 of the 4th revised edition, Smithsonian Meteorological Tables.

are combined and then all entries are modified to reduce pressures from local²¹ to standard gravity, $g=980.665$ dynes.

Another requirement is a series of double-entry tables (or their equivalent), with air pressure and temperature as arguments, giving the positive corrections which, when applied to $\log n$, will yield $\log \bar{n}$, of which the antilog is absolute index. The various members of such a series of tables relate to the different spectral lines that are to be frequently used for index measurement and these member tables will differ principally because of the dispersion of air. (There is also a slight additional difference because of the nonconstancy of α , the optical temperature coefficient of index. See discussion in part 1 of section II.) As an example of a member table of such a series, table 2 is abstracted (by decimation in each argument) from a usefully detailed tabulation for the hydrogen line, $\lambda=4861$ A.

TABLE 2.—Data for converting from relative to absolute index ($\lambda=4861$ A) by the equation $\log \bar{n}=\log n+\log \mu$

P=air pressure (in mm of mercury)	Values of $10^8 \times \log \mu_P$ for dry air at various temperatures (with normal CO ₂ content)				
	0° C ($\Delta P=0.06$)	10° C ($\Delta P=0.13$)	20° C ($\Delta P=0.24$)	30° C ($\Delta P=0.44$)	40° C ($\Delta P=0.77$)
730	12295	11857	11449	11069	10713
740	12464	12020	11606	11220	10859
750	12632	12182	11763	11372	11006
760	12800	12344	11920	11524	11153
770	12969	12507	12077	11675	11300
780	13137	12669	12234	11827	11446

NOTES

1. For each 10 percent of relative humidity subtract tabulated values of ΔP from air pressure before entering this table.
2. Tentatively it may be advisable to reduce $\log \mu$ by 5 or 10 parts in 10,000 for each increase of 20 in relative annual sunspot number above average $S=46$.
3. If it is desired to use these data for wave lengths other than 4861 A, modify air temperature according to table 4 before entering this table.

In preparing such correction tables in this laboratory, the temperature and dispersion data as given by Meggers and Peters²² have been combined with the average value of refractive index, $\mu=1.0002926$ (for $\lambda=5893$ A, $t=0^\circ$ C and $P=760$ mm), which is obtained by considering all published data²³ and adding a very slight correction for normal CO₂ content. Since the ratio of $\log \mu$ to $(\mu-1)$ is very nearly constant over the whole range in μ that is involved in temperature and pressure reductions and corrections, the actual computation of air indices is unnecessary except for 0° C and 760 mm pressure. The values of $\log \mu$ for all ordinary temperatures and pressures may be directly computed (see equation 4 and citations of footnote 22) from the equation

$$\log \mu_{tp} = \frac{1 + \beta p}{1 + \alpha t} \log \left[1 + \left(2884.3 + \frac{13.412}{\lambda^2} + \frac{0.3777}{\lambda^4} \right) \times 10^{-7} \right] \quad (22)$$

where α varies with wave length as approximately expressed by equation 9, and λ should be written in microns.

²¹ Gravity reductions are discussed on pages XXXV and XXXVI of 4th revised edition, Smithsonian Meteorological Tables.

²² See pages 722 and 735, Bul BS 14 (1918) S327.

²³ See page 115, BS J. Research 13 (1934) RP695.

If, in table 2, the values for a standard pressure are subtracted from those for other pressures in the same column, a set of air-pressure corrections to the logarithms of observed indices is obtained. Table 3 gives such corrections for limited ranges in pressure and in temperature variations. Such a table may, of course, be computed directly from the basic values of the logarithms for the indices at standard pressure, $p_0=760$ mm. For this purpose the equation

$$\Delta \log \mu_{tp} = \frac{\log \mu_{tp_0} \Delta p}{760} \quad (23)$$

is obtained from equation 22.

TABLE 3.—Data for referring relative indices to dry air at standard pressure by the equation $\log n_{760} = \log n_p + \Delta \log n$.

Use tabulated values with negative sign when $P-760$ is negative]

(P-760 mm) ≡ excess atmospheric pressure	Values of $10^8 \times \Delta \log n$ for various air temperatures		
	10° C ($\Delta P=0.13$)	20° C ($\Delta P=0.24$)	30° C ($\Delta P=0.44$)
20	325	314	303
19	309	298	288
18	292	282	273
17	276	267	258
16	260	251	243
15	244	235	227
14	227	220	212
13	211	204	197
12	195	188	182
11	179	173	167
10	162	157	152
9	146	141	136
8	130	125	121
7	114	110	106
6	97	94	91
5	81	78	75
4	65	63	61
3	49	47	45
2	32	31	30
1	16	16	15
0	0	0	0

NOTE.—For each 10 percent of relative humidity subtract tabulated values of ΔP from air pressure before entering this table.

Although the direct use of table 2 is strictly limited to the correction of indices for the F line of hydrogen, table 3 is applicable over a very wide range in the spectrum. In fact, for pressure corrections as large as 30 mm the use of table 3 may be extended over the whole visible range of frequencies without errors exceeding $\pm 5 \times 10^{-8}$ in the logarithmic corrections so determined.

As they are written, tables 2 and 3 both relate to dry air and must be modified because of the presence of water vapor. From equations 11 and 15 it is evident that any given increase in moisture can be represented as an equivalent decrease in pressure, and thus it is possible to find a simple system of modifying the actual corrected pressures to fictitious or pseudo pressures which, when used as actual pressures, permit tables 2 and 3 to serve for moist as well as dry air. Hence the ΔP equivalents which appear in the column headings of tables 2 and 3.

If for any reason it is objectionable to prepare a complete series of $\log \mu$ tables, similar to the single example given as table 2, one may use a relationship between $\Delta\mu/\Delta\lambda$ and $\Delta\mu/\Delta t$ for extending to other wave lengths the use of a table written primarily for one particular

TABLE 4.—Auxiliary data for extending use of table 2 to various wave lengths

$\lambda \equiv$ wave length (in angstroms)	Values of Δt for modification of air temperatures		
	0° C	20° C	40° C
4000	-3.18	-3.30	-3.43
4100	-2.70	-2.80	-2.91
4200	-2.25	-2.33	-2.42
4300	-1.83	-1.90	-1.98
4400	-1.45	-1.50	-1.56
4500	-1.10	-1.14	-1.18
4600	-0.76	-0.79	-0.82
4700	-0.45	-0.47	-0.49
4800	-0.16	-0.17	-0.18
4900	+0.11	+0.11	+0.12
5000	+0.36	+0.38	+0.40
5200	+0.81	+0.84	+0.87
5400	+1.21	+1.26	+1.31
5600	+1.57	+1.63	+1.69
5800	+1.88	+1.96	+2.04
6000	+2.17	+2.26	+2.35
6200	+2.42	+2.52	+2.63
6400	+2.65	+2.76	+2.88
6600	+2.86	+2.98	+3.10
6800	+3.05	+3.18	+3.31
7000	+3.21	+3.36	+3.50

wave length. For this procedure it is found advantageous to regard temperature as the fictitious argument, and table 4 gives the particular corrections of this type that should be algebraically applied to actual air temperatures before entering table 2 for wave lengths other than 4861 Å.

III. REQUISITE CONSTANCY AND UNIFORMITY FOR REFRACTIVE MEDIA

Uniformity of reference conditions is a necessary but by no means a sufficient condition for high precision in refractometry. Even the absolute indices of refraction of optical media are ratios which may, for divers reasons, vary from time to time or with respect to coordinates within the media. Temperature and pressure are obviously important factors and, in the case of optical glass, heat treatment may introduce not only a variation of index with direction of travel of the light, but also a marked physico-chemical change²⁴ in index that often varies from point to point in the glass although it is not a function of orientation. Moreover, the writer has found that striae in glass, when they exist in well-stratified layers, can affect measurements of index and of dispersion in a systematic manner that may prove misleading in some investigations. Therefore, for consistent results in index determinations, it is necessary carefully to consider the conditions which cause real or apparent changes in the optical density of media that are subject to measurement.

²⁴ A. Q. Tool and C. G. Eichlin, *J. Opt. Soc. Am.* **4**, 359 (1920); *BS J. Research* **6**, 525 (1931). A. Q. Tool, L. W. Tilton, and E. E. Hill, *J. Opt. Soc. Am. and Rev. Sci. Inst.* **12**, 490-491 (1926). L. W. Tilton, *J. Wash. Acad. Sci.* **20**, 12-13 (1930).

1. EFFECTS OF TEMPERATURE ON REFRACTIVE MEDIA

Comparatively little recent progress has been made in the theory of temperature effects on absolute index. It is considered, however, that this effect consists of at least two well-defined components which may, and often do, have opposite signs. The more frequently mentioned component is the direct result of thermally produced changes in density and its sign is always as indicated by the equation

$$\frac{\partial \bar{n}}{\partial t} = f(\bar{n}) \frac{dd}{dt} \quad (24)$$

where $f(\bar{n})$ is a positive function, which may be variously approximated (for example as $\bar{n}-1$, \bar{n}^2-1 , $\frac{\bar{n}^2-1}{\bar{n}^2+2}$, etc.). Consequently, for

media which expand when heated, this change in index is negative as temperature increases, and this effect of temperature is not a function of wave length but relates primarily to conditions for infinite wave length. As mentioned for air in part 1 of section II, the second component is ascribed to changes in the resonance frequencies of the media. The increase in volume which usually takes place with rising temperature is often accompanied by a capacity for absorbing radiation of longer wave length. Thus, under such circumstances, ultraviolet absorption bands are "shifted" toward the longer wave lengths of the visible region while the long wave lengths of infrared bands become still longer. Obviously, then, these absorption components of temperature coefficients of index are functions of wave length.

The velocity of propagation of radiation of a wave length longer than that corresponding to the effective absorbing frequency is decreased, while that of shorter wave length is increased. Consequently, for media whose principal effective absorption bands lie in the ultraviolet, this total absorption-band component of refractive index usually increases as temperature increases, while the reverse is true for media with predominant infrared absorption. In most optical glass the index change (in and near the visible spectral region) that is ascribable to the ultraviolet absorption bands predominates over the combined effects of the directly produced density change and the change caused by shifting of the infrared bands. For some crown glasses, however, the opposing tendencies are about equal in absolute value, and the index sensitivity to temperature is quite negligible. For a few crowns of very low index and somewhat unusual chemical composition the density and infrared effects predominate and negative temperature coefficients of absolute index are found, especially for the longer wave lengths of the visible spectrum.

(a) THERMAL COEFFICIENTS OF ABSOLUTE REFRACTIVE INDEX

For a few optical glasses temperature coefficients of absolute index are readily available through the work of C. Pulfrich²⁵ and of J. O. Reed.²⁶ Also, such coefficients can be readily computed from coeffi-

²⁵ Ann. Physik 45, 609-665 (1892).

²⁶ Ann. Physik 65, 707-741 (1898).

icients of relative index that have been determined by C. S. Hastings,²⁷ G. Müller,²⁸ H. Fizeau,²⁹ and J. W. Gifford;³⁰ or they may be estimated from the graphs published by C. G. Peters.³¹ From a critical examination of these data, and also from unpublished results obtained in this laboratory, it is inferred that chemical composition is a predominantly important factor in determining this thermo-optical relationship for glass. Accordingly, it is difficult to make concise general statements, with a useful degree of precision, about the quantitative effects of temperature variations on the refractivity of glasses.

At usual room temperatures the values of $10^6 \times \Delta \bar{n} / \Delta t$, for the sodium-lines indices of flint glasses vary from approximately 0 for some light flints containing barium to +14 for those with highest lead content. For low-index glasses it is often considered that the temperature coefficients of absolute index are small and that they lie within a narrow range as compared with flint glasses. Available data confirm this view for the optical crowns in frequent use, the range in their coefficients being -1 to +3. Measurements in this laboratory show, however, that certain fluor crowns have values of -3 while for Pyrex and fused-quartz glasses of similar refractivity the coefficients are +5 and +9, respectively.

For crystals the range in refractive-index sensitivity to temperature is greater than for glasses. It is sufficient to mention sylvite, which, at room temperature, has a coefficient of approximately -34×10^{-6} , and calcite with its value of $+11 \times 10^{-6}$ for the extraordinary ray.

Numerous published data concerning the temperature coefficients of refractive index of liquids show that such values are all negative (direct density effect predominating over that of the absorption shift in the ultraviolet) and very much larger in numerical value than those for most solids.³² It is useful to remember, however, that the percentage variations among such temperature coefficients for different liquids are, in general, relatively small compared with similar variations for solids. For a large number of transparent and semi-transparent liquids (including numerous oils) the value of $\Delta \bar{n} / \Delta t$ is approximately expressed as -4×10^{-4} , although a number of organic liquids have values ranging from -4 to -6×10^{-4} . Water with a value of -1×10^{-4} is a most important exception and, on the other hand, there are some optically dense liquids which have unusually high (negative) temperature coefficients of index, such, for example, as -7 and -8×10^{-4} for methylene iodide and carbon disulphide, respectively.

(b) APPROXIMATE RANGE IN TEMPERATURE TOLERANCES FOR SOLIDS AND LIQUIDS

When considering temperature tolerances for general refractometry it must be remembered that one may have no prior knowledge of the media and their temperature coefficients of index. From the values given above it is evident that even for glasses known to be the usual crowns and light flints, the temperature of the sample during refrac-

²⁷ *Am. J. Sci.* [3] **15**, 269-275 (1876).

²⁸ *Publicationen Astrophysikalischen Observatoriums Potsdam* **4**, 149-216 (1885).

²⁹ *Ann. chim. phys.* [3] **66**, 429-482 (1862).

³⁰ *Proc. Roy. Soc. (London)* **A91**, 319-321 (1915); **A100**, 621-626 (1921-22).

³¹ *BS Sci. Pap.* **36**, 635 (1926) S521.

³² The absolute value of the ratio between temperature coefficients of liquids on the one hand and of glasses and common optical crystals on the other may be very roughly expressed as 100. Consequently, for liquids, coefficients of relative and of absolute refractive index are practically identical, whereas for glasses and crystals they frequently are quite different and may be of opposite sign.

tive-index determinations must be known within the limits of approximately $\pm 0.3^\circ \text{C}$ to correspond to a limiting error of $\pm 1 \times 10^{-6}$ in index. For Pyrex glasses and for fused quartz the corresponding temperature tolerances are ± 0.2 and $\pm 0.1^\circ \text{C}$, respectively. Possibly, precision and accuracy in temperature measurement within approximately these limits can with care be obtained when a glass prism is mounted on a spectrometer table and exposed to the air of a room in which the temperature is constant or changes but slowly. When, however, the denser flint glasses are considered, it is found that the tolerances are reduced to $\pm 0.07^\circ \text{C}$ (and even to $\pm 0.03^\circ \text{C}$ for determinations with the shorter wave lengths of the visible spectrum).

Consequently, without considerable prior knowledge concerning a particular transparent solid, it is not advisable to attempt sixth-decimal-place refractometry unless temperature is controlled and measured to within $\pm 0.1^\circ \text{C}$ or better. It is, therefore, highly desirable to provide a constant-temperature prism housing, even for work in a constant-temperature room, and to determine all indices with respect to a stirred air bath surrounding the prism.

Tolerances in temperature control and measurement during refractive-index determinations on liquids to within one unit of the sixth decimal place are so strict that there seems to be no published account of a serious attempt to reach such precision. The requirements are $\pm 0.01^\circ \text{C}$ for water at room temperature, and approximately $\pm 0.002^\circ \text{C}$ for liquids of average thermo-optical sensitivity.

2. EFFECTS OF PRESSURE ON REFRACTIVE MEDIA

L. Zehnder³³ found that water under pressure increases in absolute index by 15×10^{-6} per atmosphere at 20°C , and he favored the Gladstone and Dale relation, the constancy of $(n-1)/d$, when the density of liquids is varied by pressure. W. C. Röntgen and L. Zehnder³⁴ give data on similar increases in index for several other liquids. Although they do not choose between the Gladstone and Dale relation and that of Lorenz-Lorentz, their results are in general somewhat more favorable to the former.

F. Pockels³⁵ investigated the effects of unidirectional pressure on seven kinds of glass, using Neumann's equations for expressing the influence of elastic deformation on the propagation of light, and he found the Newton relation, $(n^2-1)/d$, somewhat more nearly constant than that of Gladstone and Dale or of Lorenz-Lorentz. L. H. Adams and E. D. Williamson³⁶ also have published results on the relation between birefringence and stress in various types of glass and have confirmed Pockels' results where comparisons can be made.

There is, however, a general lack of direct experimental evidence relating to the effect of atmospheric-pressure variations on the absolute refractivity of transparent media, and particularly so for optical glasses. Both Hovestadt³⁷ and Adams and Williamson have mentioned the computation of such effects from Pockels' work and the equations of Neumann but, unfortunately, the necessary constants

³³ Ann. Physik [3] 34, 91-121 (1888).

³⁴ Ann. Physik [3] 44, 49-50 (1891).

³⁵ Ann. Physik [4] 7, 745-771 (1902); also, for important corrections, see 9, 221 (1902.) and 11, 652 (1903).

³⁶ J. Wash. Acad. Sci. 9, 609-623 (1919).

³⁷ H. Hovestadt, Jena Glass, appendix H, page 409 (Macmillan and Co., London, 1902).

characteristic of different media are known only for the seven glasses used by Pockels, and two ³⁸ of these are unusual aluminoborates.

(a) VARIATIONS IN ATMOSPHERIC PRESSURE

Some indications concerning the effects of atmospheric-pressure changes on liquid and solid refractive media may, however, be obtained from a consideration of the index-density relationships together with the published data on β , the compressibility at constant temperature, and on its reciprocal, K , the "bulk modulus" or modulus of volume elasticity. If, in accord with Pockels' results for glass, the Newton form $(n^2-1)=C'd$ is written to express the index-density relation for a medium having a modulus $K=-V dP/dV$, and a density $d=m/V$, then

$$d\bar{n} = \frac{(n^2-1)}{2nK} dP \quad (25)$$

is obtained as the differential relation between refractive index and pressure. Or, if the index-density relation is $(n-1)=C''d$, one finds according to this Gladstone and Dale premise

$$d\bar{n} = \frac{(n-1)}{K} dP \quad (26)$$

as an alternative index-pressure relationship. Similarly, from the Lorenz-Lorentz expression

$$d\bar{n} = \frac{(n^2-1)(n^2+2)}{6nK} dP \quad (27)$$

would be written.

In view of the analysis which Pockels made from his experiments on glass under pressure, equation 25, rather than 26 or 27, is preferable for solids, while from the work of Zehnder and of Röntgen and Zehnder with pressure applied to liquids it may be inferred that equation 26 is more suitable for liquid media. The writer has found, however, that Pockels' data agree with an index-density relation

$$\left(\frac{n^2-1}{n^2+2}\right)^5 = C'''d^3 \quad (28)$$

much more closely than with the Newton relation, and consequently the differential equation

$$d\bar{n} = \frac{(n^2-1)(n^2+2)}{10nK} dP \quad (29)$$

is preferable to equation 25. Since $K=E/[3(1-2\sigma)]$, where E is Young's modulus of elasticity and σ is Poisson's ratio, equation 29 may be written

$$d\bar{n} = \frac{3(1-2\sigma)(n^2-1)(n^2+2)}{10nE} dP \quad (30)$$

³⁸ The Pockels glass listed as 0.428, $n=1.5123$, was not, as originally supposed, an aluminoborate like his two other crown glasses but, according to subsequently published corrections (see footnote 35, p. 409), was a borosilicate glass. The revised data are free from peculiarities which marked the original exhibit. Nevertheless the errors continue to be propagated in the literature or, as in the International Critical Tables, 2, page 106, this important glass is omitted from consideration.

or, the change in index may be computed approximately from

$$\Delta\bar{n} = \frac{0.15(n^2-1)(n^2+2)}{nE} \Delta P \quad (31)$$

if the constant value $\sigma=0.25$ is used instead of experimentally determined values³⁹ which with few exceptions lie within the range from 0.21 to 0.27.

Equation 26 has been used in computing $\Delta\bar{n}$ for water, ethyl ether, ethyl alcohol, benzene, and carbon disulphide, using for $1/K$ averages of several published values for the compressibility of these liquids. The results agree within the limits of ± 10 percent with published experimental values. Equation 29, which is based on the new index-density relation, has been used in computing $\Delta\bar{n}$ for Pockels' glasses when subjected to changes in hydrostatic pressure, and the results lie within approximately ± 5 percent of those computed from the actual experimental constants in the manner suggested by Pockels. These 5 percent differences compare with 15 and 20 percent differences that are obtained when a similar computation is made by equation 25, which is based on the Newton index-density relation as favored by Pockels. If, however, the approximate form (equation 31) is used instead of equation 29 the differences are as large as 15 percent.

From equation 26 the tolerance in atmospheric- or other hydrostatic-pressure changes for work on liquid media is

$$T_{\Delta P} = \pm \frac{0.000001}{(n-1)\beta} \quad (32)$$

for a limiting error of $\pm 1 \times 10^{-6}$ in refractive index. For ethyl ether or carbon disulphide the limits ± 12 mm are found, and for ethyl alcohol or benzene they are ± 17 mm. Consequently, pressure corrections of this nature should not be entirely neglected. The less compressible liquids water and glycerin have the correspondingly more liberal tolerances of ± 47 and ± 72 mm, respectively, and they require corrections only for extremes of pressure variations or for reductions to standard conditions after measurements are made where the prevailing barometer level is appreciable different from 760 mm.

From equation 31 one may write

$$T_{\Delta P} = \pm \frac{0.000007nE}{(n^2-1)(n^2+2)} \quad (33)$$

as the tolerance in ΔP for sixth-decimal-place refractometry of glass. The values of E seem to range within ± 10 percent of the average 0.72×10^6 atmospheres for borosilicate glasses not containing lead oxide or phosphoric acid,⁴⁰ and except for a few extremes all other glasses seem to have values of E which lie within ± 10 percent of 0.58×10^6 atmospheres. Hence, considering only the absolute index of glass, it is evident that the tolerance in measurement or control of air pressures is seldom if ever⁴¹ less than about 1/2 atmosphere, and therefore of no moment.

³⁹ R. Straubel, Ann. Physik [3] 68, 409 (1899); or see page 193 of reference cited in footnote 37, page 409.

⁴⁰ See page 159 of reference cited in footnote 37, page 409.

⁴¹ Glasses containing approximately 74 percent of PbO may perhaps be exceptions. See part b of this section.

In connection with the subject of pressure effects on absolute index it should be remembered, however, that corrections of this nature should be considered if one attempts a precise comparison between relative indices and those observed under actual vacuum conditions. In the case of liquids, it becomes imperative, even for accuracy in the fifth decimal place, to distinguish between values actually measured in a near vacuum (under their own vapor pressure) and those which are reduced from relative measurements by considering only the effect of pressure on the ambient air.

(b) STRESS AND BIREFRINGENCE

In addition to hydrostatic-pressure effects there should also be considered those effects which are due to unequally distributed stresses within media. Such stress distribution is caused, for example, by improper annealing of glass and by the improper mounting or tight clamping of optical components, especially under varying temperature conditions. The birefringence that is produced in this manner is commonly used as a measure of the internal stress and it seems desirable, if possible, to use birefringence as a measure of that change in refractive index which accompanies the stresses.⁴²

From Pockels' ⁴³ work it is possible to express the ratio between the change in index, Δn_y or Δn_z , and the absolute birefringence, $\Delta n_y - \Delta n_z$. For the ray which vibrates parallel to the axis of stress

$$\frac{\Delta n_y}{\Delta n_y - \Delta n_z} = \frac{2\sigma}{1 + \sigma} - \frac{(1 - 2\sigma)\frac{q'}{v'}}{(1 + \sigma)\left(\frac{p'}{v'} - \frac{q'}{v'}\right)} \quad (34)$$

and for the transverse ray

$$\frac{\Delta n_z}{\Delta n_y - \Delta n_z} = \frac{2\sigma}{1 + \sigma} - 1 - \frac{(1 - 2\sigma)\frac{q'}{v'}}{(1 + \sigma)\left(\frac{p'}{v'} - \frac{q'}{v'}\right)} \quad (35)$$

where p'/v' and q'/v' are experimentally determined constants for a given medium and σ is Poisson's ratio. For glass in the index range 1.5 to 1.75 the Pockels' data lead to higher absolute values for the transverse ray ratio, while for his glass of $n=1.96$, equation 34 gives the larger ratio. Consequently, it may be said that the tolerance in absolute stress-birefringence $\Delta n_b = (\Delta n_y - \Delta n_z)$, in millimicrons per centimeter, for sixth-decimal-place measurements of refractive index is at least as large as

$$T_{\Delta n_b} = \pm \frac{10}{f\left(\sigma, \frac{p'}{v'}, \frac{q'}{v'}\right)} \text{ millimicrons per cm} \quad (36)$$

⁴² For clearness it may be necessary to mention that any piece of unannealed glass in which such stresses exist is a balanced system involving both tensions and compressions so that the index changes which are produced are of opposite sign and offset each other. (See L. H. Adams, *J. Franklin Inst.* **216**, 39-71 (1933).) Nevertheless, portions of the glass may show an average birefringence of a given sign and therefore experience a correspondingly changed index. Such limited portions of the medium should be individually considered because such a procedure corresponds closely to conditions under which optical-glass components are often measured and used, namely, at partial rather than at full apertures.

⁴³ *Ann. Physik* [4] **7**, 745-771 (1902); also for important corrections, see **9**, 221 (1902), and **11**, 652 (1903).

where the denominator is computed according to equation 35 for glasses of index $<1.8+$ and by equation 34 for glasses of index >1.9 , approximately.

Equation 36 with each of these denominators written in turn, has been used in computing the corresponding values of this tolerance for the seven Pockels glasses, and figure 1 shows the chief results. It does not seem clear where the curves of this figure should cross⁴⁴ and accordingly they are not drawn in full. In any event, it is evident that stress-birefringence differs greatly in the degree of sensitivity with which it can serve to measure the variations in index which may

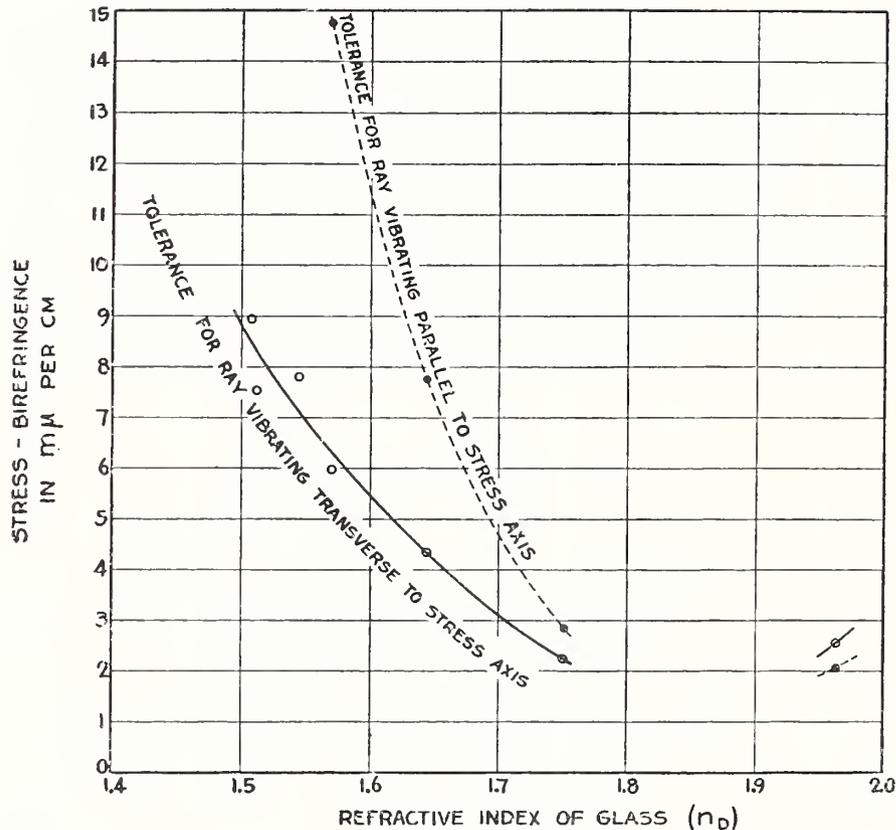


FIGURE 1.—Tolerances in stress-birefringence for optical glass on which refractive index is measured to $\pm 1 \times 10^{-6}$.

Where the orientation is indefinite, the safer tolerance is obviously that indicated by the lower curve.

exist because of the stresses in various kinds of glass. For flints it is obvious that the birefringence present should be much lower than for crowns, and consequently the annealing requirements and schedules for flints should not be too readily neglected because of the comparative ease with which their stress-birefringence seems to be reduced. Nevertheless, remembering the Rayleigh limit of one-quarter wave

⁴⁴ Pockels concludes that p' must equal q' for some glass containing about 74 percent of PbO (index 1.8+) and consequently from equations 34, 35, and 36 it seems that for p' and q' not zero the crossing of the curves of figure 1 should occur at zero tolerance (at least for all values of σ other than 0.5, which is the limiting value for the case of an incompressible medium). On the other hand, for $\sigma=0.5$ when $p'=q'$, or whenever $p'=q'=0$, the ordinate of the point of crossing appears indeterminate as judged from these equations.

length of permissible phase difference and the resulting requirement⁴⁵ that optical glass be uniform in index within ± 0.000007 per cm, it seems that tolerance limits of from 5 to 10 $m\mu$ birefringence per cm are of a proper order of magnitude for most of the requirements of optical glasses.

3. HEAT TREATMENT, CHEMICAL HETEROGENEITY, AND STRIATION OF GLASS

When a piece of glass has been annealed in such manner that the stress-birefringence is within the tolerances shown in figure 1 it is very likely that it is also homogeneous in physico-chemical sense. The work of Tool⁴⁶ and others has shown, however, that there is a possibility of residual inhomogeneity that can be caused by furnace-temperature gradients during the annealing and which may not be accompanied by appreciable birefringence in the finished product. The sensitivity of optical glass to variations in annealing temperature is such that decreases in refractive index of from 2 to 6×10^{-5} are found in various glasses at ordinary temperatures corresponding to increases of 1° C in the annealing temperature.⁴⁷ These annealing-temperature coefficients of index of glass are in general approximately ten times as large as the ordinary temperature coefficients of refractive index, which were considered in part 1 of this section.

In measuring refractivities to five or six decimals, and when making intercomparisons of such results, it is necessary, therefore, before drawing inferences and conclusions, to consider the possibility of small temperature gradients in the annealing furnaces and also to remember the great difficulties of securing sufficiently exact duplications of annealing temperatures and cooling rates during runs in different furnaces or even in successive runs of a given furnace.

Before the optical effects of nonuniform heat treatment were known, it was considered difficult or perhaps impossible to secure chemical homogeneity to the extent necessary for refractive-index uniformity to the sixth or even to the fifth decimal place, even when concerned with distances of only a few centimeters in good optical glass. A careful study and analysis of Fritz Eckert's⁴⁸ investigation shows, however, that after "fine annealings" the maximum spread chargeable to chemical heterogeneity within each of three melts of a barium flint glass was $\pm 1 \times 10^{-5}$ or less in refractive index. Also, a report on careful annealings of six lens blanks at this Bureau⁴⁹ shows that the chemical heterogeneity, if any, was confined entirely to a few units of the sixth decimal place of refractive index.

Such gross and intensely localized heterogeneities as striae do not occur to any large extent in the best optical glass, but it is sometimes desirable to extend minimum-deviation measurements to less perfect glasses. Consequently, it would be of interest to know the extent to which striation can be present without seriously affecting index measurements. Unfortunately little definite information is available.

Striae commonly exist in two well-known forms—as "cords" or "veins" and as sheet striae or "ream." Their optical properties are,

⁴⁵ See page 720 of BS Sci. Pap. **22**, (1928) S572.

⁴⁶ A. Q. Tool and C. G. Eichlin. *J. Opt. Soc. Am.* **4**, 359 (1920); BS J. Research **6**, 525 (1931) RP292.

⁴⁷ A. Q. Tool, L. W. Tilton, and E. E. Hill, *J. Optical Soc. Am. and Rev. Sci. Inst.* **12**, 490-491 (1926). L. W. Tilton, *J. Wash. Acad. Sci.* **20**, 12-13 (1930). See also Annual Report of the Director of the Bureau of Standards, U. S. Department of Commerce, pages 11, 23, 21, and 25, of years 1925, 1927, 1928, and 1929, respectively.

⁴⁸ *Z. tech. Physik* **7**, 282-287 (1926).

⁴⁹ L. W. Tilton, A. N. Finn, and A. Q. Tool, BS Sci. Pap. **22**, 719 (1928) S572.

of course, different from those of the adjacent media, and apparently there is a rather abrupt transition⁵⁰ at the interfaces. According to prevailing ideas⁵¹ it may perhaps be assumed that in general the principal and only important effect is a scattering of light, which results in loss of contrast and consequent poor definition of the image of the collimator slit. This is probably true if the striae are present mainly as cords and if they do have abrupt boundaries.

On the other hand, when the striae consist of well-stratified layers or extensive sheets of "ream" the conditions are quite different. Unless the planes of stratification are approximately parallel to the light paths, all of the rays traverse striae and suffer a consequent acceleration or retardation, but it is usually found that an image of the slit is fairly well outlined even when one uses prisms in which such striae are very numerous. If the striae are so thin, or the difference in refractive index so small that beams emerging from adjacent kinds of glass differ in phase by less than one-quarter wave length, then the measured deviation produced by such a composite prism corresponds to that for a similar prism having a fictitious index, n_3 , intermediate in value between n_1 and n_2 , indices of the "normal" glass and the striae, respectively. In fact, if s is the proportion by volume of the striae present, one may write

$$n_3 = n_1 + s(n_2 - n_1) \quad (37)$$

as an estimated condition which seems probable for the case of a quasi regular composite prism of this type.

Since striae may differ in index from the normal glass by 3×10^{-4} (an average of some values which were observed by Smith, Bennett, and Merritt) it seems that the presence of certain types of striae in amounts exceeding 0.3 percent of the whole volume of glass should be considered a potential source of errors that may appreciably affect the sixth decimal place of refractive-index determinations.

IV. REQUISITE PRECISION IN SOURCE OF RADIATION

It is desirable to know to what extent doublets and complex lines may be used in sixth-decimal-place refractometry, and to decide whether or not it is necessary to specify the conditions under which a source of radiation is operated. For such purposes it is sufficient quantitatively to consider the dispersion in relative index for a few highly dispersive substances.

If the simple Cauchy dispersion formula

$$n - 1 = a + \frac{b}{\lambda^2} \quad (38)$$

is written to approximate (with two constants) the result of experiments over a limited spectral interval it is evident that

$$\frac{dn}{d\lambda} = -\frac{2b}{\lambda^3} \quad (39)$$

⁵⁰ A. A. Michelson, BS Sci. Pap. 15, 41 (1919) S333.

⁵¹ T. T. Smith, A. H. Bennett, and G. E. Merritt, BS Sci. Pap. 16, 75 (1920) S373. A. Arnulf, Rev. optique 6, 6-20 (1927).

can be written to show how refractive index varies with wave length within the specified interval. Consequently, one may consider

$$T_{\Delta\lambda} = \pm \frac{5\lambda^3}{b \times 10^7} \quad (40)$$

as a suitable tolerance in wave length of source for a limiting error of $\pm 1 \times 10^{-6}$ in index.

For use with wave lengths measured in angstroms the constant b is seldom as large as $+25$ or $+30 \times 10^5$ (values for carbon disulphide and densest flint glass of index $n_D = 1.92$, respectively, computed for the shortest wave-length region of the visible spectrum) and thus any uncertainty or variation in the source of less than ± 0.01 angstroms is not of consequence for wave lengths longer than approximately 4000 Å. For the great majority of substances on which refractive indices are precisely determined, the tolerance is appreciably more liberal. Water and fluorite, for example, have the corresponding minimum or safe tolerances of ± 0.10 and ± 0.13 Å, respectively.

V. PRECISION AND ACCURACY ATTAINABLE IN MINIMUM-DEVIATION REFRACTOMETRY

In this and in previous papers⁵² many of the difficulties of precise prism refractometry, particularly by the minimum-deviation method, have been discussed. The number, N , of sources of error is fairly large. At least nineteen of them have been quantitatively discussed in these investigations and the discussions are briefly summarized in table 5. Several of the errors, say $(N - N_1)$ of them, are easily held within limits which are small compared with $\pm 1 \times 10^{-6}$ in index. In no respect is it found impossible, and in few if any instances does it seem unreasonably difficult to control or correct within that tolerance limit. With the exception of error arising from incorrect prism orientation, practically all contributions to the final error in index are equally likely to have positive or negative signs in any extensive and well-arranged observational program. Consequently the algebraic sum is never large.

If, as an approximation to the worst case, N_1 of the contributed errors are large compared with the rest and are all about equal in absolute value, say 1×10^{-6} , then the probable error in the final index is $\sqrt{N_1} \times 10^{-6}$. For several years the individual tolerances corresponding to $\pm 1 \times 10^{-6}$ have been used in this refractometric laboratory and it seems that probable errors in refractive indices precisely determined by the minimum-deviation method seldom exceed ± 2 or 3×10^{-6} . Hence the existence of some five or ten fairly important sources of accidental error is inferred. Whenever less precision is obtained after the expenditures of reasonable effort in the use of fairly good equipment, the difficulty is probably to be sought not in any one or two particularly important sources of error but in a combination of several matters which have been overlooked or deliberately neglected as inconsequential.

⁵² See references listed in table 5.

TABLE 5.—Summary of sources of refractive-index error that have been quantitatively discussed

References to BS J. Research		Quantity evaluated or controlled				±Tolerances for various refractivities of sample ($\Delta n = \pm 1 \times 10^{-6}$, $A = 60^\circ$)				Provisory remarks ¹
Volume	Page	Formula	Name	Sym- bol	Unit	$n=1.3$	$n=1.5$	$n=1.7$	$n=1.9$	
1929	8		Prism angle	ΔA	Second of arc	0.57	0.33	0.22	0.16	
2	9		Double deviation	$\Delta 2D$	do	0.56	0.62	0.80	1.5	
1931	72		Prism orientation	P, E, i	Minute of arc	3.9	3.1	2.7	2.4	(Index error, always+).
1933	41		Prism surface sagitta	s'	Wave length	0.27	0.14	0.08	0.04	1 cm diam. of surface area.
11	42		Prism translation	P, E, ϵ	Millimeter	1.3	0.7	0.4	0.2	$s' = 0.02\lambda$
11	47		Eccentricity of prism-table axis	ϵ	do	0.3	(Very large)	0.2	0.1	$s' =$ tabulated values.
11	52		Collimator focusing	ΔFc	do	2.9	2.0	1.8	2.7	$f_s = 400$ mm, $\epsilon = 0.2$ mm.
1934	13		Air composition		Proportion by volume		(Relatively large tolerances—see reference cited)			CO ₂ -free dry air.
1935	14	10	Air temperature	Δt_a	Degree C	0.77°	0.67°	0.59°	0.53°	$t = 0^\circ$ C.
14	400	12	Air pressure	ΔP_a	Millimeter of Hg	2.2	1.9	1.6	1.5	
14	401	16	Air humidity	Δh	do	15.	13.	12.	11.	
14	403	21	Abnormal CO ₂ in air	Δc	Proportion by volume	0.005	0.004	0.004	0.003	
14	408		Temperature of certain glasses		Degree C		{ Soda lime 0.3° Fused quartz 0.1° Many oils 0.002°	Dense flint 0.2° CS ₂ 0.001°	Densest flint 0.07°	$\lambda = 5893$ A. $\lambda = 5893$ A.
14	408		Temperature of certain liquids		do	Water 0.01°				
14	411	32	Hydrostatic pressure on glass	ΔP	Atmosphere		0.5	0.5	0.5	
14	411	33	Hydrostatic pressure on certain liquids	ΔP	Millimeter of Hg	{ Water 47. Ether 12.	Glycerin 72. Benzene 17.	CS ₂ 12.		
14	412	36	Stress-birefringence in glass	Δn_b	mm/cm		9.	3.	2.	(λ at $n = 1.8+$).
14	415	37	Striae in glass	s	Proportion by volume	0.003	0.003	0.003	0.003	$n_2 - n_1 = 0.0003$.
14	416	40	Wave length of light source	$\Delta \lambda$	Angstrom	Water 0.10	Fluorite 0.13	CS ₂ 0.014	Densest flint 0.010	$\lambda = 4000$ A.

¹ For explanations and for other pertinent limitations and conditions, see discussions given in references cited in columns 1 to 3

It is a much more difficult matter to state precisely what accuracy is attainable. Tests of the accuracy of angle measurement by the use of a prism polished on all three faces are of fundamental importance. Torsion and mechanical strains in the goniometer constitute a particularly dangerous source of systematic error which is seldom entirely constant and cannot always be effectively eliminated. Fortunately, those errors which would be systematic if neglected are automatically replaced by residual accidental errors when proper corrections can be and are applied. In this laboratory no reason has been found for suspecting that residual systematic errors in precise index measurement with a spectrometer are materially in excess of the precision which is attained.

WASHINGTON, January 26, 1935.

Memorandum on a Procedure for Obtaining Spectral Radiant Intensities of Tungsten- Filament Lamps, 400–700 m μ

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WITHIN recent months there has been evidence of increased need for standards of spectral radiant intensity. The purpose of this memorandum is to point out that within the spectral range 400 to 700 m μ , a tungsten-filament lamp calibrated for luminous intensity and for color temperature can be used as such a standard. The values of spectral radiant intensity of such a calibrated lamp can be calculated by the procedure to be described in which the following constants are used,

$$C_1 = 374 \times 10^{-14} \text{ watt-cm}^2,$$

$$C_2 = 1.438 \text{ cm-deg.}$$

Luminance of a blackbody at the freezing point of platinum = 60 candelas/cm². Temperature of freezing platinum = 2042°K.

Table 75 of the *Smithsonian Physical Tables*, ninth revised edition, gives for a series of blackbody temperatures the spectral luminous intensity per 10 m μ band per unit total luminous intensity. For example at 2800°K, it lists at 550 m μ a value of 0.08517 candle/10 m μ which is equivalent to 0.08517 lu/sterad-10 m μ . Since the maximum spectral luminous efficacy* of radiant flux is 680 lu/w, and since 0.995 is the luminous efficiency* of radiant flux of wavelength 550 m μ , 1 w at 550 m μ yields $0.995 \times 680 = 677$ lu/w. The radiant power corresponding to 0.08517 lu is thus $0.08517/677 = 126 \times 10^{-6}$ w, and the spectral radiant intensity at 550 m μ of a lamp of color temperature 2800°K is 126×10^{-6} w/sterad per 10 m μ for each candela of the lamp. The spectral radiant intensities at other wavelengths may then be found by the use of tables of relative spectral radiant intensities of blackbodies such as Table 55 of the *Smithsonian Physical Tables* referred to previously (100 m μ intervals) or Harvard University Computation Laboratory "Tables of blackbody radiation functions" (10 m μ intervals).

The uncertainty of spectral data computed by this procedure is probably no greater than 3% but may be as much as 5%. This estimate of total uncertainty is based upon the following estimates of contributing uncertainties: 2% from the assumption of a blackbody distribution within the specified range of wavelengths,¹ 2% from uncertainty in the determination of color temperature, and 1% from photometric uncertainty.

For convenience Table I gives values of spectral radiant

TABLE I. Microwatts per (sterad-10 m μ -candela) at the designated color temperatures (°K).

Wave-length m μ	2300	2400	2500	2600	2700	2800	2854
400	8.0	9.8	11.7	13.9	16.2	18.6	20.0
410	10.4	12.5	14.7	17.2	19.8	22.5	24.1
420	13.3	15.7	18.2	21.0	23.9	26.9	28.6
430	16.7	19.4	22.5	25.4	28.5	31.8	33.6
440	20.7	23.7	27.0	30.3	33.7	37.2	39.1
450	25.3	28.7	32.2	35.8	39.4	43.1	45.1
460	30.7	34.4	38.1	41.9	45.7	49.5	51.5
470	36.8	40.7	44.6	48.6	52.5	56.3	58.4
480	43.7	47.8	51.9	55.9	59.8	63.7	65.7
490	51.4	55.6	59.7	63.7	67.6	71.4	73.5
500	60.0	64.2	68.3	72.2	76.0	79.6	81.6
510	69.4	73.5	77.5	81.2	84.8	88.2	90.0
520	79.8	83.7	87.4	90.8	94.1	97.2	98.8
530	91.0	94.5	97.9	101	104	106	108
540	103	106	109	111	114	116	117
550	116	118	121	123	124	126	127
560	130	132	133	134	135	136	136
570	145	145	146	146	146	146	146
580	160	160	159	158	157	156	156
590	177	175	173	171	169	167	166
600	194	190	187	183	180	177	176
610	212	206	201	196	192	188	186
620	230	223	216	210	204	198	196
630	249	240	231	223	216	209	206
640	269	257	246	236	227	219	215
650	290	275	262	250	239	230	225
660	310	293	277	263	251	240	234
670	332	311	293	277	262	250	244
680	353	329	308	290	274	260	253
690	375	348	324	303	285	269	262
700	397	366	340	317	296	279	270

intensities per candela computed by the procedure described previously for seven color temperatures. Wavelengths slightly longer than 570 m μ will be recognized from the table as the Crova wavelengths for the color temperatures tabulated.

* These two terms are used herein in accord with their definitions as follows recently adopted by the Illuminating Engineering Society (see *IES Lighting Handbook*, 3rd edition, pp. 3-6). *Spectral luminous efficacy, K λ* : the ratio of the luminous flux at a given wavelength to the radiant flux at that wavelength. It is expressed in lu/w and is equal to 680 (lu/w) times the spectral luminous efficiency data. The spectral luminous efficiency for a particular wavelength is the ratio of the spectral luminous efficacy for that wavelength to the value at the wavelength (555 m μ) of maximum spectral luminous efficacy.

¹ Deane B. Judd, J. Research, Natl. Bur. Standards **44**, 5 (1950), RP2053.

Parallel Testing Interferometer

James B. Saunders

The conventional methods of testing the parallelism of opaque bodies, such as gage blocks, by interferometry require wringing of the body to an optical flat. This operation disturbs the temperature equilibrium, necessitating long periods between tests, especially for long blocks. It often injures the surfaces of both the optical flat and the test body. Also, if the body is a standard gage block, repeated wringings during use ultimately change the dimension. This paper describes an interferometer for measuring the parallelism of gage blocks and other bodies of any reasonable length without the necessity of the wringing operation. Two forms of this instrument are used—one for testing long blocks and another for testing short blocks. Either form can be constructed for testing blocks of any length, but two forms are found to be more practical.

1. Introduction

The conventional procedures for measuring the parallelism of gage blocks^{1,2} require the wringing of the blocks onto an optical flat. The wringing operation often injures the contacted surfaces and repeated wringings necessitate frequent refinishing of the optical flat that is used as a base. A method for measuring parallelism, without the wringing operation, has significant advantages because the danger of injury to the contacted surfaces is eliminated. Accordingly, two instruments that utilize this method are described: one is for testing very long blocks and similar bodies, whereas the other is designed for short blocks. Both instruments use low orders of interference and neither requires the use of a standard.

2. Optics of the Interferometer

A description of the optics for either form of this instrument covers a large portion of that for the other. For distinction we will designate them as "the long-block interferometer" and "the short-block interferometer". The double-image prism used in these instruments is adjusted during construction³ so that a ray of light, shown in the plane of figure 1A, after division into two component rays, 1 and 2, at P_0 will, on reflection at P_1 and P_2 , lie in planes that are parallel to the semireflecting plane of the prism but deviate equally toward or from opposite sides of the plane of figure 1A. The projection of the light rays on the dividing plane is shown in figure 1B, which is perpendicular to the plane of figure 1A. This deviation is effected by rotating one component of the prism relative to the other about an axis normal to the dividing plane of the prism. If this deviation is held constant, the width of the interference fringes in the direction normal to the plane of figure 1A is fixed. This component of fringe width is, therefore, frozen into the system⁴ when the cement between the component prisms

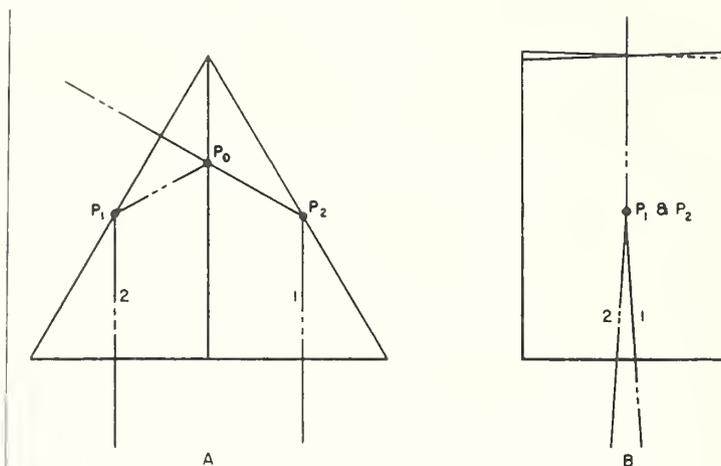


FIGURE 1. Kösters double-image prism.

A small angle is formed by the two 30° edges shown exaggerated in B.

becomes hard by cooling after adjustments are complete. Furthermore, the tilting of any plane surface outside the prism that affects the two component beams between division and recombination, will produce equal effects in this direction and, consequently, will not affect the fringe width. However, the rotation of plane surfaces about an axis normal to the plane of figure 1A will produce equal effects on the two component beams but in opposite directions, thus producing a proportionate effect on the fringe width in the direction parallel to this plane.

Because of the above-described properties of this prism, adjustments of the instrument in which it is used affect the fringe width in one direction only. Consequently, when measuring the parallelism of gage blocks the test can be applied to parallelism in only one direction at a time. To test for parallelism in other directions the block must be rotated.

2.1. Long-Block Interferometer

Figure 2A is a horizontal section through the optical elements of this instrument. The light from a source at $S_1(S_2)$ is collimated by lens $L_1(L_2)$ and divided into two equal components by the beam-dividing plane $B_1(B_2)$. Each component suffers total internal reflection in the prism and emerges in planes

¹ Gauges and fine measurements, by F. H. Rolt, I, p. 204 (Macmillan and Co., Ltd., London 1929).

² The science of precision measurement, by The DoAll Co., p. 143 (1953).

³ Construction of a Kösters double-image prism, by J. B. Saunders, J. Research NBS 58, 21 (1957) RP2729.

⁴ NBS Tech. News Bul. 42, 30 (1958).

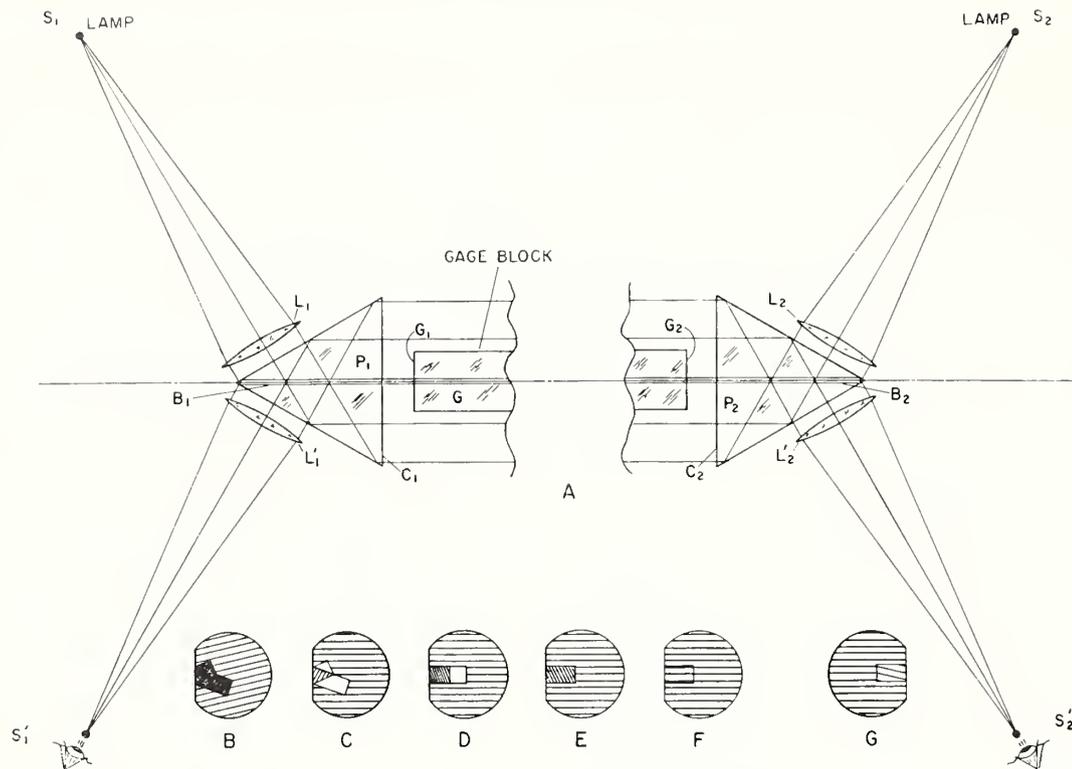


FIGURE 2. Optics of interferometer for testing parallelism of long blocks.

Figures B through G represent observed interference fringes.

parallel to $B_1(B_2)$, but at a small angle to the base surface $C_1(C_2)$. The latter condition is obtained by a slight rotation of $P_1(P_2)$ about an axis normal to the dividing plane $B_1(B_2)$. This serves to permit the elimination of light reflected from $C_1(C_2)$.

The two prisms, P_1 and P_2 , are separated by a distance exceeding the length of the longest block to be tested. If desired, this distance may be made adjustable. The two dividing planes, B_1 and B_2 , are adjusted to be coplanar and the two base faces, C_1 and C_2 , are adjusted parallel to each other. A line joining the centers of P_1 and P_2 is adjusted to form a small angle with the normal to faces C_1 and C_2 .

The light emerges from prism $P_1(P_2)$ as two separated components, one on each side of the dividing plane. It enters prism $P_2(P_1)$, again suffers total internal reflection, and each pair of component rays recombines in the plane of $B_2(B_1)$. One-half of each beam proceeds to the neighborhood of source $S_2(S_1)$ and the other half to $S_2'(S_1')$. An observer at $S_2'(S_1')$ sees a set of interference fringes that cover the entire aperture.

If a gage block, G , is inserted in the position shown with its end face, G_1 , adjusted normal to the light beams, it will reflect equal and corresponding parts of the two component light beams from S_1 back through P_1 to S_1' . Accordingly, the observer at S_1' sees a background set of fringes, produced by light from S_2 and another set on the face of G_1 , that is produced by light from S_1 . Since G_1 is normal to the light beams these two sets of fringes will be parallel to each other and to the plane of figure 2A.

If the other end, G_2 , of the gage block is parallel to G_1 , the fringes seen at S_2' will likewise be parallel to each other and to the plane of figure 2A. If, however, G_2 is not parallel to G_1 , in the plane of figure 2A, it will not be normal to the light beams, and the sets of fringes seen at S_2' will not be parallel to each other. The angle between these two sets of fringes is a measure of the angle between G_1 and G_2 in the plane of figure 2A. The component of the angle between G_1 and G_2 that is perpendicular to the plane of figure 2A (or horizontally in figs. 2B to 2G) does not affect the fringes because it affects all pairs of component beams equally. If the component of the angle between the gage-block surfaces that is normal to the plane of figure 2A (or vertically in figs. 2B to 2G) is desired, the block must be rotated 90° and the operation repeated.

Since each prism is adjusted for complete compensation in the plane of figure 2A, white light can be used. A measure of the vertical width of the fringes (perpendicular to fig. 2A) for a known monochromatic light, with a micrometer eyepiece at S_2' , gives a calibration of the micrometer scale in units (microns, millionths of an inch, etc.) of length for measuring the displacement of white-light fringes from a chosen reference point on the gage-block surface.

The procedure for adjusting a gage block is explained with the aid of inserts in figure 2. In general, when the block is placed on its supports, the light reflected from its end will not reach the observer because of excessive angular deviation from the

eyepiece. The block will appear in silhouette, as indicated in figure 2B. When the surface G_1 is adjusted approximately normal to the light, fine fringes will usually be visible in the area covered by both images of it, as shown in figure 2C. The images of the two parts of G_1 are made to coincide by rotating G about the center line of figure 2A, causing the image to change from that of figure 2C to figure 2D. A lateral motion, without rotation, will then change the image from that shown in figure 2D to figure 2E. A further small rotation of G about an axis normal to figure 2A brings G_1 normal to the light and the fringes on G_1 will appear horizontal and parallel to the background fringes as shown in figure 2F. The observer then moves to position S_2' and observes the set of fringes shown in figure 2G. The angle between these two sets of fringes corresponds to the angle between surfaces G_1 and G_2 .

A photograph of the long-block interferometer is shown in figure 3. The base of the instrument is designed for rigidity so as to avoid flexure. Rigidity is quite important. The two prisms are mounted on rigid tables at each end of the base. Three screws (D_1 and two others not shown) permit raising and lowering of prism P_2 . They also permit rotation or tilting of this prism about any chosen horizontal axis. The prism housings are fastened to the table tops by means of large-headed screws, in oversize holes, that permit lateral adjustments of the prisms relative to each other. Two screws, D_2 and another concealed by the housing of prism P_2 , permit small rotations of this prism about a vertical axis by applying lateral torques to the legs of the table. Similarly, screws D_3 and D_4 permit rotary adjustments of the other prism housing.

The adjustments described above permit the alinement of the two prisms. This adjustment is critical, rather difficult to attain, but when once obtained is very stable. The final adjustments are executed while observing interference fringes produced by lens L'_2 , in one of the many images of the

source S_2 . This author uses a pinhole source of approximately 1-mm diam and a short-focus lens to observe its image at S_2' . There are two sets of fringes, superimposed upon each other, in the proper image to be used. When these two sets of fringes are horizontal and very broad, fringes can be seen with the two eyepieces when focused on the pinhole. These fringes, in white light, are horizontal with the zero order in the center of the field.

The supports on which the long blocks rest are located at the Airy points so as to reduce changes in the angle between G_1 and G_2 due to gravitational distortion.⁵ These supports rest on an adjustable plate which, in turn, is supported at one end by two points and at the other by one point. This plate is adjustable at one end, laterally with screw D_5 and vertically with another screw D_6 which is concealed in figure 3.

2.2. Short-Block Interferometer

The optics of the short-block interferometer are shown in figure 4. The double-image prism, lenses light source, and viewing position in figure 4B are identical to that of either end of the long-block instrument described above. A reflecting prism, P_R in figure 4, replaces one of the prism assemblies of the long-block instrument. Also, the optical axis of the instrument is vertical instead of horizontal. Figure 5 is a photograph of the short-block interferometer. Figure 4A may be considered a section through the center of 4B, coincident with the dividing plane of prism P . The indicated rays, 1 and 2 in figure 4A, however, do not lie in this plane. Their positions relative to it are indicated in 4E, which is a vertical view through 4B. The two surfaces, G_1 and G_2 , of the gage block (figs. 4A and 4D) appear as G and G' in figure 4E. G'_2 in figure 4A is an image of G_2 as seen by light reflected from the right-angle prism, P_R .

⁵ F. H. Rolt, *Gauges and fine measurements* II, 340 (Macmillan and Co., 1929).

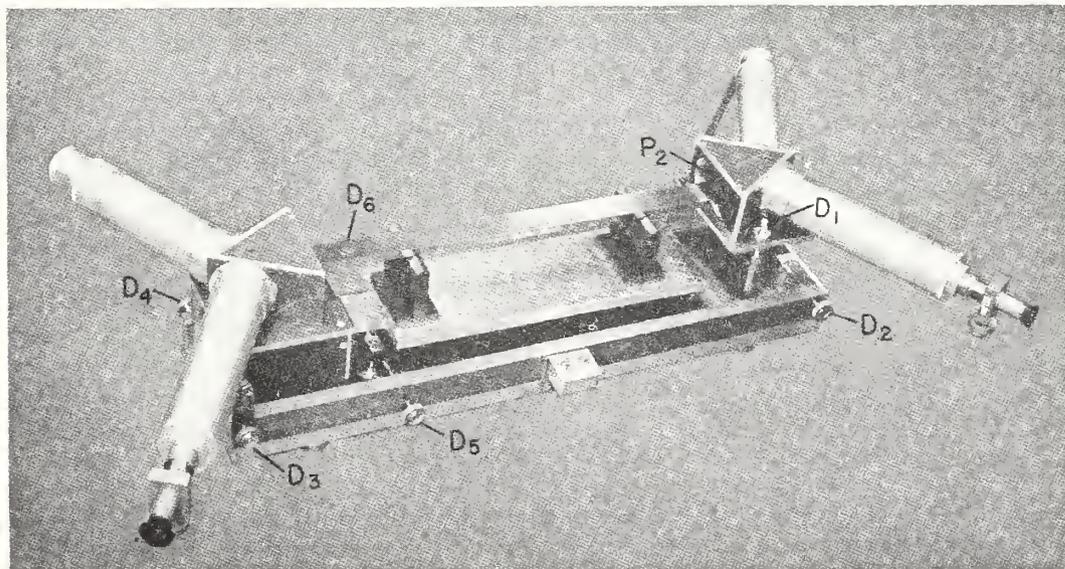


FIGURE 3. Photograph of the long-block interferometer.

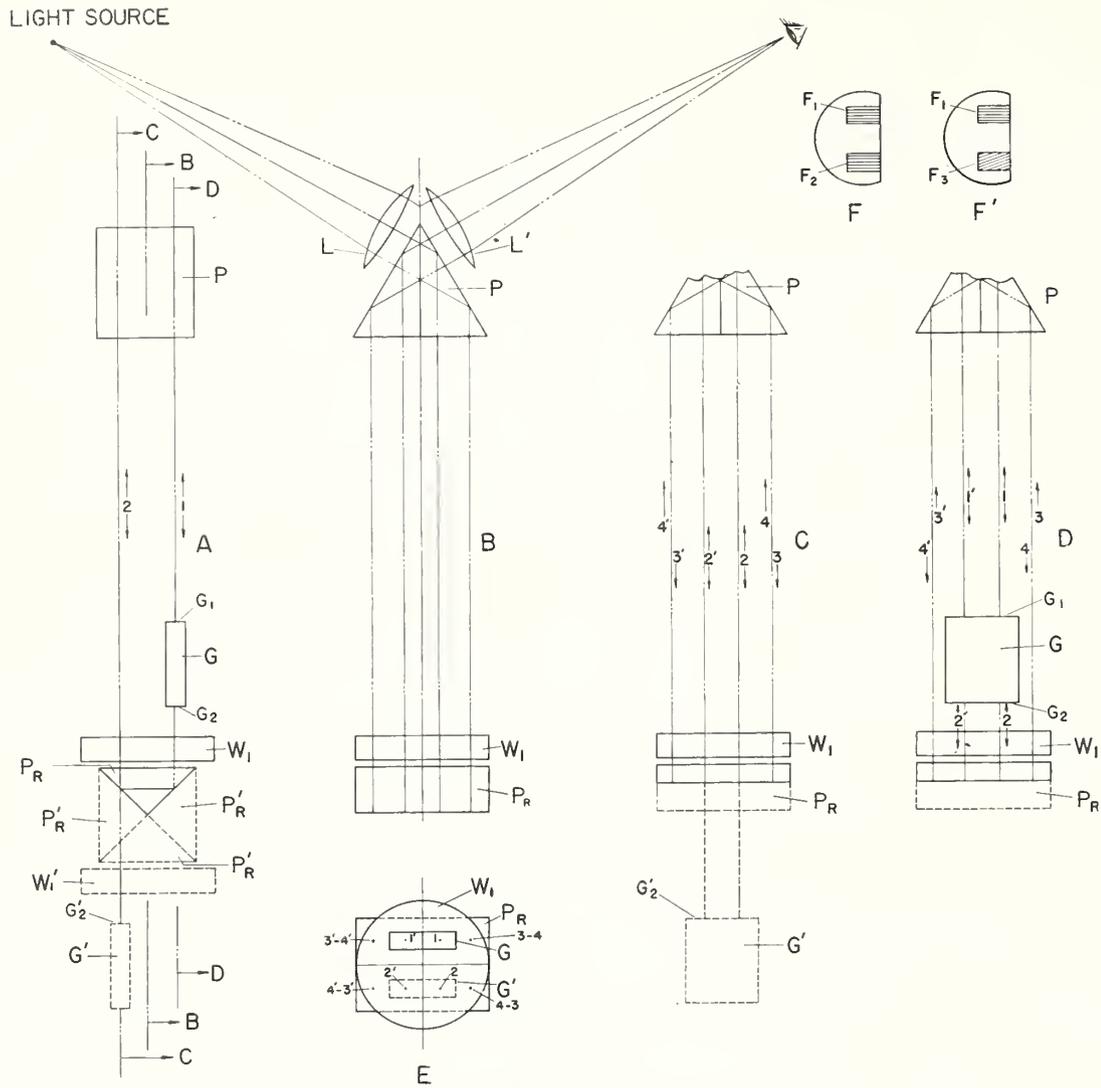


FIGURE 4. Optics of the interferometer for testing parallelism of short blocks.

Figures B, C, and D are sections through A. Figure E is a vertical view. Figures F and F' represent interference fringes for a parallel and a nonparallel block, respectively.

A plane optical wedge, W_1 , and its image, W'_1 , (in figure 4A) are shown between G and its image G' . Figures 4B, 4C, and 4D are sections through 4A, along the lines indicated. The gage block is not located in the center as was the case in the instrument described above.

The two component rays of light, 1 and 1' (figs. 4A and 4D), are caused to reflect normally from G_1 by adjusting G with a leveling screw. They return into P where they recombine to produce the interference fringes, F_1 , shown in figure 4F. The two component rays, 2 and 2' (figs. 4A and 4C) are transmitted downward through the optical wedge, W_1 , suffer two internal reflections in P_R , and if P_R is properly adjusted, return upward and parallel to their directions of incidence through W_1 to G_2 .

The wedge, W_1 , is adjusted initially by rotation so that its thickness is constant at all points in either of the planes B, C, and D. When in this neutral position it does not affect the interference fringes because of compensation in each pair of component beams that pass through it. The function of W_1 will be explained later.

In order to measure the angle between G_1 and G_2 , the deviation of the light by P_R toward or from the dividing plane of P must either be reduced to zero or its effect eliminated by measuring the observed angle for two orientations of G , which are 180° apart. The light that is not intercepted by G forms an interference pattern of uniform tint (or color) that fills the background about and between the two images of the gage block, shown in figure 4F or 4F'. Figures 4F and 4F' represent the conditions observed when the ends of the block are parallel and nonparallel, respectively. A typical pair of component rays, which form this interference pattern, is indicated by 3 and 3'. They travel downward in figure 4C and upward in figure 4D. If the right-angle edge of P_R is normal to the dividing plane of P , the pair of rays 3 and 3' in figure 4C can be made to return in planes that are parallel to the dividing plane, by rotating P_R about an axis parallel to the plane of 4A and normal to the incident light. This condition is attained when the background fringe is infinitely broad. The direction of the background fringes, when not infinitely broad,



FIGURE 5. Photograph of the short-block interferometer.

remain parallel to the dividing plane, because for each pair of component rays, such as 3 and 3', there is a corresponding pair, 4 and 4', that travel identical paths but in opposite directions. The optical path differences are, therefore, equal to each other and also equal to that for any other pair of component rays in the plane of 4C and 4D. The order of interference along the dividing plane corresponds to the optical path difference that was introduced into the double-image prism by the built-in wedges at the point that corresponds to the point of intersection of the right-angle edge of P_R and the dividing plane of P. This point is located in the center of figure 4E.

In general, due to the inherent error of judging when the background fringes are infinitely broad and to imperfections in the optical elements, a more precise method of evaluating the wedge between G_1 and G_2 is to measure the wedge for two positions that differ by 180° . If the background fringes are unaltered, the instrumental errors will be equal for the two positions and the value of the wedge unchanged except in sign. Consequently, the algebraic difference yields twice the value of the wedge.

There are three ways that one might evaluate the wedge between G_1 and G_2 . The first is to rotate P_R until the order of interference at points C and E (fig. 6A) are equal; then rotate the gage block until the orders at A and B are equal; and finally, observe the difference in order of interference at points F and H. The second method is: After performing the above operations, instead of reading the order difference between F and H, re-

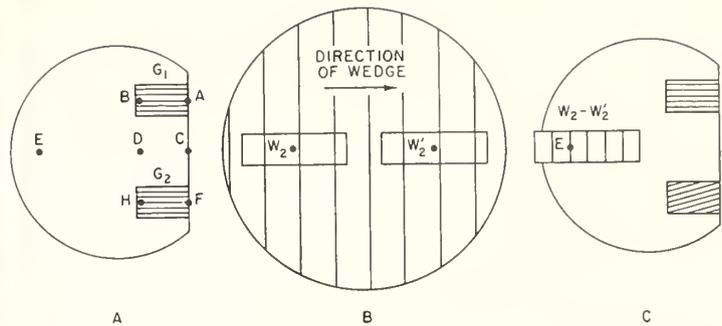


FIGURE 6. Figures A and C represent the positions of reference points relative to the different sets of interference fringes.

Figure B shows the wedge from which the two smaller wedges, W_2 and W_2' , were cut and the difference in optical thickness between them.

duce this order difference to zero by rotating W_1 and read the resultant change on a scale attached to W_1 , figure 4E. This scale may be calibrated with monochromatic light and the units may be radians, degrees, or the corresponding variation in height of the block. A third method is to leave the wedge in its neutral position, adjust G so that the orders of interference of A and B are equal, and change the order at H to equal that at F by rotating P_R about an axis normal to the incident light and parallel to the plane of figure 4A. The order of interference between two points such as C and D (fig. 6A) after rotating P_R will be equal to one-half of that between F and H before this rotation was performed. By choosing a point, such as E in figure 6, such that CE equals K times CD, the order difference between C and E will be K times that between C and D.

When using this last method for testing gages that are almost parallel, the angle between G_1 and G_2 will be small and the background fringes will be too broad for reading fractions of fringes. To eliminate this difficulty, an optical wedge, illustrated in figure 6B, is constructed and from it two sections W_2 and W_2' are cut and placed on P_R as shown in figure 7. The wedges W_2 and W_2' are equal, but when placed in the position shown, the effect is to narrow the background fringes seen through them. The results are illustrated in figure 6C. The difference in thickness of W_2 and W_2' at a selected reference point E (fig. 6C) is determined by the choice of the corresponding positions on the plate from which they were cut. This difference in thickness is chosen so as to cause the zero order of interference to pass through the chosen point when the background fringes about W_2-W_2' are infinitely broad.

If the angles of wedges W_2 and W_2' are properly chosen, the width of the fringes seen through them will be most favorable for measuring the fractional parts of fringes. Also, the position of the zero-order fringe, relative to point E, may be calibrated to read directly the angle between the ends of the gage blocks.

The recommended procedure for measuring a block is: (1) Adjust the two sets of fringes seen on the ends of the block so that they are perpendicular to the dividing plane, as in figure 4F; (2) note the position

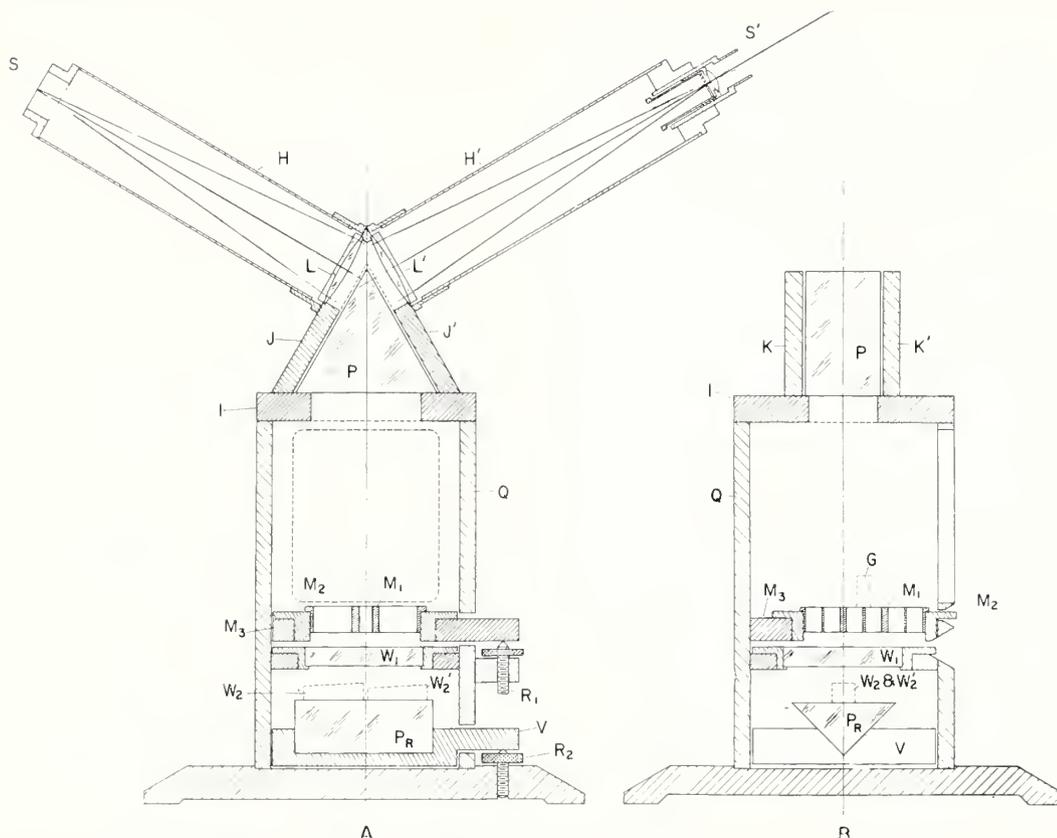


FIGURE 7. Two vertical sections, at right angles to each other, through the short-block interferometer.

of the zero-order fringe (or absolute order at E, fig. 6C); (3) rotate the block 180° about a vertical axis through its center; (4) readjust the fringes to restore the condition of (1) above; (5) again note the position of the zero-order fringe (or absolute order at E); (6) the difference in the two observed orders at E, or positions of the zero-order fringe, multiplied by the constant K, described above, is a measure of the angle between the ends of the block.

Figure 7 shows two vertical sections through the center of the short-block interferometer that are mutually perpendicular to and through the centers of each other. A pinhole, S, illuminated either with monochromatic or polychromatic light from outside the tube H, serves as source. The position of the pinhole is adjustable in the focal plane of the collimator lens, L, and the collimator tube is adjustable in length. The prism, P, rests on a thick plate, I, to which is fastened the lens-holding plates, J and J'. The plates, K and K', which cover the ends of P, are not fastened to J or J'. Consequently, small stresses applied to tube H' while adjusting the eyepiece or manipulating a micrometer in it, are not transmitted to P.

The material of the instrument, except for the collimator, the eyepiece tubes and optical elements, is made of steel. Steel was chosen because its expansivity approximates that of the glass elements more nearly than other usable materials.

The gage block, G, rests on a rotatable plate, M₁, that is perforated so as to transmit the required parts of the light beams used for making measurements. See also figure 8 for a vertical view of M₁ and its supporting parts. The plate, M₁, rotates in an annulus ring, M₂, which in turn is rotatable, from outside the instrument, in another annulus, M₃. The aperture in M₃ has its center displaced from the center of the instrument in a direction parallel to the dividing plane of P and by an amount equal to one-half the horizontal separation between the centers of the gage block, G, and its image, G'. The gage block rests on the center of M₁ (see fig. 6B). Accurate placement of the block is facilitated by stops.

The eccentric annulus, M₃, has an arm that projects through the wall, Q, of the instrument and is supported by this arm at one of its three supporting points by an adjusting screw, R₁. The other two supports for M₃ are steel balls, T₁ and T₂, (fig. 8B), which are held in conical holes by means of two screws, U₁ and U₂, respectively. The ends of these screws have eccentric conical depressions that permit a limited amount of rotation of M₃ about an axis normal to the dividing plane of P. The screw R₁ permits fine adjustment of M₂, and consequently the gage block, which it supports, about a horizontal axis parallel to the dividing plane of P. A similar pair of balls, screws, and the adjustable screw R₂, permits rotation of P_R about two axes parallel to those used for adjusting M₃.

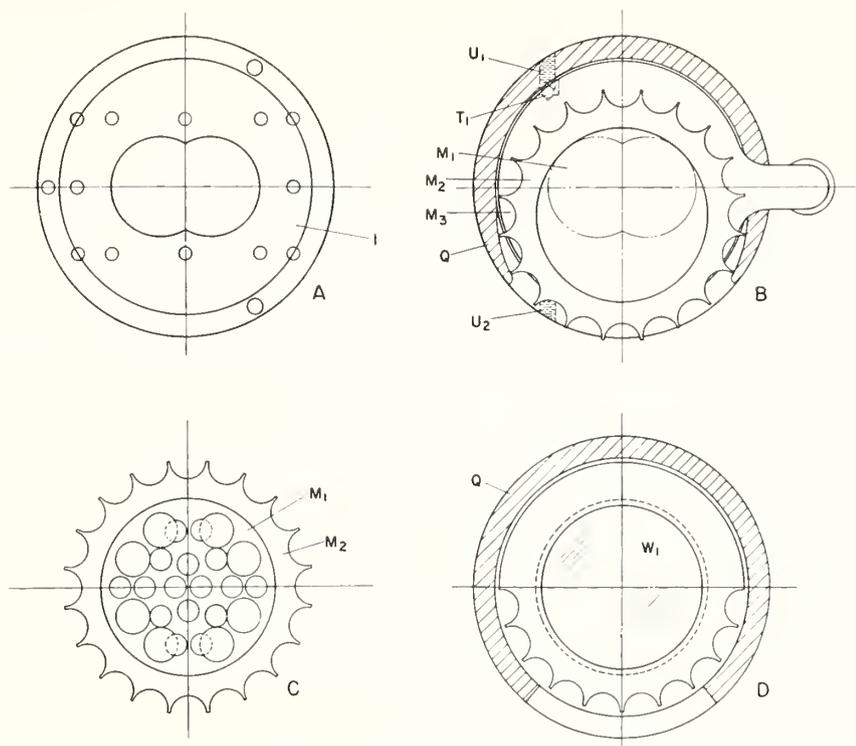


FIGURE 8. Several views showing individual parts of the short-block interferometer.

If the refracting edge of the optical wedge, W_1 , is made perpendicular to the dividing plane of P (i. e., parallel to the plane of fig. 7B), each pair of component rays will traverse this plate at points of equal thickness. Consequently, W_1 in this orientation, does not affect the fringes of interference. It does, however, serve as a window, protecting the prism P_R from the accumulation of dust. Other functions of W_1 will be discussed later.

The prism, P , is centered over the aperture in plate I (figs. 7A and 8A) with its ends parallel to plates K and K' (fig. 7B). Using the base surface of P as a plane mirror, the pinhole aperture S is located in the focal plane of lens L by varying the length of tube H and at a point in this plane where the light will form an image of the pinhole upon itself. This places the light beams, after division at the dividing plane, normal to the prism base and, consequently, parallel to each other.

A gage block, whose end faces are parallel to each other, is placed on the center of plate M_1 with its lower surface parallel to the top surface of M_1 . When the surface of the block is adjusted parallel to the base of P by means of screws R_1 , U_1 , and U_2 , the light from S is reflected normally from the top surface of the block and observed at the eyepiece as interference fringes.

The parts of the light beams that are not intercepted by the block and its support M_1 , traverse the optical wedge W_1 , and enter the right-angle prism P_R . After two internal reflections in P_R , the light returns along a path that is symmetrical to its incident path with respect to the 90° edge of P_R . The 90° edge

of P_R is made normal to the dividing plane of P by means of screws in its support similar to U_1 and U_2 and the fine-adjusting screw R_2 (fig. 7A). This light forms the background fringes used in the test, but no interference is observed until this prism edge is nearly normal to the dividing plane. When it is not normal the two images of this edge, formed by the two component light beams, intersect in the extension of the beam dividing plane. The prism P_R is rotated until the two images coincide—perfection being attained when the background fringes have maximum contrast.

The above-mentioned contrast in the background fringes is not affected by screw, R_2 , since it has no vertical rotational effect on the prism. Its effect is to change the width of the background fringes only. When the background fringes are made infinitely broad, the collimated beam of light returns toward P parallel to the incident beam—all rays having suffered a horizontal shift in P_R , as illustrated in figure 4A.

The holes in the gage-block support, M_1 (fig. 8C), are so spaced that when M_1 is rotated to one of the four positions for which the rectangular sides of the block are either parallel or perpendicular to the dividing plane, all light that goes through M_1 returns again through it. That is, the apertures in M_1 are symmetrical both with respect to the dividing plane and to the 90° edge of P_R . Parts of the beam (rays 3, 3', 4, and 4' in fig. 4) will pass downward through M_1 to P_R , shift horizontally in P_R , and pass upward through other apertures in M_1 to P . Other parts of the beam (rays 2 and 2' in fig. 4) will pass downward through M_1 , shift horizontally in P_R , pass

upward through M_1 to the lower surface of the gage block, return through M_1 to P_R and again upward through M_1 to P . If the two end faces, G_1 and G_2 , are parallel, the light will be incident on G_1 and G_2 at equal angles. When G_1 is adjusted normal to the light, G_2 will also be normal to it.

We have three sets of fringes, shown in figure 4F', to consider: (1) The set F_1 is formed by light reflected from the top surface, G_1 , of the gage block; (2) the background fringe between and about F_1 and F_2 , formed by light reflected from P_R but not incident on the gage block; and (3) the set F_3 formed by light reflected from P_R to G_2 and back through P_R . The direction or orientation of F_1 determines the angle between G_1 and the incident wavefront; the width of the background fringes determine the direction between the incident and reflected beams to and from P_R ; and the orientation of F_3 determines the angle between G_2 and the wavefront that is reflected from it.

The set of fringes, F_1 , is adjusted by means of R_1 normal to the dividing plane of P , for which condition G_1 is normal to the incident light. The background fringe is made infinitely broad, for which condition the light beams returning from P_R are parallel to the incident beams. If the two surfaces of G are parallel, the set of fringes appearing on G_2 will be parallel to those on G_1 and indicated as the set F_2 in figure 4F. If G_1 and G_2 are not parallel, the fringes seen on them and indicated as F_1 and F_3 in figure 4F' will not be parallel to each other. The angle between these two sets of fringes is a measure of the angle between the two ends of the gage block.

WASHINGTON, May 5, 1958.

Interferometer for Large Surfaces

James B. Saunders and Franz L. Gross¹

An interferometer is described that permits the testing of large areas, such as layout plates. The extension to large areas is obtained by causing a collimated beam of light to reflect from the specimen at a large angle of incidence. The resultant fringe pattern is a contour map of the surface relative to an arbitrarily chosen plane and the contour interval is a function of wavelength and angle of incidence.

1. Introduction

An interferometer for testing large surfaces has been described by Linnik.² However, in spite of certain advantages, the Linnik interferometer has not been used extensively in this country. This lack of application may be due to the complexity of the instrument.

The instrument described here is relatively simple and easy to operate, is twice as sensitive as the Linnik interferometer, and is relatively free from vibrations.

2. Optics of the Interferometer

The optics of this interferometer are shown schematically in figure 1. The light from a source at *S* is collimated by the lens *L*₁, and separated into two coherent beams by the semireflecting dividing plane of a Kösters double image prism.^{3,4} The entrance angle of the collimated light may be adjusted to give any desired deviation θ between the component beams 1 and 2. Beam 1 is reflected normally from mirror *M*₁ and returns upon itself. Beam 2 is reflected from the surface to be tested at an angle of incidence of $90-\beta$ degrees, then normally from mirror *M*₂ and returns along its previous path to the dividing plane of the prism, where it

recombines with beam 1. The observer at *E* sees interference fringes on the superimposed images of the two mirrors.

The gross aspects of the fringe pattern (fringe direction and spacing) are controlled by the wedge angle between the wave fronts of beams 1 and 2, while the small irregularities of the fringe pattern are a function of the irregularities of the test surface. If the test surface were perfectly flat, the fringes would be straight and parallel. Any curvature of the test surface introduces a corresponding curvature in the wave front of beam 2, and this wave front, when compared with the plane wave front of beam 1, introduces curvature into the otherwise straight fringes. By adjusting the tilt of the test surface (i.e., setting $\beta=\theta$ and introducing a slight tilt across the width of the test area), it is possible to adjust the fringes so that they run parallel to the long dimension of the test area (as seen by the observer at *E*). In this case, the curvature of the fringes is a direct and precise measure of the curvature of the test area (in its long dimension). The fringe pattern is a contour map of the area of the specimen surface that is being tested.

3. Sensitivity

The sensitivity of the instrument depends upon the value of β . Since the light is reflected twice from the specimen surface, the sensitivity is double that obtained with the Linnik interferometer for the same angle of incidence. When the instrument is adjusted as described in the preceding section, one fringe departure from straightness corresponds to a departure from flatness of $\lambda/(4 \sin \beta)$, where λ is the wavelength of light.

¹ Present address: University College, London, England.

² V. P. Linnik, *Compt. rend. acad. sci. U.R.S.S.* **35**, 16 (1942).

³ J. B. Saunders, Construction of a Kösters double-image prism, *J. Research NBS*, **58**, 21 (1957) RP2729.

⁴ The Kösters double image prism is made from two 30°-60°-90° prisms, one of which is partially silvered on the face opposite the 60° angle. The two prisms are then cemented together to form the equilateral prism shown in figure 1. The partially silvered surface becomes a semireflecting plane.

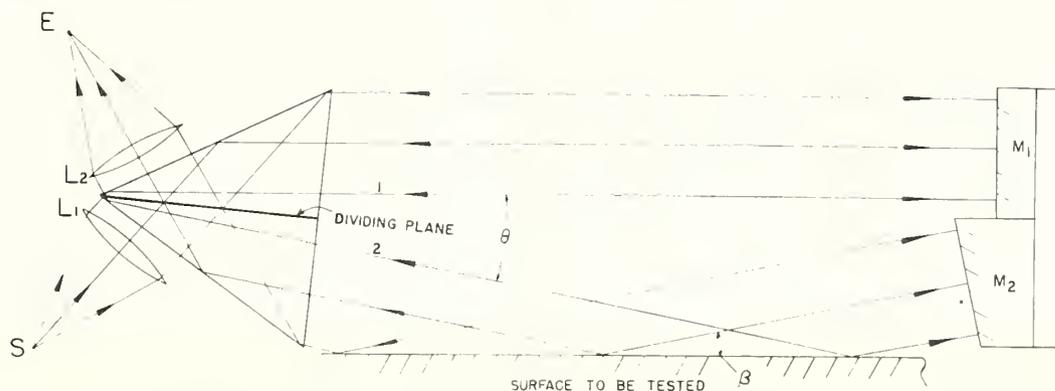


FIGURE 1. Optical arrangement of the surface plate interferometer.

In a finished instrument, the angles θ and β could be made adjustable so that any length surface could be made to fill the aperture of the system. This would give maximum sensitivity for any size surface measured. However, since the value of β must be known for evaluating the fringe pattern, it may be more practical to use fixed values for θ and β (with M_1 and M_2 bound into a rigid unit) and bind the mirror unit to the prism housing. This would eliminate the necessity of making frequent measurements of β and also add stability to the instrument. The resulting instrument would have a fixed adjustment and could be used by an unskilled operator.

The maximum length of surface that may be covered with one setting is $A \csc \beta$, where A is the aperture of the prism. Thus by decreasing β , any length surface could be covered with a prism of a given aperture, but of course the sensitivity of the instrument would also decrease.

4. Experimental Model

Figure 2 is a photograph of the pilot model that was used to test the interferometer. The surface plate used in this assembly was 91 cm long. The

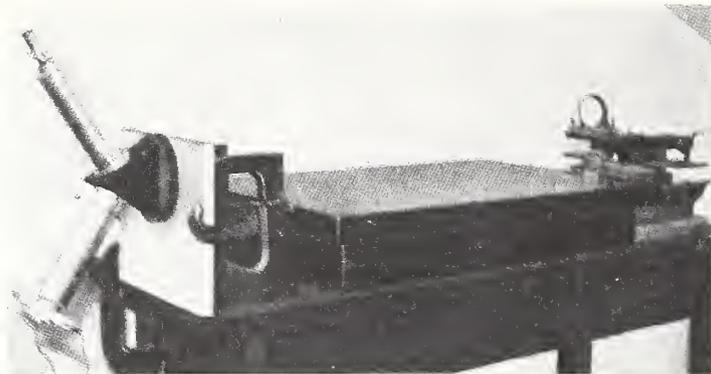


FIGURE 2. Pilot model of surface plate interferometer for testing feasibility of the optical principals with a typical granite surface plate.

The reflectivity of the end mirrors is designed to give approximately equal intensity for the two lightbeams at the receiving point E.

wedge angle between the wave fronts was controlled by adjusting the end mirrors, which produced the same effect as tilting the surface plate. The Kösters double image prism and lens assembly shown in figure 2 were taken from another instrument that was designed for use with a clear circular aperture of

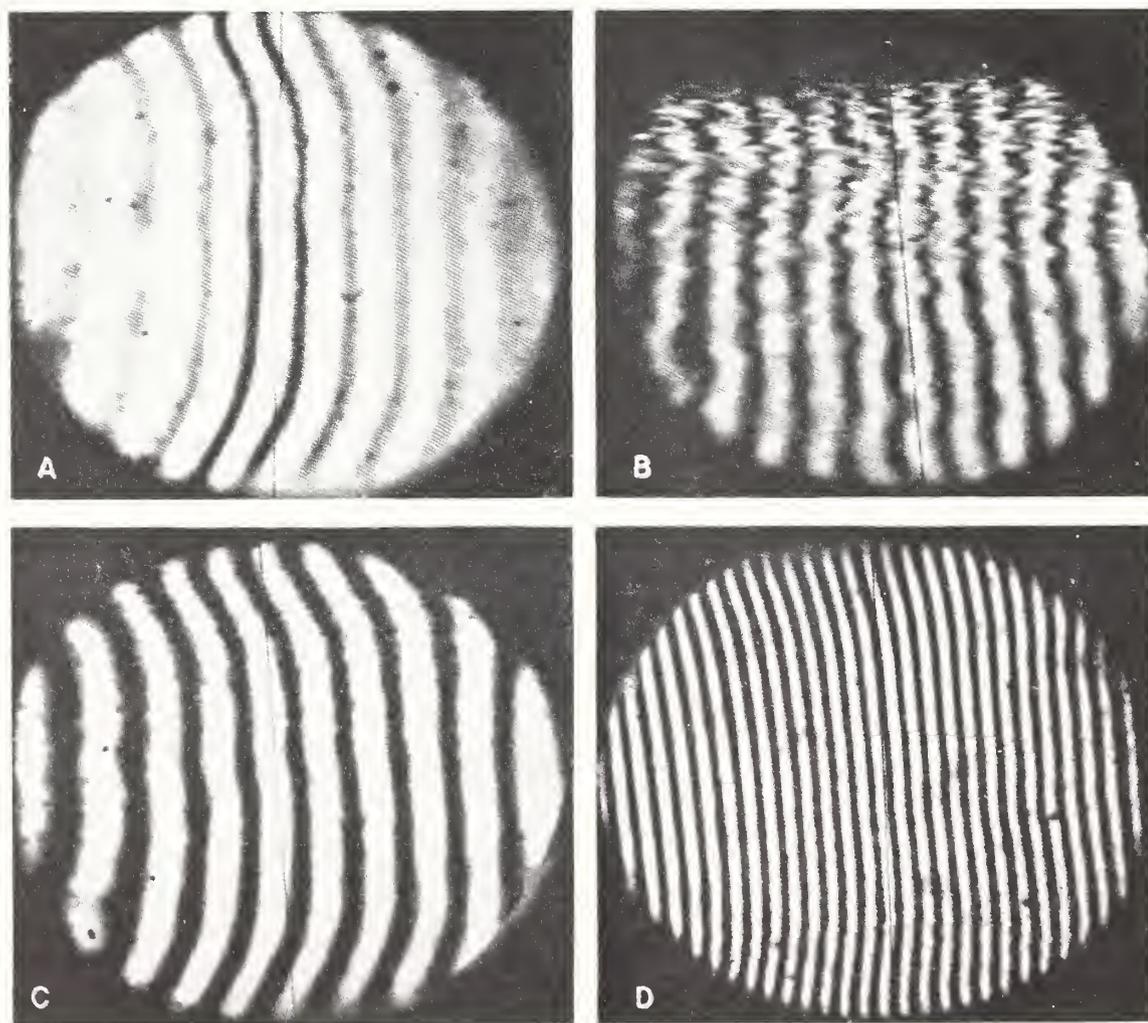


FIGURE 3. Fringe patterns.

A, White light from granite surface plate; B, monochromatic light from a badly scratched cast iron surface; C and D, monochromatic light from a granite surface. Angle of incident on surface plate is approximately $2\frac{1}{2}^\circ$.

51-mm diam (2 in.).⁵ Since the prism had an aperture 51-mm square, better coverage of the specimen surface could have been obtained by increasing the circular aperture to $51\sqrt{2}$ mm. This would have left the square aperture of the prism unobstructed, so that the area of the specimen being tested would have been a rectangle 51 mm by 91 mm, and the unfavorable narrowing of the field at the ends of the specimen due to the circular aperture would have been eliminated.

5. Results

The interference fringes shown in figure 3 were taken with the instrument described above and shown in figure 2. Photograph A was made with white light and the others with monochromatic ($\lambda=5876 \text{ \AA}$) light. The difference between photographs C and D was obtained by changing the angle between the two interfering wavefronts, thus changing the width of the fringes. Photographs A, C, and D of figure 3 were made with the plate of black granite, shown in figure 2, whereas photograph B was made with an old cast iron plate (dated 1918) that was badly scratched and marred. In this model the angle β was $2\frac{1}{2}^\circ$ and thus a departure from straightness of one fringe corresponds to a deviation from flatness of 5.75λ (approximately 0.00013 in.).

The optical performance of this instrument was found to be highly satisfactory. Some mechanical development is necessary in order to produce a more practical instrument. Accordingly, a few suggestions given here may be of help to the designer. The prism should be designed to use its entire rectangular aperture. The apertures of the lenses should be sufficiently large to prevent constriction of this aperture. The prism and end mirrors should be rigidly bound together if vibrations of the fringes are to be avoided. White light may be used but the resulting difficulty of finding and adjusting the fringes exceeds the inconveniences associated with monochromatic sources.

⁵ This prism assembly was designed for use with $\theta=0^\circ$. Using it at $\theta=2\frac{1}{2}^\circ$, the circular aperture became an oval aperture with a vertical diameter of 45 mm.

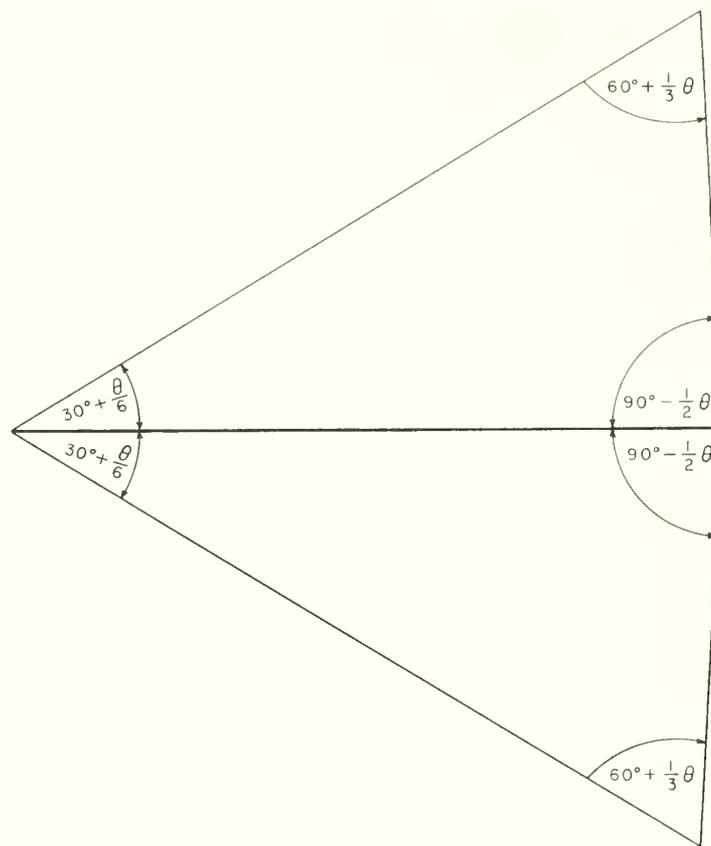


FIGURE 4. Modified Kösters double-image prism for use with monochromatic light.

A cover that encloses the light beams, between the prism and end mirrors, greatly enhances the stability of the fringes, and should be used unless the surrounding air is in a steady state. If white light is to be used, differential refraction may be reduced to a minimum by using the form of Kösters prism shown in figure 4. The procedure for making such a prism differs from that described in footnote 4 in that all surfaces are finished before cutting the prism into two parts.

WASHINGTON, December 8, 1959.

Colorimetry

by Deane B. Judd



National Bureau of Standards Circular 478

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Price 40 cents

Preface

The judgment of color permeates our industrial life. The farmer finds his vegetables and fruits often graded and priced according to color. Raw wool, cotton, and silk are graded and priced according to color. Only in this way can the textile manufacturer produce goods of uniform color at a price low enough to sell. Oils and resins used for paint have their colors measured with extreme care, and pigments and dyes likewise. Poor quality edible oils (cottonseed, cocoanut, olive, peanut) can often be detected by color; and light-colored cooking oils bring a premium price apart from quality because bakers know they can sell the resulting light-colored cakes and pies without any trouble.

Color measurement is especially vital in mass production. The assembly of parts from different sources to make a complicated machine like a refrigerator often requires a color specification for each of several parts. The white plastic door to the freezing unit must be related in color to the interior walls of the refrigerator by color tolerances no less important to the final sale than are the length and width tolerances that insure the fit of the door. There is scarcely a segment of industry untouched by color considerations.

Precise judgment of color is made possible by color measurement, or colorimetry. During the past two decades the National Bureau of Standards has played a leading part in establishing practical working standards of color and in setting up and administering color tolerances. These standards and methods have been described in some scores of scientific and technical publications. It is the purpose of this Circular to bring together this information in concise form, and by giving the basis for each technique to show more clearly how they supplement each other than has been possible in the several separate descriptions of them now available. It is hoped that this information will still further facilitate the purchase and sale of commodities and manufactured goods on a fair basis. Conformity to a color requirement can now be determined with the same assurance as size, shape, or strength.

E. U. CONDON, *Director.*

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Colorimetry

By Deane B. Judd

The definition of color as a characteristic of light is given, and the basic principles of its measurement are outlined. Reduction of spectrophotometric data is outlined, and three methods are illustrated by examples. The various methods of direct colorimetry are treated briefly, and a more detailed treatment is given of visual and photoelectric methods of colorimetry by difference from a material color standard. The most useful collections of material color standards (Lovibond, Army, Maerz & Paul, TCCA, Ostwald, Munsell) are described, and the most widely used one-dimensional color scales (color temperature, Union, Saybolt) are explained. Five widely used specifications of color having general applicability are summarized.

I. Introduction

Traditionally, two techniques are designated by the name colorimetry. In the first, the color of a transparent medium is used as an indication of the amount of some constituent in it, as in so-called chemical colorimetry. The development of this technique has shown that the appearance of the medium serves as an unnecessarily complicated indication of its constitution, but that the absorption spectrum may give a comparatively direct and precise index. For each wavelength the logarithm of the reciprocal of the internal transmittance ($\log 1/T$) is often directly proportional to the concentration of the solution. The variation of $\log 1/T$ with wavelength often identifies the constituents in the solution. The second technique relates to the measurement of objects, both self-luminous and nonself-luminous, so that the aspect of their appearance known as color can be expressed numerically. By *colorimetry* in this work we mean only this second technique.

1. Definition and Nature of Color

It is common practice to regard color as a property of objects, and in a limited sense this is true. However, light sources as well as objects have color. The flame of a bunsen burner can be changed from bluish purple to orange by the introduction of sodium. And since even objects lose their colors and become invisible unless they reflect, scatter, or transmit radiant energy, or form a part of an illuminated scene, a broader view is that color is a property of light, and of light alone.

Since it is possible to measure by a spectrophotometer the spectral-energy distribution of any light beam, and since the color of a light correlates closely with its spectral composition, some of the more physically minded have contended that color is a physical entity measurable by the techniques of physics; but this is not the most useful view. The color change of the bunsen flame from bluish purple to orange can be shown by a purely physical measurement to be caused by a change in the spectral composition of the emitted

energy; but it takes more than physics to decide whether this flame has the same orange color as the light reflected from the peel of a given citrus fruit in daylight. Application of the spectrophotometer to the orange peel will show that the spectral composition of the light reflected from it under daylight illumination is radically different from that emitted by the sodium flame. The former includes energy from every portion of the visible spectrum (about 380 to 770 $m\mu$) except for a number of narrow regions (Fraunhofer lines). The latter is confined almost entirely to two narrow spectral regions (589.0 and 589.6 $m\mu$) and is indeed comprised of energy almost completely missing from the light reflected from the orange peel illuminated by daylight. Physically, therefore, the two lights are almost as different as it is possible to obtain, but they have closely the same color. The two lights must therefore be identical in some respect other than physical identity; this identity consists in some aspect of the response made by a normal observer to the sodium flame being the same as the corresponding aspect of the response to the peel of the citrus fruit. The broader view of color must, therefore, include not only the spectral composition of the radiant energy reaching the eye of the observer, but the properties of the observer as well. These properties have been evaluated by finding equivalent stimuli, somewhat like the energy of the sodium flame and that reflected from a daylight-illuminated orange peel, that have different spectral compositions, but still manage to have exactly the same appearance to the normal observer. Such equivalent stimuli are called *metamers* and are said to form a *metameric pair*. The properties of the observer are completely defined by stating which pairs of stimuli are found to be metameric by him. Modern colorimetry is based upon spectrophotometry interpreted according to the properties of an observer.

The most widely accepted technical definition of color is that given by the Committee on Colorimetry of the Optical Society of America [107]:¹ "Color

¹ Figures in brackets indicate the literature references at the end of this Circular.

consists of the characteristics of light other than spatial and temporal inhomogeneities; light being that aspect of radiant energy of which a human observer is aware through the visual sensations which arise from the stimulation of the retina of the eye." Colorimetry is here taken to be the measurement of color. It will be noted that this definition relates color and light to radiant energy only insofar as the energy produces a visual effect within an observer. On this account, color and light are said to be psychophysical entities, and in their evaluation it is ordinarily not necessary to pay attention to energy of wavelength less than $380\text{ m}\mu$ nor greater than $770\text{ m}\mu$, because the eye is relatively insensitive to such energy.

2. Basic Principle of Measurement

If a normal observer attempts to adjust one controllable element of his central visual field so that it matches a neighboring element, he will ultimately discover that three independent adjustments have to be at his disposal. If he is using the red, yellow, and blue paints frequently found in primary grade schools, only by chance will he obtain a match from a mixture of two of them. Even a brown color requires blue in addition to red and yellow. Within the color gamut of the three paints, an exact match for any given color is easily possible, but three primary paints are the irreducible minimum. The same considerations apply to the magenta, yellow, and cyan colorants used in color photography, lithography, and color printing. Similarly, if he is trying to color-match one spot of light by shining several spotlights of different color onto the same neighboring spot of a screen, he finds, in general, that either three lights of fixed spectral composition are required, or, if two lights be added together, not only the amounts of both but also the spectral composition of at least one has to be adjustable. The same rule applies to rotary mixture on a sector disk, four sectors, giving three independent adjustments, are necessary and sufficient. Normal color vision is tridimensional.

Since the color vision of a normal observer is tridimensional, it follows that a color specification is expressible by three numbers. For normal observers three numbers are necessary, for partially colorblind observers only two numbers are necessary, and for totally colorblind observers, only one.

In the examples given (paints, spotlights, sector disks) the observer by adjustment of three variables obtains a color match, that is, he has set up a second stimulus equivalent to the first. Except by accident, however, the ternary or binary mixture does not match the unknown in spectral composition. In the usual case the mixture is equivalent to the unknown in color but not in spectral composition, and the unknown and the mixture therefore form a metameric pair. There are, however, degrees of difference in spectral composition. If one painted panel be matched by a mixture of red, yellow, and blue paints, the degree of metamerism is likely to be only moderate; but if the paint panel illuminated by daylight be matched by shining on a white card three spotlights,

each one of which contains energy restricted to a narrow wavelength band (such as spectrum red, green, and blue), the degree of metamerism will ordinarily be extreme. Normal observers will agree closely on matches that are but slightly metameric; but they may disagree widely on highly metameric matches.

(a) Ocular Pigmentation

Studies of extremely metameric pairs in which mixtures of two parts of the spectrum are set up to color match other two-part spectrum mixtures have yielded our most valuable knowledge regarding the properties of the average normal eye [1, 2, 42, 70, 83, 140]. An outstanding fact derived from observation of such metamers is that the center of the retina (fovea centralis) has somewhat different properties from that immediately surrounding it; that is, a color match set up for the central 2° or 3° of the retina becomes an easily detectable mismatch if the eye be turned so as to allow the stimuli to affect a portion of the retina, say, 6° from the fovea. Furthermore, if the metamers are compared in large patches (6° or more), this mismatch causes a central spot to appear temporarily on a field that is physically uniform. This dependence of metamerism on the portion of the retina used arises chiefly from the existence of a spot of brownish or yellowish pigment irregularly covering and interpenetrating the central 3° or 4° of the normal retina; it is called the macula lutea, or sometimes the yellow spot. The pigment acts as a selective filter interposed between the vitreous humor and receptor layer at the retina. Metamers set up for one normal observer usually fail to hold strictly for anyone else. This failure is ascribable to variation in amount of pigmentation of the eye media, the macular pigment being one of the chief variables. Table 1 and figure 1 show the spectral transmittance of the macu-

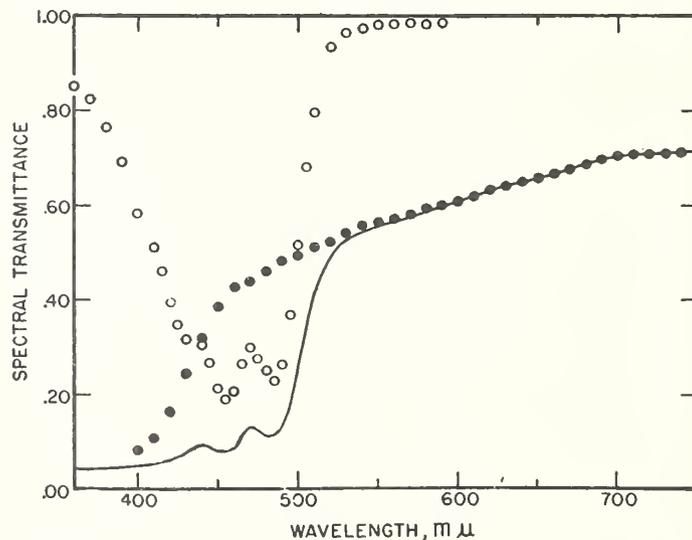


FIGURE 1. Spectral transmittance of the ocular media, Ludvigh-McCarthy [75], Wald [135].

The macula, or yellow spot, absorbs chiefly in the blue region of the spectrum (see circles); all other ocular media (chiefly the crystalline lens) absorb more strongly in the ultraviolet and violet regions (see dots). The center of the eye is thus protected against over-stimulation by short-wave energy by the combination of these two absorptions (see curve).

lar pigment according to Wald [135] and the spectral transmittance of all other ocular media (aqueous, vitreous, lens, and cornea) according to Ludvig and McCarthy [75]. The product of these two (also shown) is an estimate of the average transmittance of the ocular media, but there is a large individual-observer difference. The properties of the normal eye derived from a study of extreme metamers therefore refer only to the central 2° of the retina, and they refer to an hypothetical average eye. Probably nobody has an eye that is sufficiently close to the average to be a substitute for it in all applications.

TABLE 1. Spectral transmittance of ocular media

Wave-length	Spectral transmittance of cornea, lens, and aqueous and vitreous humors (Ludvig-McCarthy)	Spectral internal transmittance of macula lutea (Wald)	Spectral transmittance of ocular media, including the macula lutea
mμ			
360	¹ 0.052	0.859	0.045
370	¹ .056	.826	.046
380	¹ .062	.762	.047
390	¹ .069	.695	.048
400	.086	.577	.050
410	.106	.506	.054
420	.160	.396	.063
430	.248	.316	.078
440	.318	.305	.097
450	.388	.212	.082
460	.426	.206	.088
470	.438	.299	.131
480	.458	.250	.115
490	.481	.263	.126
500	.495	.516	.256
510	.510	.798	.407
520	.525	.935	.491
530	.543	.968	.526
540	.559	.977	.546
550	.566	.985	.557
560	.572	.989	.566
570	.583	.989	.577
580	.594	.989	.587
590	.602	.989	.595
600	.610	¹ 1.000	.610
610	.619	¹ 1.000	.619
620	.631	¹ 1.000	.631
630	.641	¹ 1.000	.641
640	.649	¹ 1.000	.649
650	.657	¹ 1.000	.657
660	.664	¹ 1.000	.664
670	.676	¹ 1.000	.676
680	.690	¹ 1.000	.690
690	.698	¹ 1.000	.698
700	.705	¹ 1.000	.705
710	.707	¹ 1.000	.707
720	.708	¹ 1.000	.708
730	.710	¹ 1.000	.710
740	.711	¹ 1.000	.711
750	.713	¹ 1.000	.713

¹ Extrapolated.

(b) Grassman's Law

From a knowledge of spectral metamers it has been possible to summarize concisely the properties of a reasonably average normal eye. This summary is made in accord with the principle known as Grassman's law [39] foreshadowed by Newton's laws of color-mixture [98]. If a light composed of known amounts of three components (called primaries) is equivalent in color to an unknown light, the three known amounts may be used as a color specification for this light. These amounts are called the *tristimulus values* of the color.

Grassman's law states that when equivalent lights are added to equivalent lights, the sums are equivalent. Thus, if an unknown spot of color were matched by shining on the same spot of a white screen two component spotlights of tristimulus values, X_1, Y_1, Z_1 , and X_2, Y_2, Z_2 , respectively, by Grassman's law, the tristimulus values, X, Y, Z , of the unknown spot of color would be simply:

$$\left. \begin{aligned} X &= X_1 + X_2, \\ Y &= Y_1 + Y_2, \\ Z &= Z_1 + Z_2. \end{aligned} \right\} \quad (1)$$

Any beam of light, whether it originates from a self-luminous body, or comes by transmission, scattering, or reflection, from a nonself-luminous object, may be considered as made up of a large number of portions of the spectrum. The amounts of these various portions may be determined by spectrophotometry. The tristimulus specifications, $\bar{x}_\lambda, \bar{y}_\lambda, \bar{z}_\lambda$, of each of these portions have been determined for a number of normal observers, and average values are given in table 2 in arbitrary units for a spectrum of unit spectral irradiance. The amounts, X, Y, Z , of the primaries required to produce a stimulus equivalent to any light beam may therefore, by Grassman's law, be found simply by adding together the tristimulus values of the various parts of its spectrum. Modern colorimetry is based upon this application of Grassman's law.

The principle expressed in Grassman's law has been established by repeated experiment over a wide middle range of retinal illuminance. It breaks down for very high retinal illuminance [136, 141, 142] that begins to approach those sufficient to do the eye permanent harm; and it breaks down if the illuminance of the whole retina continues for 10 min. or more to be so slight that vision by the retinal rods (twilight vision) intrudes significantly [71]. Between these two extremes, however, Grassman's law holds independent of the adaptive state of the eye. Thus, if two stimuli of different wavelength distributions of energy be found that are once responded to alike by the eye, they will be seen alike even after exposure of the eye to another stimulus sufficient to change considerably the appearance of the two equivalent stimuli. For example, if a portion of the spectrum near 640 mμ (red) be superposed on a portion near 550 mμ (yellowish green), it will be found possible to obtain the color of this combination from an intermediate portion of the spectrum, say 590 mμ (orange). If the retina of the eye be highly illuminated by light of wavelength near 640 mμ, and its sensitivity to radiant flux of this wavelength region considerably reduced in this way, it is found that, although neither of the equivalent stimuli any longer appears orange, they still give identical colors, for example, they may yield identical yellows or identical greenish yellows. The eye thus cannot be trusted to yield the same color perception from a given stimulus; simultaneous and successive contrast affect it profoundly. But it is still a satisfactory null instrument and obeys Grassman's law.

By Grassman's law it is possible to test whether any two beams of light of differing spectral composition form a metameric pair for any observer defined by $\bar{x}_\lambda, \bar{y}_\lambda, \bar{z}_\lambda$. The condition for metamorphism of two beams of light of spectral irradiance, $(E_1)_\lambda$ and $(E_2)_\lambda$, is that simultaneously:

$$\left. \begin{aligned} \sum_0^8 (E_1)_\lambda \bar{x}_\lambda \Delta\lambda &= \sum_0^8 (E_2)_\lambda \bar{x}_\lambda \Delta\lambda, \\ \sum_0^8 (E_1)_\lambda \bar{y}_\lambda \Delta\lambda &= \sum_0^8 (E_2)_\lambda \bar{y}_\lambda \Delta\lambda, \\ \sum_0^8 (E_1)_\lambda \bar{z}_\lambda \Delta\lambda &= \sum_0^8 (E_2)_\lambda \bar{z}_\lambda \Delta\lambda. \end{aligned} \right\} \quad (2)$$

The wavelength interval, $\Delta\lambda$, to be used in these summations depends upon the irregularity of the curve of spectral irradiance with wavelength; intervals of 20 $m\mu$ are sometimes sufficient to yield a significant result; intervals of 10 $m\mu$ often do; and intervals of 5 $m\mu$ are usually sufficient except for discontinuous spectral distributions such as those characterizing gaseous discharge lamps. The wavelength functions, $\bar{x}_\lambda, \bar{y}_\lambda, \bar{z}_\lambda$, characterize the observer; they indicate the amounts of the three primaries required by him to match the various parts of the spectrum at unit irradiance. If these functions be divided by the spectral transmittance of the ocular media (such as estimated in table 1), the ratios so obtained characterize the foveal receptors of the observer.

Any three lights may be used as primaries in a system of tristimulus color specifications, provided only that no one of them is equivalent to a combination of the other two. Tristimulus specifications, X, Y, Z , expressed relative to one set of primaries may be transformed into specifications, R, G, B , relative to any other set of primaries by transformation equations of the form:

$$\left. \begin{aligned} R &= K_1 X + K_2 Y + K_3 Z \\ G &= K_4 X + K_5 Y + K_6 Z \\ B &= K_7 X + K_8 Y + K_9 Z \end{aligned} \right\} \quad (3)$$

II. The Standard Observer, Illuminants and Coordinate System

In 1931 the International Commission on Illumination recommended that all subsequent color data be expressed in terms of the same tristimulus system, so that the results would be immediately comparable. The standard observer and coordinate system recommended [20, 44, 56, 126] are defined by the tristimulus values of the spectrum colors given in table 2 and plotted in figure 2. It will be noted that the primaries chosen are such that none of these tristimulus values is less than zero. It is further true that the green primary chosen, whose amounts are designated by Y , is such as to carry all of the luminosity, the other two primaries (red, blue) whose amounts are designated by X and Z , respectively, being unassociated with luminosity. Therefore the values of \bar{y}_λ for the spectrum correspond to the standard luminosity function, and it is convenient and customary to

The constants, K_1 to K_9 , may take on any arbitrary values, positive, negative, or zero, provided they are not such as to make one of the new primaries identical to a combination of the other two; that is, provided that:

$$\begin{vmatrix} K_1 & K_2 & K_3 \\ K_4 & K_5 & K_6 \\ K_7 & K_8 & K_9 \end{vmatrix} \neq 0. \quad (3a)$$

Since the exceptions that cause the determinant of the system to vanish are trivial, the choice of coordinate system is very wide. The primaries do not even have to correspond to physically realizable lights. Imaginary lights defined by spectral compositions having negative values for some parts of the spectrum are admissible, and indeed are preferred for routine colorimetry, because by their use the computation of tristimulus values from spectrophotometric data is somewhat simplified.

It is generally presumed that the initial responses of the retinal cones are photochemical in nature, and that there are three of them, each independent of the other two. If we knew what colors (imaginary or real) correspond to each of these responses, we could evaluate the constants K_1 to K_9 , and by finding R, G, B , by setting $\bar{x}, \bar{y}, \bar{z}$, for X, Y , and Z , in formula 3 and by dividing by the spectral transmittance of the ocular media (such as estimated in table 1), we could find the spectral sensitivities of the presumed photosensitive substances required for the observer. It will be noted, however, that lack of this theoretical information is no hindrance to the use of the functions $\bar{x}_\lambda, \bar{y}_\lambda, \bar{z}_\lambda$, for colorimetric purposes. It makes no difference in the end result whether these functions or any analogous functions computed from formula 3 be used. Grassman's law is a well-established experimental fact, and is not dependent upon any theory of vision.

express the Y value of a luminous area as its *luminance* (photometric brightness) in terms of some recognized unit (such as candles per square meter, millilambert, or foot lambert), and the Y value of a transmitting specimen is customarily put in terms of *luminous transmittance* (ratio of transmitted to incident luminous flux). However, the more complicated concept, *luminous directional reflectance*, which refers only to that flux reflected in the direction of the observer, is more closely related to the appearance of the specimen, and is therefore the more commonly used as an evaluation of Y for opaque specimens. *Luminous directional reflectance* is the reflectance that a perfectly diffusing surface illuminated and viewed like the specimen would need to have in order to attain the same luminance as the specimen. Directional reflectance was called apparent reflectance by

McNicholas, originator of the concept [84, 107]. The Y value of an opaque specimen may also be conveniently expressed as its *luminous reflectance* (ratio of reflected to incident luminous flux).

If, as is usual, light combinations or mixtures are not the chief interest, it is convenient to substitute for the tristimulus values, X, Y, Z , the two ratios $X/(X+Y+Z)$ and $Y/(X+Y+Z)$, combined with the luminous value, Y . The two ratios are known as chromaticity coordinates, x, y , because they serve to specify the chromatic aspect of the light. The analogous ratio, $Z/(X+Y+Z)$, is also known as a chromaticity coordinate, z , but only two of the three coordinates, x, y, z , give independent information since by definition the sum of all three is unity. Table 2 also gives the chromaticity coordinates, x, y, z , of the

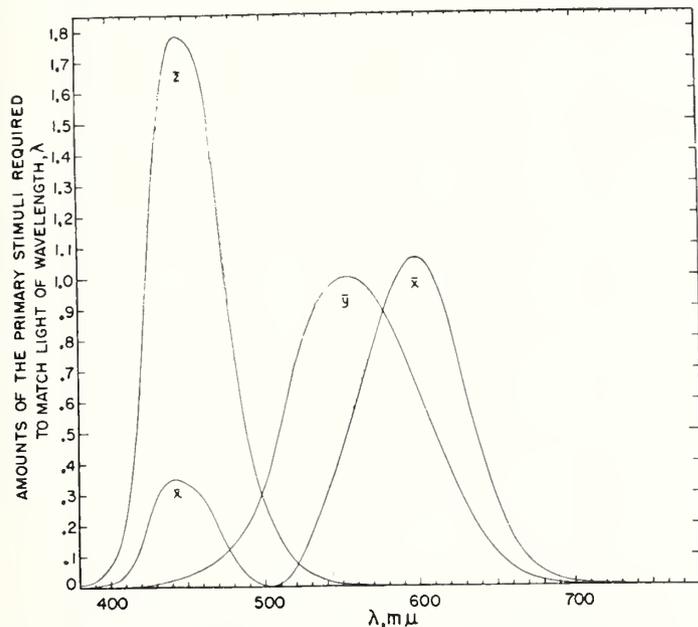


FIGURE 2. Tristimulus values of spectrum stimuli of unit irradiance according to the standard observer and coordinate system recommended by the International Commission on Illumination in 1931 (the ICI or CIE system).

The y -function is the relative luminosity function for daylight vision. By reading the three ordinates at any wavelength the amounts of the three primaries required in an additive combination to color match the spectrum at that wavelength may be found. For example, equal amounts of the X - and Y -primaries combine to produce for the standard observer a close match for the color of the spectrum at 578 $m\mu$.

spectrum colors; and figure 3 shows the points representing the spectrum colors in the (x, y) -chromaticity diagram. This diagram is also known as a *Maxwell triangle* because of Maxwell's first use of such a diagram.

Furthermore, it has aptly been called a *mixture diagram* because it indicates in a very simple way the chromaticity of the color resulting from the additive combination of any two lights. The point representing this chromaticity is found on the straight line connecting the points representing the two lights. The primary lights are represented by points at the corners of a triangle, and every point within the triangle represents the chromaticity of a mixture of the primary lights whose proportions are indicated by the chromaticity coordinates, x, y, z . The spectrum colors are shown by dots connected by a smooth

curve known as the spectrum locus. They are identified by wavelength in millimicrons. It will be noted from figure 3 that the spectrum locus is substantially straight from 540 $m\mu$ to the longwave extreme. This means that the standard observer would find binary mixtures of, say, 540 $m\mu$ with 640 $m\mu$, closely equivalent to some intermediate portion of the spectrum. But the spectrum locus from 540 $m\mu$ to the shortwave extreme is convex. This means that for the standard observer a binary mixture of 540 $m\mu$ with, say, 440 $m\mu$ would differ importantly in chromaticity from the intermediate parts of the spectrum. By drawing straight lines through any central point (such as $x = y = 1/3$, representing the stimulus whose spectrum has constant radiance per unit wavelength, the so-called equal-energy stimulus) and extending them until they cut the spectrum locus, we may find the spectral complementaries relative to a stimulus represented by that point, that is, we may find the two parts of the spectrum that when combined in proper proportions will for the standard observer be equivalent to the central stimulus.

The straight line in figure 3 joining the extremes of the spectrum locus represents the chromaticities of the mixtures of the two extremes of the visible spectrum. The area bounded by the closed curve made up of the spectrum locus and this straight line is the locus of all physically realizable chromaticities. Note that the points representing the primaries of the ICI coordinate system, the apices of the triangle ($x = 1, y = z = 0$; $y = 1, x = z = 0$; $z = 1, x = y = 0$), all fall outside this area; that is, the primaries are imaginary. Note also that both the X and Z primaries fall on the line $y = 0$, which is unassociated with luminosity and is known as the *alychne* or *lightless line*; this means that although it has the power to elicit in the standard observer a considerable X and Z response, resulting in a vivid bluish purple color, radiant flux of wavelength 380 to 420 $m\mu$ is only slightly luminous.

The areas in figure 3 corresponding to common color designations for lights are those proposed by Kelly [66] on a basis to be discussed later.

Figure 3a illustrates the use of the (x, y) -chromaticity diagram for the specification of railway signal colors [4a].

At the time of setting up the standard observer and coordinate system, the International Commission on Illumination [20] recommended three standard illuminants for colorimetry, and standard angular conditions and a reflectance standard for the colorimetry of opaque specimens. The standard illuminants are: illuminant A , representative of gas-filled incandescent lamps; illuminant B , representative of noon sunlight; and illuminant C representative of average daylight such as that from a completely overcast sky. Illuminant A is an incandescent lamp operated at a color temperature of 2,854°K on the international temperature scale ($C_2=14,380$), or at a color temperature of 2,850°K if the value of $C_2=14,360$ recommended by Wensel [137] be used in Planck's formula for spectral emittance. Illuminant B is obtained by using

TABLE 2. The 1931 ICI standard observer

Chromaticity coordinates			Wave-length $m\mu$	Tristimulus values of equal-energy spectrum			Chromaticity coordinates			Wave-length $m\mu$	Tristimulus values of equal-energy spectrum		
x	y	z		\bar{x}	\bar{y}	\bar{z}	x	y	z		\bar{x}	\bar{y}	\bar{z}
0.1741	0.0050	0.8209	380	0.0014	0.0000	0.0065	0.5125	0.4866	0.0009	580	0.9163	0.8700	0.0117
.1740	.0050	.8210	385	.0022	.0001	.0105	.5448	.4544	.0008	585	.9786	.8163	.0014
.1738	.0049	.8213	390	.0042	.0001	.0201	.5752	.4242	.0006	590	1.0263	.7570	.0011
.1736	.0049	.8215	395	.0076	.0002	.0362	.6029	.3965	.0006	595	1.0567	.6949	.0010
.1733	.0048	.8219	400	.0143	.0004	.0679	.6270	.3725	.0005	600	1.0622	.6310	.0008
.1730	.0048	.8222	405	.0232	.0006	.1102	.6482	.3514	.0004	605	1.0456	.5668	.0006
.1726	.0048	.8226	410	.0435	.0012	.2074	.6658	.3340	.0002	610	1.0126	.5030	.0003
.1721	.0048	.8231	415	.0776	.0022	.3713	.6801	.3197	.0002	615	0.9384	.4412	.0002
.1714	.0051	.8235	420	.1344	.0040	.6456	.6915	.3083	.0002	620	.8544	.3810	.0002
.1703	.0058	.8239	425	.2148	.0073	1.0391	.7006	.2993	.0001	625	.7514	.3210	.0001
.1689	.0069	.8242	430	.2839	.0116	1.3856	.7079	.2920	.0001	630	.6424	.2650	.0000
.1669	.0086	.8245	435	.3285	.0168	1.6230	.7140	.2859	.0001	635	.5419	.2170	.0000
.1644	.0109	.8247	440	.3483	.0230	1.7471	.7190	.2809	.0001	640	.4479	.1750	.0000
.1611	.0138	.8251	445	.3481	.0298	1.7826	.7230	.2770	.0000	645	.3608	.1382	.0000
.1566	.0177	.8257	450	.3362	.0380	1.7721	.7260	.2740	.0000	650	.2835	.1070	.0000
.1510	.0227	.8263	455	.3187	.0480	1.7441	.7283	.2717	.0000	655	.2187	.0816	.0000
.1440	.0297	.8263	460	.2908	.0600	1.6692	.7300	.2700	.0000	660	.1649	.0610	.0000
.1355	.0399	.8246	465	.2511	.0739	1.5281	.7311	.2689	.0000	665	.1212	.0446	.0000
.1241	.0578	.8181	470	.1954	.0910	1.2876	.7320	.2680	.0000	670	.0874	.0320	.0000
.1096	.0868	.8036	475	.1421	.1126	1.0419	.7327	.2673	.0000	675	.0636	.0232	.0000
.0913	.1327	.7760	480	.0956	.1390	0.8130	.7334	.2666	.0000	680	.0468	.0170	.0000
.0687	.2007	.7306	485	.0580	.1693	.6162	.7340	.2660	.0000	685	.0329	.0119	.0000
.0454	.2950	.6596	490	.0320	.2080	.4652	.7344	.2656	.0000	690	.0227	.0082	.0000
.0235	.4127	.5638	495	.0147	.2586	.3533	.7346	.2654	.0000	695	.0158	.0057	.0000
.0082	.5384	.4534	500	.0049	.3230	.2720	.7347	.2653	.0000	700	.0114	.0041	.0000
.0039	.6548	.3413	505	.0024	.4073	.2123	.7347	.2653	.0000	705	.0081	.0029	.0000
.0139	.7502	.2359	510	.0093	.5030	.1582	.7347	.2653	.0000	710	.0058	.0021	.0000
.0389	.8120	.1491	515	.0291	.6082	.1117	.7347	.2653	.0000	715	.0041	.0015	.0000
.0743	.8338	.0919	520	.0633	.7100	.0782	.7347	.2653	.0000	720	.0029	.0010	.0000
.1142	.8262	.0596	525	.1096	.7932	.0573	.7347	.2653	.0000	725	.0020	.0007	.0000
.1547	.8059	.0394	530	.1655	.8620	.0422	.7347	.2653	.0000	730	.0014	.0005	.0000
.1929	.7816	.0255	535	.2257	.9149	.0298	.7347	.2653	.0000	735	.0010	.0004	.0000
.2296	.7543	.0161	540	.2904	.9540	.0203	.7347	.2653	.0000	740	.0007	.0003	.0000
.2658	.7243	.0099	545	.3597	.9803	.0134	.7347	.2653	.0000	745	.0005	.0002	.0000
.3016	.6923	.0061	550	.4334	.9950	.0087	.7347	.2653	.0000	750	.0003	.0001	.0000
.3373	.6589	.0038	555	.5121	1.0002	.0057	.7347	.2653	.0000	755	.0002	.0001	.0000
.3731	.6245	.0024	560	.5945	0.9950	.0039	.7347	.2653	.0000	760	.0002	.0001	.0000
.4087	.5896	.0017	565	.6784	.9786	.0027	.7347	.2653	.0000	765	.0001	.0000	.0000
.4441	.5547	.0012	570	.7621	.9520	.0021	.7347	.2653	.0000	770	.0001	.0000	.0000
.4788	.5202	.0010	575	.8425	.9154	.0018	.7347	.2653	.0000	775	.0000	.0000	.0000
.5125	.4866	.0009	580	.9163	.8700	.0017	.7347	.2653	.0000	780	.0000	.0000	.0000
										Totals	21.3713	21.3714	21.3715

this same lamp in combination with a two-cell Davis-Gibson liquid filter giving a nearest color temperature of about 5,000°K. Illuminant C is obtained similarly and results in a source of nearest color temperature about 6,800°K. These illuminants are recommended for general use, or whenever there is no special reason for using some other source. Detailed directions for making the filters are given by Davis and Gibson [22], and the composition of the solutions is specified in many accessible publications [44, 56, 108]. Table 19 gives the relative spectral irradiance of illuminants A, B, and C.

The angular conditions recommended for the colorimetry of opaque specimens are that the light shall strike the specimen at 45° and that the specimen shall be viewed along the perpendicular to its surface. Since, as in the inspection of the glossy and semi-glossy materials making up the bulk of commerce, these conditions avoid by a wide margin the inclusion of the main beam of specularly reflected light, they should be used particularly when appearance is the chief concern, and in the absence of circumstances requiring another choice of angular conditions. However, one nonstandard set of angular conditions deserves special mention because of its

wide use; this is to illuminate the specimen along or near the perpendicular and to collect by means of a white-lined sphere all of the reflected light regardless of direction, except for a small solid angle centering along the average direction of the illuminating beam. The particular merit of this choice of angular conditions is that it does not waste any of the reflected flux; and so it is well suited to photoelectric devices with a small margin of sensitivity. The results obtained by using these normal-diffuse conditions correlate with the appearance of the specimen viewed under a completely uniform sky. But since inspectors are usually not concerned with the appearance of specimens so illuminated, it is customary to apply to results so obtained a correction adjusting them at least approximately to the standard angular conditions.

The reflectance standard recommended for the colorimetry of opaque specimens is a white surface prepared by collecting on a metallic or other suitable surface an opaque layer formed by the smoke from magnesium ribbon or shavings burning in air. Details for the preparation of a magnesium-oxide reflectance standard are given in NBS Letter Circular LC547 [113]. It is now nearly universal practice

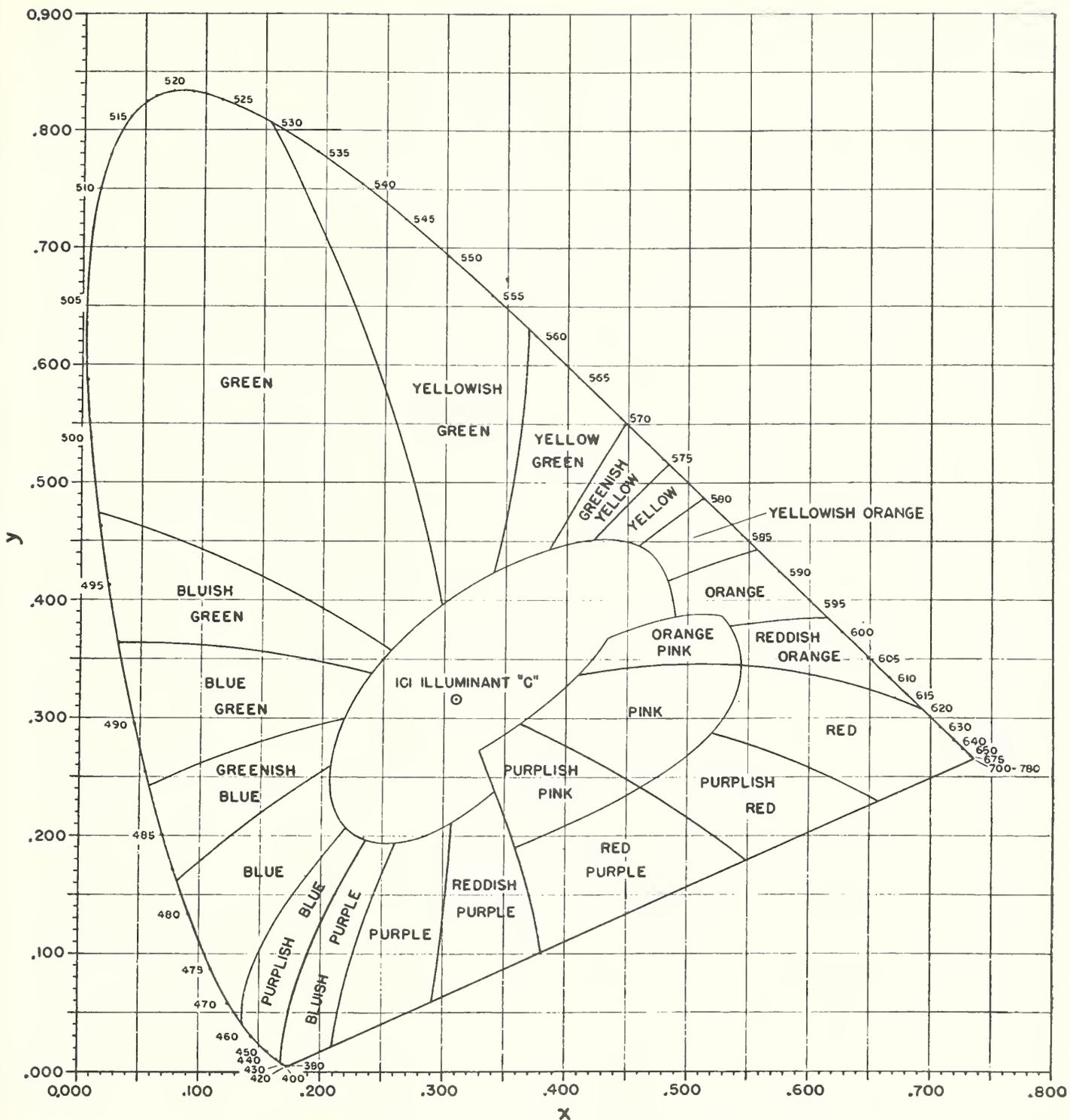


FIGURE 3. *The (x,y)-chromaticity diagram of the ICI system.*

The abscissa, x , is the ratio of the tristimulus value, X , to the sum of all three ($X+Y+Z$). The ordinate, y , is the ratio of Y to this sum. The parts of the spectrum locus are identified by wavelength in millimicrons. The region bounded by this locus and the straightline (purple border) joining its extremes represents all chromaticities producible by actual stimuli. The division of this chromaticity diagram into a number of regions corresponding to various hues surrounding a central region to which no hue name is given is from Kelly's work [66] on color designations for lights. It refers to observation of self-luminous areas against a dark background.

to carry out the spectrophotometry of color standards relative to this magnesium-oxide standard; that is, the ratio of the directional reflectance of the unknown specimen to that of the magnesium-oxide standard, similarly illuminated and viewed, is found rather than the absolute value of directional reflectance itself. For the standard (45° -normal) angular con-

ditions, it has been found [114, 117] that a freshly prepared magnesium-oxide reflectance standard has the very convenient directional reflectance of 1.00 with variations throughout the visible spectrum of less than 0.01. Because a magnesium-oxide surface gradually becomes yellow with age, it is often more feasible to use a secondary standard of more perma-

ment nature, such as a vitreous plaque. These secondary standards are, however, calibrated by comparison with freshly prepared magnesium oxide, and the results obtained by using them are corrected to correspond with what would have been obtained had the magnesium-oxide standard, itself, been used.

1. Reduction of Spectrophotometric Data to Colorimetric Terms

The fundamental nature of the tristimulus specification of color permits it to be used as a common

denominator by means of which colorimeters (involving color standards of glass, plastic, or solutions) or systems of material color standards, transparent and opaque, may be intercompared. To demonstrate how the ICI standard observer and coordinate system may be used for this purpose, the steps in the evaluation of two of the reference samples issued by us, Kitchen Green (SKC-15) [94] and Orchid (SBC-20) [93] are reproduced in detail. Figure 4 shows spectral reflectances of the master standards for these two kinds of vitreous enamel samples.

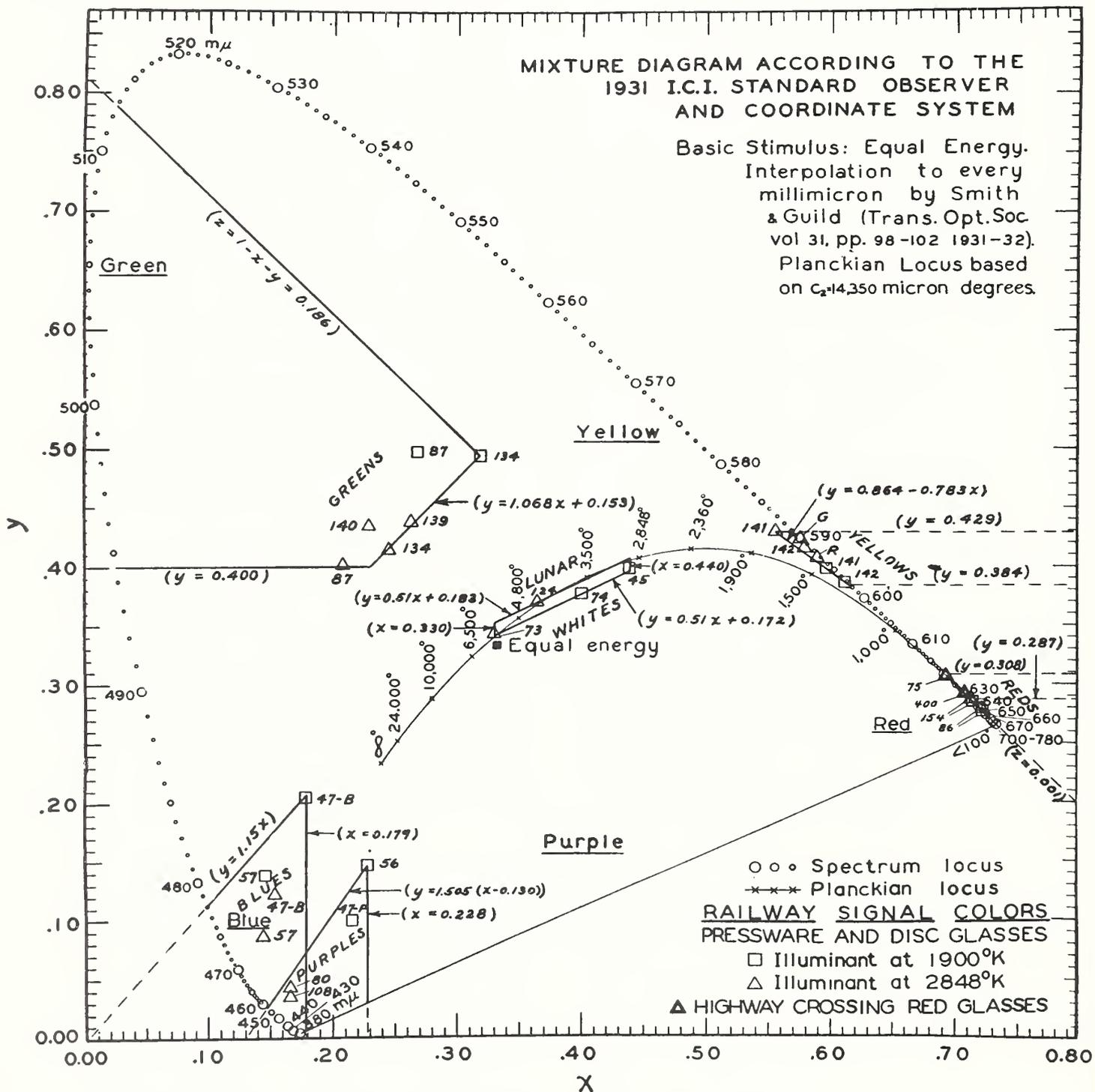


FIGURE 3a. Illustration of the use of the (x,y)-chromaticity diagram to specify the railway signal colors. Note that areas corresponding to permitted colors for red, yellow, green, blue, purple, and lunar white signals are shown (AAR, [4a]).

TABLE 3(a)

ICI Coordinates.

Illuminant A (2,854°K)

Sample.....

Source of Trans Data.....

Submitted by

λ	$(\bar{x}E)\lambda$	$(\bar{y}E)\lambda$ <small>$\frac{\bar{y}}{\bar{x}}$</small>	$(\bar{z}E)\lambda$	$T\lambda$	$(\bar{x}ET)\lambda$	$(\bar{y}ET)\lambda$	$(\bar{z}ET)\lambda$
380	1		6	0.			
90	5		23	.			
400	19	1	93	.			
10	71	2	340	.			
20	262	8	1256	.			
30	649	27	3167	.			
40	926	61	4647	.			
450	1031	117	5435	.			
60	1019	210	5851	.			
70	776	362	5116	.			
80	428	622	3636	.			
90	160	1039	2324	.			
500	27	1792	1509	.			
10	57	3080	969	.			
20	425	4771	525	.			
30	1214	6322	309	.			
40	2313	7600	162	.			
550	3732	8568	75	.			
60	5510	9222	36	.			
70	7571	9457	21	.			
80	9719	9228	18	.			
90	11579	8540	12	.			
600	12704	7547	10	.			
10	12669	6356	4	.			
20	11373	5071	3	.			
30	8980	3704		.			
40	6558	2562		.			
650	4336	1637		.			
60	2628	972		.			
70	1448	530		.			
80	804	292		.			
90	404	146		.			
700	209	75		.			
10	110	40		.			
20	57	19		.			
30	28	10		.			
40	14	6		.			
750	6	2		.			
60	4	2		.			
70	2			.			
Sums →	109828	100000	35547	Sums →	X =	Y =	Z =
x_{10}, y_{10}, z_{10}	0.4476	0.4075	0.1449	$S = X + Y + Z$			
$x, y, \text{ and } z$ ($x = X/S, y = Y/S, z = Z/S$)							

Planck 2,854°K, $C_2 = 14,380$

Planck 2,848°K, $C_2 = 14,350$

Computed by..... Checked by.....

TABLE 3(b)

ICI Coordinates.

Illuminant B

Sample..... Source of Trans Data.....

Submitted by

λ	$(\bar{x}E)\lambda$	$(\bar{y}E)\lambda$	$(\bar{z}E)\lambda$	$T\lambda$	$(\bar{x}ET)\lambda$	$(\bar{y}ET)\lambda$	$(\bar{z}ET)\lambda$
380	3		14	0.			
90	13		60	.			
400	56	2	268	.			
10	217	6	1033	.			
20	812	24	3899	.			
30	1983	81	9678	.			
40	2689	178	13489	.			
450	2744	310	14462	.			
60	2454	506	14085	.			
70	1718	800	11319	.			
80	870	1265	7396	.			
90	295	1918	4290	.			
500	44	2908	2449	.			
10	81	4360	1371	.			
20	541	6072	669	.			
30	1458	7594	372	.			
40	2689	8834	188	.			
550	4183	9603	84	.			
60	5840	9774	38	.			
70	7472	9334	21	.			
80	8843	8396	16	.			
90	9728	7176	10	.			
600	9948	5909	7	.			
10	9436	4734	3	.			
20	8140	3630	2	.			
30	6200	2558		.			
40	4374	1709		.			
650	2815	1062		.			
60	1655	612		.			
70	876	321		.			
80	465	169		.			
90	220	80		.			
700	108	39		.			
10	53	19		.			
20	26	9		.			
30	12	4		.			
40	6	2		.			
750	2	1		.			
60	2	1		.			
70	1			.			
Sums	99072	100000	85223	$S = X + Y + Z$			
x_w, y_w, z_w	0.34848	0.35175	0.29977				
$x, y, \text{ and } z$ $(x = X/S, y = Y/S, z = Z/S)$	→						

Computed by..... Checked by.....

TABLE 3(c)

ICI Coordinates.

Illuminant C

Sample.....

Source of Trans Data.....

Submitted by

λ	$(\bar{x}E)\lambda$	$(\bar{y}E)\lambda$	$(\bar{z}E)\lambda$	$T\lambda$	$(\bar{x}ET)\lambda$	$(\bar{y}ET)\lambda$	$(\bar{z}ET)\lambda$
380	4		20	0.			
90	19		89	.			
400	85	2	404	.			
10	329	9	1570	.			
20	1238	37	5949	.			
30	2997	122	14628	.			
40	3975	262	19938	.			
450	3915	443	20638	.			
60	3362	694	19299	.			
70	2272	1058	14972	.			
80	1112	1618	9461	.			
90	363	2358	5274	.			
500	52	3401	2864	.			
10	89	4833	1520	.			
20	576	6462	712	.			
30	1523	7934	388	.			
40	2785	9149	195	.			
550	4282	9832	86	.			
60	5880	9841	39	.			
70	7322	9147	20	.			
80	8417	7992	16	.			
90	8984	6627	10	.			
600	8949	5316	7	.			
10	8325	4176	2	.			
20	7070	3153	2	.			
30	5309	2190		.			
40	3693	1443		.			
650	2349	886		.			
60	1361	504		.			
70	708	259		.			
80	369	134		.			
90	171	62		.			
700	82	29		.			
10	39	14		.			
20	19	6		.			
30	8	3		.			
40	4	2		.			
750	2	1		.			
60	1	1		.			
70	1			.			
Sums	98041	100000	118103	Sums \longrightarrow	X =	Y =	Z =
x_w, y_w, z_w	.31012	.31631	.37357	$S = X + Y + Z$			
$x, y, \text{ and } z$ ($x = X/S, y = Y/S, z = Z/S$)	\longrightarrow						

Computed by..... Checked by.....

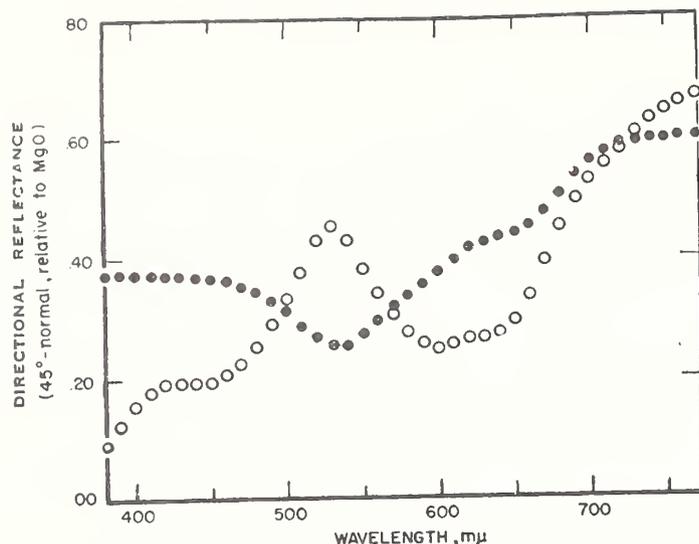


FIGURE 4. Spectral directional reflectance (45°-normal relative to magnesium oxide) of two of the master standards of colors for kitchen and bathroom accessories (Orchid, see solid circles; Kitchen Green, see open circles).

It is often hard to tell from inspection of spectrophotometric curves such as these what color will be perceived. Note, for example, that the Kitchen Green standard reflects more strongly in the long-wave (red) extreme of the spectrum than the Orchard standard does.

(a) Summation

Tables 3(a), (b) and (c) give computation forms for evaluation of the colors of nonself-luminous specimens that transmit, scatter, or reflect incident light. Table 3(a) refers to illuminant A; table 3(b) to illuminant B; and table 3(c), to illuminant C. Table 4 gives the spectral reflectances of the two master standards illustrated in figure 4, and it also gives the products provided for in the computation form shown in table 3(c). These products evaluate the spectral irradiance of the observer's eye by energy reflected perpendicularly from the master standards illuminated by standard illuminant C (representative of average daylight), relative to a magnesium-oxide reflectance standard similarly illuminated and viewed. This evaluation is in terms of the amount of each of the primaries $x_\lambda, y_\lambda, z_\lambda$ required by the standard observer to form a stimulus equivalent to each 10-m μ band of the spectrum of the reflected energy. The sums of these products gives by Grassman's law a similar evaluation of the entire beam reflected from the specimen toward the eye of the observer; these sums are called the *tristimulus values, X, Y, Z*. The luminous directional reflectance is found as $Y/100,000$; and the chromaticity coordinates, x, y, z , are found by dividing X, Y , and Z , respectively, by the corresponding sum, $X+Y+Z$. Table 5 lists these results for the two master standards and they are engraved on each duplicate standard issued by the National Bureau of Standards. Figure 5 is the (x, y)-chromaticity diagram showing the location of the points representing the chromaticities of these standards in accord with their chromaticity coordinates (x, y). Comparison of figure 5 with figure 3 indicates that the light reflected from these two stand-

ards, if seen as from a self-luminous source with dark surroundings, would differ too little from daylight to deserve a special hue name. The orchid specimen illuminated by daylight would reflect light almost distinctive enough to take the designation, purplish pink; that reflected from the kitchen green specimen would almost take the designation, yellowish green. It is thus important to note that color perception is not related in a simple way to the chromaticity diagram, but depends importantly on whether the object is seen as a self-luminous source in a dark surrounding field, or as a part of an illuminated scene in an object-color environment.

TABLE 4. Spectral directional reflectances of the Orchard and Kitchen Green standards together with details of computation of their tristimulus values X, Y, Z

Wave-length <i>mμ</i>	Re- flect- ance	Kitchen Green tristimulus values			Re- flect- ance	Orchid tristimulus values		
		X	Y	Z		X	Y	Z
380	0.091	0	2	0.372	1	7
390	.122	2	11	.372	7	33
400	.153	13	0	62	.372	32	1	150
410	.179	59	2	281	.371	122	3	582
420	.191	236	7	1,136	.371	459	14	2,207
430	.193	578	24	2,823	.371	1,112	45	5,427
440	.193	767	51	3,848	.370	1,471	97	7,377
450	.196	767	87	4,045	.367	1,437	163	7,574
460	.208	699	144	4,014	.362	1,217	251	6,985
470	.224	509	237	3,354	.355	807	376	5,315
480	.251	279	406	2,375	.344	383	557	3,255
490	.291	106	686	1,535	.330	120	778	1,740
500	.331	17	1,126	948	.312	16	1,061	894
510	.376	33	1,817	572	.289	26	1,397	439
520	.428	247	2,766	305	.267	154	1,725	190
530	.451	687	3,578	175	.253	385	2,007	98
540	.429	1,195	3,925	84	.255	710	2,333	50
550	.380	1,627	3,736	33	.271	1,160	2,664	23
560	.341	2,005	3,356	13	.294	1,729	2,893	11
570	.308	2,255	2,817	6	.318	2,328	2,909	6
580	.277	2,332	3,214	4	.338	2,845	2,701	5
590	.260	2,336	1,723	3	.355	3,189	2,353	4
600	.249	2,228	1,324	2	.378	3,383	2,009	3
610	.256	2,131	1,069	1	.399	3,322	1,666	1
620	.263	1,859	829	1	.416	2,941	1,312	1
630	.267	1,418	585	0	.426	2,262	933
640	.276	1,019	398433	1,599	625
650	.295	693	261441	1,036	391
660	.335	456	169454	618	229
670	.394	279	102476	337	123
680	.450	166	60504	186	68
690	.495	85	31534	91	33
700	.529	43	15559	46	16
710	.554	22	8575	22	8
720	.580	11	3587	11	4
730	.607	5	2592	5	2
740	.628	3	1596	2	1
750	.644	1	1598	1	1
760	.657	1	1600	1	1
770	.668	1	0602	1
Total	27,170	33,561	25,633	35,574	31,750	42,378

TABLE 5. Luminous directional reflectance, Y , and chromaticity coordinates, x, y , of the Kitchen Green and Orchard master standards for standard illuminant C

Color standard	Luminous directional reflectance, Y	Chromaticity coordinates	
		x	y
Kitchen Green	0.34	0.315	0.389
Orchid32	.324	.289

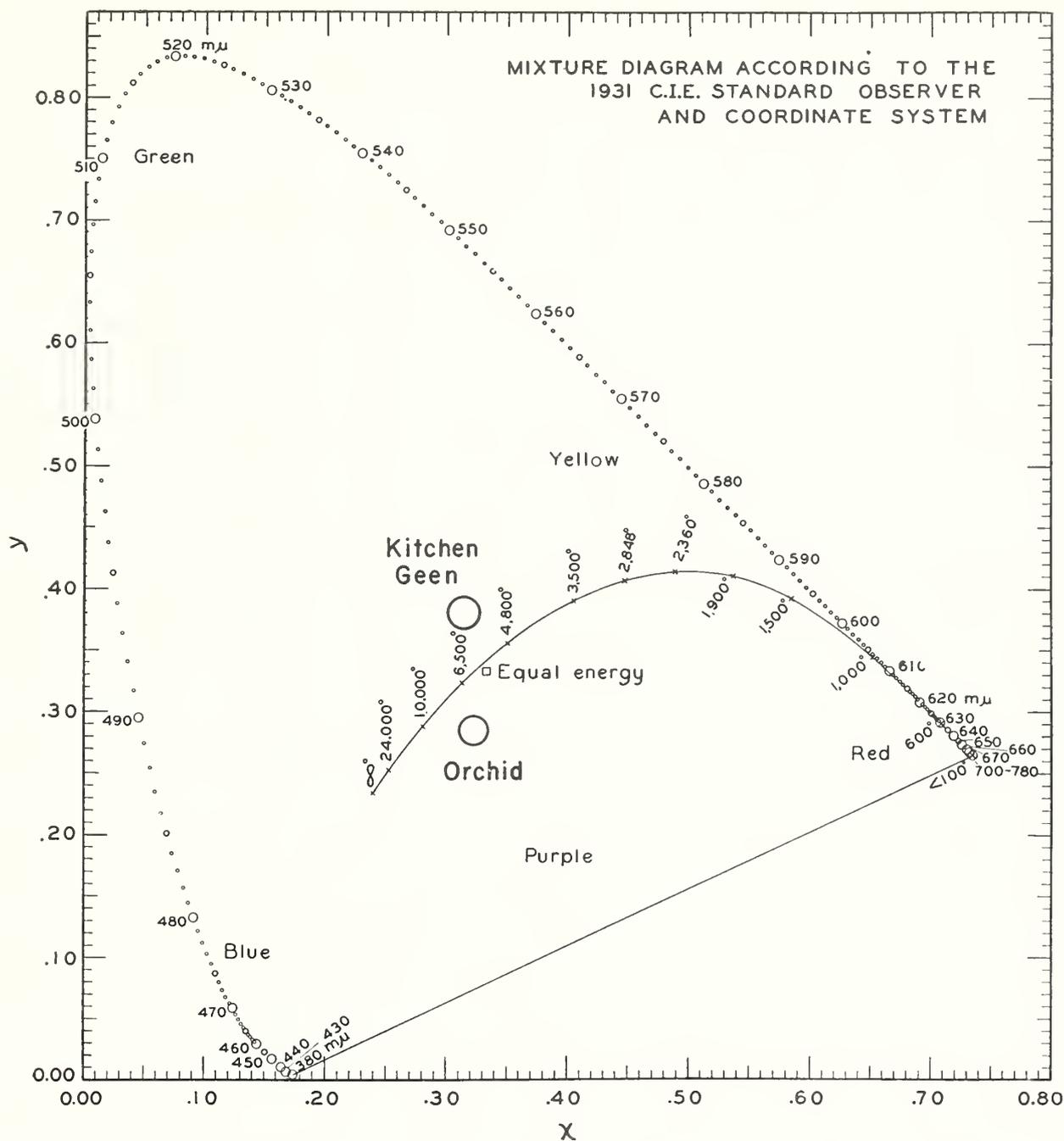


FIGURE 5. Chromaticities of Kitchen Green and Orchid on the (x,y) -chromaticity diagram.

Note that both of these colors are represented by points within the area found by Kelly (see fig. 3) to be too little different from daylight to deserve a hue name. This serves to emphasize that color perception is not related to position on the chromaticity diagram in any simple way. It depends importantly upon whether the object is self-luminous and viewed against a dark background or is nonself-luminous and viewed in the object-color environments that we are accustomed to.

(b) Selected Ordinate Method

The labor of computing X, Y, Z or Y, x, y , corresponding to pairs of spectrophotometric curves in accord with tables 3(a), 3(b), or 3(c) to see how the colors of the corresponding specimens compare is considerable. Often the degree of metamerism exhibited by the pair is sufficiently small that the comparison can be made directly from the curves, themselves. There is still frequent need, however, in the establishment of color standards and tests for conformity to those standards to compute the tristimulus values, X, Y, Z , and a short-cut method yielding

nearly the accuracy of the summation method has come into considerable use. In this method the spectrophotometric curve, instead of being read at every 5 or 10 $m\mu$, is read at a series of selected ordinates different for each illuminant. Instead of multiplying the reflectance or transmittance of the specimen by the tristimulus values of the spectrum of the illuminant, $\bar{E}_x, \bar{E}_y, \bar{E}_z$, the selected ordinates are spaced proportionately closer in the wavelength regions where the tristimulus values are higher, and the corresponding readings of spectral reflectance are simply added. Table 6 gives [44] selected ordinates

TABLE 6. Selected ordinates (in millimicrons) for computing tristimulus values, *X, Y, Z*, for specimens under standard illuminants

Number	<i>X</i>	<i>Y</i>	<i>Z</i>	<i>X</i>	<i>Y</i>	<i>Z</i>	<i>X</i>	<i>Y</i>	<i>Z</i>
STANDARD ILLUMINANT A			STANDARD ILLUMINANT B			STANDARD ILLUMINANT C			
1	444.0	487.8	416.4	428.1	472.3	414.8	424.4	465.9	414.1
2*	516.9	507.7	424.9	442.1	494.5	422.9	435.5	489.4	422.2
3	544.0	517.3	429.4	454.1	505.7	427.1	443.9	500.4	426.3
4	554.2	524.1	432.9	468.1	513.5	430.3	452.1	508.7	429.4
5*	561.4	529.8	436.0	527.8	519.6	433.0	461.2	515.1	432.0
6	567.1	534.8	438.7	543.3	524.8	435.4	474.0	520.6	434.3
7	572.0	539.4	441.3	551.9	529.4	437.7	531.2	525.4	436.5
8*	576.3	543.7	443.7	558.5	533.7	439.9	544.3	529.8	438.6
9	580.2	547.8	446.0	564.0	537.7	442.0	552.4	533.9	440.6
10	583.9	551.7	448.3	568.8	541.5	444.0	558.7	537.7	442.5
11*	587.2	555.4	450.5	573.1	545.1	446.0	564.1	541.4	444.4
12	590.5	559.1	452.6	577.1	548.7	448.0	568.9	544.9	446.3
13	593.5	562.7	454.7	580.9	552.1	450.0	573.2	548.4	448.2
14*	596.5	566.3	456.8	584.5	555.5	451.9	577.3	551.8	450.1
15	599.4	569.8	458.8	588.0	559.0	453.9	581.3	555.1	452.1
16	602.3	573.3	460.8	591.4	562.4	455.8	585.0	558.5	454.0
17*	605.2	576.9	462.9	594.7	565.8	457.8	588.7	561.9	455.9
18	608.0	580.5	464.9	598.1	569.3	459.8	592.4	565.3	457.9
19	610.9	584.1	467.0	601.4	572.9	461.8	596.0	568.9	459.9
20*	613.8	587.9	469.2	604.7	576.7	463.9	599.6	572.5	462.0
21	616.9	591.8	471.6	608.1	580.6	466.1	603.3	576.4	464.1
22	620.0	595.9	474.1	611.6	584.7	468.4	607.0	580.5	466.3
23*	623.3	600.1	476.8	615.3	589.1	470.8	610.9	584.8	468.7
24	626.9	604.7	479.9	619.1	593.9	473.6	615.0	589.6	471.4
25	630.8	609.7	483.4	623.3	599.1	476.6	619.4	594.8	474.3
26*	635.3	615.2	487.5	628.0	605.0	480.2	624.2	600.8	477.7
27	640.5	621.5	492.7	633.4	611.8	484.5	629.8	607.7	481.8
28	646.9	629.2	499.3	640.1	619.9	490.2	636.6	616.1	487.2
29*	655.9	639.7	508.4	649.2	630.9	498.6	645.9	627.3	495.2
30	673.5	659.0	526.7	666.3	650.7	515.2	663.0	647.4	511.2
Multiplying factors:									
30 ordinates	0.03661	0.03333	0.01185	0.03303	0.03333	0.02842	0.03268	0.03333	0.03938
10 ordinates	.10984	.10000	.03555	.09909	.10000	.08526	.09804	.10000	.11812

for illuminant A (incandescent lamp light), illuminant B (noon sunlight), and illuminant C (average daylight). Table 7 gives the spectral directional reflectances of the Kitchen Green standard read by interpolation from a large-scale copy of figure 4 for the selected ordinates for illuminant C, together with the sums of these reflectances both for 10 ordinates and for 30. It will be noted that after applying the multiplying factors listed in table 6 under standard illuminant C, the tristimulus values, *X, Y, Z*, for the Kitchen Green standard are found again to a close approximation (compare 0.271, 0.335, and 0.256 from table 7 with 0.272, 0.336, and 0.256, respectively, found from table 4 by dividing the totals by 100,000).

Ten selected ordinates sometimes give significant information; note in table 7 that they have yielded results differing by not more than 3 in the third decimal from those (table 4) obtained by 10-*mμ* summation. Thirty selected ordinates often are sufficient; and one hundred selected ordinates are sufficient for all but a few very irregular spectral distributions (such as produced by gaseous discharge tubes) to which 5 *mμ*-summation is also inapplicable. These wavelengths are available for many illuminants in other publications [16, 108]. Nickerson [100] has published a study of the reliability of the selected-ordinate method of computation and of 10-*mμ* sum-

mation, both relative to 5-*mμ* summation taken as correct.

(c) Semigraphical Centroid Method

A still more rapid method has been developed by Robinson [121] for the reduction of spectrophotometric curves that can be satisfactorily approximated by a series of straight lines. The curve is divided into a number of segments, each segment covering a wavelength interval for which the wavelength centroid has already been computed, and a straight-line approximation is drawn in by visual estimation for each segment. The straight-line approximating one segment of the curve need not, and usually does not, join that approximating the next segment. A few spectrophotometric curves will be encountered that are sufficiently simple to be reduced by means of two segments or even by a single straight line; four to eight segments are most often used; segments as small as 20 *mμ* are occasionally required. For curves so irregular as to require segments smaller than 20 *mμ* this method is scarcely shorter than 10-*mμ* summation.

The method is based upon two principles. First, the spectral distribution of each primary may be considered as a variable mass distribution along the spectrophotometric curve. If the curve is a straight

line for a certain wavelength interval, the center of gravity for that interval must fall somewhere on the line, and the wavelength of this center of gravity can be computed from the spectral distribution of the primary itself. Table 8 gives the wavelength centroids for X , Y , and Z in the spectra of illuminants A, B, and C from 400 to 700 $m\mu$ taken in 1, 2, 4, 8, and 15 intervals.

TABLE 7. Spectral directional reflectances of Kitchen Green standard (see fig. 5) read for the selected ordinates for standard illuminant C (see table 6)

Number	X		Y		Z	
1	0.192		0.216		0.187	
2*	.193	0.193	.285	0.285	.192	0.192
3	.194		.334		.193	
4	.197	.209	.371		.193	
5*	.209	.209	.401	.401	.193	.193
6	.232		.430		.193	
7	.450		.445		.193	
8*	.410	.410	.451	.451	.193	.193
9	.370		.447		.193	
10	.346		.439		.193	
11*	.328	.328	.423	.423	.194	.194
12	.311		.406		.194	
13	.297		.388		.195	
14*	.284	.284	.372	.372	.196	.196
15	.275		.359		.199	
16	.268		.346		.201	
17*	.262	.262	.334	.334	.203	.203
18	.256		.325		.205	
19	.250		.311		.208	
20*	.248	.248	.299	.299	.211	.211
21	.251		.287		.214	
22	.254		.276		.217	
23*	.258	.258	.269	.269	.221	.221
24	.261		.261		.226	
25	.263		.251		.233	
26*	.266	.266	.250	.250	.242	.242
27	.267		.254		.255	
28	.271		.262		.276	
29*	.285	.285	.266	.266	.294	.294
30	.354		.289		.382	
Totals	8.302	2.743	10.047	3.350	6.489	2.139
Times factor	0.271	0.269	0.335	0.335	0.256	0.253

The second principle is that the center of gravity of a reflectance curve over any interval lies somewhere on the straight line connecting the centers of gravity of any two subdivisions of that interval. The method is therefore simply to locate the point on the straight-line approximations for each segment that corresponds to the wavelength in table 8; then connect these points in pairs by straight lines, locate new points at the wavelengths given in table 8 for the double intervals, and so on until one straight line for the entire spectrum is found. The tristimulus value is then found by reading on the scale of spectral reflectance the ordinate on this line corresponding to the wavelength given in the final columns of table 8 for X , Y , or Z , as the case may be, and applying the indicated multiplying factor.

Figure 6 shows the application of this method to the curve of spectral-directional reflectance for the Kitchen Green (SKC-15) standard to obtain its luminous directional reflectance for illuminant C.

Eight segments have been used, 400 to 440, 440 to 480, 480 to 520, 520 to 560, 560 to 600, 600 to 640, 640 to 680, and 680 to 700 $m\mu$. The straight-line approximations to these segments are shown by solid lines. The points on these straight lines for the wavelength centroids given in table 8 under Y_2 for illuminant C (431.7, 466.0, 505.0, 541.8, 577.4, 616.4, 653.3, and 688.0 $m\mu$) are connected in pairs by four dashed lines. The points on these lines for the wavelength centroids given under Y_3 (463.1, 531.0, 588.6, 655.2 $m\mu$) are connected in pairs by two dashed lines. The points on these lines for the wavelength centroids given under Y_4 (526.6, 592.2 $m\mu$) are connected by the final dashed line, and the scale of directional reflectance read for the wavelength centroid given under Y_5 (557.0 $m\mu$) is found to be 0.334 (point indicated by small square). This value multiplied by the factor 1.0000 given in table 8 is the desired evaluation of luminous directional reflectance, $Y=0.334$, which is seen to be in good agreement with the value (0.336) found by 10- $m\mu$ summation in table 4.

2. Direct Colorimetry

The scales of the standard coordinate system [20] are defined independent of the properties of the standard observer, and it is possible at least in theory to obtain the tristimulus values, X, Y, Z , of any color by comparison of it by a normal observer with a mixture of a limited number of fixed or variable stimuli.

(a) Tristimulus Colorimeters

Tristimulus values, X, Y, Z , may be obtained by direct comparison of the unknown light with an optical mixture of three primary lights in a divided

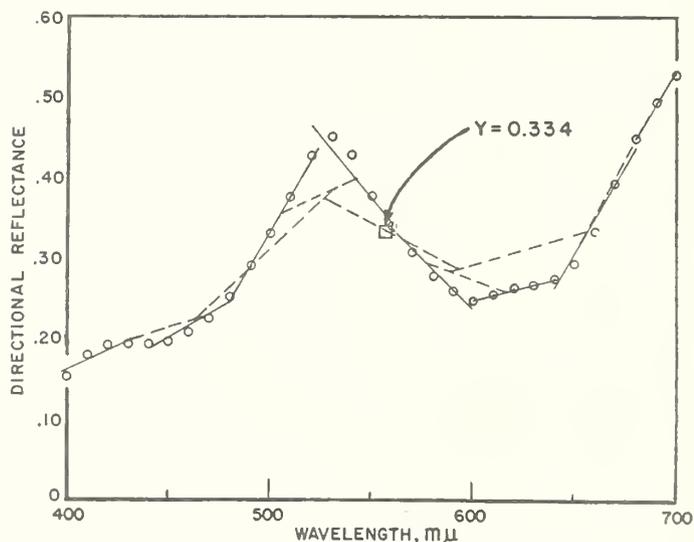


FIGURE 6. Application of the Robinson [121] centroid method to find the reflectance of the Kitchen Green standard for standard illuminant C (representative of average daylight).

The solid lines are the approximate representation of the spectrophotometric curve, segment by segment. Four dashed lines connect the centroids of these segments (see table 9, Y -scale for illuminant C). Two dashed lines in turn connect the centroids of these four lines; and the value of daylight reflectance ($Y=0.334$) is read from the centroid (about 557 $m\mu$) of the line connecting the centroids of the semifinal lines.

TABLE 8.

X scale for A—illuminant 400 to 700 mμ						Y scale for A—illuminant 400 to 700 mμ						Z scale for A—illuminant 400 to 700 mμ					
λ	X ₁	X ₂	X ₃	X ₄	X ₅	λ	Y ₁	Y ₂	Y ₃	Y ₄	Y ₅	λ	Z ₁	Z ₂	Z ₃	Z ₄	Z ₅
400	414.33					400	414.44					400	414.34				
420						420						420					
421	432.25	429.83				421	433.47	431.85				421	432.32	429.95			
440						440						440					
441	450.64		449.96			441	452.47		465.26			441	450.87		452.08		
460						460						460					
461	469.06	458.44				461	472.27	467.42				461	469.66	459.84			
480				514.83		480						480					
481	486.79					481	492.24			532.52		481	489.04			461.83	
500						500						500					
501	515.90	499.02				501	512.08	506.95				501	508.77	494.74			
520						520						520					
521	532.95		544.50			521	531.24		534.45			521	528.64		498.73		
540						540						540					
541	551.89	547.10				541	550.81	542.48				541	548.05	532.64			
560					595.22	560					572.07	560					462.11
561	571.42					561	570.49					561	569.23				
580						580						580					
581	590.93	583.12				581	590.16	579.81				581	589.55	576.74			
600						600						600					
601			601.63			601	609.84		593.17			601	608.39		582.30		
620	610.30					620						620					
621	629.58	618.33				621	629.37	617.06				621	627.70	612.31			
640				608.94		640				598.07		640				582.46	
641	649.00					641	648.92					641	644.17				
660						660						660					
661	668.56	654.03				661	668.53	653.83				661	0	644.17			
680			656.35			680			656.09			680			644.17		
681	688.29	688.29				681	688.28	688.28				681	0	0			
700						700						700					
Multiplying factor — 1.0983						Multiplying factor — 1.0000						Multiplying factor — 0.3555					

X scale for B—illuminant 400 to 700 mμ						Y scale for B—illuminant 400 to 700 mμ						Z scale for B—illuminant 400 to 700 mμ					
λ	X ₁	X ₂	X ₃	X ₄	X ₅	λ	Y ₁	Y ₂	Y ₃	Y ₄	Y ₅	λ	Z ₁	Z ₂	Z ₃	Z ₄	Z ₅
400	414.40					400	414.50					400	414.42				
420						420						420					
421	432.14	429.71				421	433.37	431.72				421	432.21	429.83			
440						440						440					
441	450.33		447.98			441	452.17		463.74			441	450.55		449.99		
460						460						460					
461	468.81	457.40				461	472.00	466.46				461	469.40	458.75			
480						480						480					
481	486.55					481	491.87			528.45		481	488.68			456.78	
500						500						500					
501	515.70	496.34				501	511.70	505.55				501	508.39	493.43			
520						520						520					
521	532.84		542.78			521	531.10		531.95			521	528.51		496.33		
540						540						540					
541	551.73	546.69				541	550.66	541.97				541	547.92	532.27			
560					576.20	560					561.45	560					456.89
561	571.17					561	570.24					561	568.98				
580						580						580					
581	590.68	582.14				581	589.91	578.80				581	589.32	575.76			
600						600						600					
601			599.04			601	609.70		590.42			601	608.28		580.37		
620	610.16					620						620					
621	629.46	617.85				621	629.25	616.59				621	627.65	611.95			
640				605.19		640				594.28		640				580.49	
641	648.91					641	648.83					641	644.07				
660						660						660					
661	668.43	653.68				661	668.40	653.49				661	0	644.07			
680			655.71			680			655.46			680			644.07		
681	688.12	688.12				681	688.11	688.11				681	0	0			
700						700						700					
Multiplying factor — 0.9907						Multiplying factor — 1.0000						Multiplying factor — 0.8522					

TABLE 8—Continued

X scale for C—illuminant 400 to 700 m μ						Y scale for C—illuminant 400 to 700 m μ						Z scale for C—illuminant 400 to 700 m μ					
λ	X ₁	X ₂	X ₃	X ₄	X ₅	λ	Y ₁	Y ₂	Y ₃	Y ₄	Y ₅	λ	Z ₁	Z ₂	Z ₃	Z ₄	Z ₅
400	414.42					400	414.52					400	414.43				
420						420						420					
421	432.09	429.64				421	433.32	431.66				421	432.16	429.76			
440						440						440					
			447.17												449.12		
441	450.20					441	452.05					441	450.42				
460						460						460					
461	468.71	456.98				461	471.90	466.05				461	469.29	458.30			
480						480						480					
				486.21						526.57							454.99
481	486.46					481	491.72					481	488.54				
500						500						500					
501	515.63	495.35				501	511.55	504.97				501	508.24	492.94			
520						520						520					
			542.00							530.95						495.51	
521	532.81					521	531.06					521	528.47				
540						540						540					
541	551.69	546.57				541	550.61	541.82				541	547.88	532.17			455.06
560					565.28	560					556.95	560					
561	571.07					561	568.27					561	568.88				
580						580						580					
581	590.59	581.78				581	589.82	577.35				581	589.22	575.39			
600						600						600					
			598.03							588.62						579.68	
601	610.10					601	609.64					601	608.20			579.78	
620						620						620					
621	629.42	617.67				621	629.21	616.40				621	627.66	611.82			
640						640						640					
				603.74													
641	648.87					641	648.79					641	643.83	643.83	643.83		
660						660						660					
661	668.37	653.53				661	668.35	653.34				661					
680						680						680					
			655.46							655.21							
681	688.05	688.05				681	688.03	688.03				681					
700						700						700					

Multiplying factor — 0.9804

Multiplying factor — 1.0000

Multiplying factor — 1.1810

photometric field. Since the primaries of the ICI standard colorimetric coordinate system are imaginary, such a tristimulus colorimeter cannot be made directly. It must be calibrated by measurements of four known stimuli, and then may yield tristimulus values, X, Y, Z , by a transformation the reverse of that indicated in eq. 3. Since, except by accident, the color matches set up in a tristimulus colorimeter designed to cover any substantial part of the color totality with a single set of primaries exhibit serious metamerism, the field has to be relatively small, such as to subtend about two degrees at the observer's eye. Even with this small field, the fixation point has to be kept near the center of the field in order to prevent the observer from becoming confused by the appearance of the parafoveal mismatch (see fig. 1, circles). This small angular size of field severely limits the precision of setting compared to what is possible by direct comparison of large specimens in daylight. Furthermore, the metamerism also prevents one normal observer from getting the same reading as another except by accident. To mitigate the effects of these individual observer differences, it is customary to adjust the scales of the instrument separately for each observer so that for some standard stimulus (such as illuminant B or C), the tristimulus values will agree with those for the standard observer. Even with this correction, if a reasonable approximation to the standard values of X, Y, Z , is to be assured, either the readings of a group of five or ten observers must be averaged, or a color standard yielding a spectral composition similar to that of the unknown specimen must be used.

It will be seen that tristimulus colorimeters give but poor information regarding the unknown specimen. Their application to product-control problems is negligible. But because of the ease of calibration and simplicity of the theory, they are very useful visual research tools. Tristimulus colorimeters have been built and described by Allen [4], Guild [40], Wright [139], Verbeek [134], Donaldson [25], and Newhall [95]. The Wright instruments have spectrum primaries; the other five have primaries formed by combining a light source with glass filters. To the Guild and Wright instruments we owe our accurate information regarding the properties of the normal visual system, which have been expressed in terms of the standard observer. Only the Donaldson [25] colorimeter in the six-primary form is applicable to product control.

The foregoing instruments make up the comparison-field mixture by optical combination of light beams from different sources either so that a sum of the separate effects is obtained by continuous exposure, or so that a time-weighted average is obtained by successive stimulation at a frequency too high to produce flicker. A very simple and widely used tristimulus colorimeter based on this latter principle is obtained by taking four disks that have been cut along a radius, by interlocking them so as to expose a sector of each, and by causing them to rotate on the spindle of a motor so rapidly that neither the separate sectors nor even flicker is perceived. Such an arrangement for combining colors by rotary mixture is called a Maxwell disk. The four disks provide the necessary three degrees of freedom in the

adjustment for a match, and if the tristimulus values of the component disks be known (X_1, Y_1, Z_1 ; X_2, Y_2, Z_2 ; X_3, Y_3, Z_3 ; X_4, Y_4, Z_4) the tristimulus values (X, Y, Z) of the mixture can be computed from the fractions, f_1, f_2, f_3, f_4 , of the total area occupied by the respective sectors:

$$\left. \begin{aligned} X &= f_1 X_1 + f_2 X_2 + f_3 X_3 + f_4 X_4, \\ Y &= f_1 Y_1 + f_2 Y_2 + f_3 Y_3 + f_4 Y_4, \\ Z &= f_1 Z_1 + f_2 Z_2 + f_3 Z_3 + f_4 Z_4 \end{aligned} \right\}, \quad (4)$$

where, of course, $f_1 + f_2 + f_3 + f_4 = 1$.

If the disks are chosen anew for each kind of unknown color to be measured so as to be all fairly similar in color to the unknown, the spectral composition of the mixture color is usually sufficiently nonmetameric that no restriction to the central 2° of the retina is required. Furthermore, any two normal observers with some experience at making the adjustment can check each other closely. The chief drawback of this simple arrangement for product-control work in color is the time lost in adjustment of the sector-disk areas. The motor must be turned off, brought to a stop, the disks loosened and readjusted, the motor turned on and allowed to resume speed several times to obtain a final setting of reasonably good precision.

Nickerson has described a disk colorimeter [99] that avoids the difficulties of the elementary Maxwell disk. Light reflected from the unknown specimen fills one half of a photometric field, and that from a stationary sector disk fills the other. By having the observer look at the sector disk through a rapidly rotating glass wedge, each sector is presented to view in sufficiently quick succession that no flicker is produced; and at the same time the sectors, since they are stationary may be continuously adjusted until a color match is obtained. A further advantage is obtained by extending the rotary scanning to the unknown specimen. In this way the average color of a notably nonuniform specimen such as that made up of coarse salt crystals may be obtained. The disk colorimeter is extensively used by the U. S. Department of Agriculture [102] for the color-grading of food products, and is well adapted to product-control colorimetry of many kinds. Frequent checks of the working standards of color are required for accurate work lest fading or soiling of them render calibration by formula 4 inapplicable.

(b) Dominant Wavelength and Purity

Another form of identifying a combination of lights to specify a color, alternate to the tristimulus form, is to determine the luminance (photometric brightness) of one spot of light of fixed spectral composition (such as average daylight) and the luminance of a spot of light of continuously variable spectral composition separately identified (as by wavelength in the spectrum). In this way the requisite three degrees of freedom in adjustment to a color match are supplied. This form of identification

leads naturally into a specification in which the luminance (or luminous transmittance, or luminous reflectance) of the unknown is given and the chromaticity is specified by two variables in polar coordinates. One of these variables is an angle, the other a radius, and both can be computed from the chromaticity coordinates of the fixed spot of light, the variable spot of light, and the unknown, these coordinates serving to locate the respective positions in the Maxwell triangle. If the fixed light is nearly achromatic, the angle often correlates well with the hue of the color perception, and the radius fairly well with its saturation.

The most fundamental way to specify the direction on the Maxwell triangle from the point representing the fixed light to the point representing the unknown light is by wavelength of the part of the spectrum required to make the match. If the unknown color can be matched by adding some part of the spectrum to the fixed light, it is said to have a spectral color, and the required wavelength is called its *dominant wavelength*. But if a color match is produced for the fixed light by adding some part of the spectrum to the unknown color, the unknown is said to be nonspectral, and the required wavelength is called the *complementary wavelength*. Either dominant wavelength or complementary wavelength may be obtained for the standard observer by drawing on the standard Maxwell triangle a straight line through the point representing the fixed light and that representing the unknown color, and then reading the wavelength corresponding to the point at which this line extended intersects the locus of spectrum colors. If the unknown color is plotted between the fixed light and the spectrum, the intersection gives the dominant wavelength; but if the fixed light is represented by a point intermediate to the unknown and the intersection of the straight line with the spectrum locus, the intersection indicates the complementary wavelength. Tables of slopes (or reciprocals of slopes) of lines connecting points representing various fixed lights (illuminants A, B and C, and the "equal energy" point) with various parts of the spectrum locus on the (x, y)-diagram have been published by Judd [56] as an aid in finding dominant wavelength from given chromaticity coordinates, x, y .

The degree of approach of the unknown color to the spectrum color is commonly indicated by the ratio of the amount of the spectrum color to the total amount of the two-part combination; this ratio is called purity, and if the amounts are specified in luminance units, the ratio is called *colorimetric purity*.

Colorimetric purity for nonspectral colors has been defined in two ways [53, 55, 131], both awkward and little used in actual practice. By far the most common convention is to express the amounts in units of the excitation sum, $X + Y + Z$; the resulting ratio is called *excitation purity* and corresponds simply to distance ratios on the Maxwell triangle of the standard colorimetric coordinate system. It is also conventional to define excitation purity for non-

spectral colors by assigning to colors that plot on the purple border a value of excitation purity equal to unity. Excitation purity, p_e , can therefore be defined simply in terms of the chromaticity coordinates, x_b, y_b , of the border (be it spectrum locus or purple boundary) at the intersection of the straight line connecting the point, x, y , representing the unknown color with the point, x_w, y_w , representing the fixed light, thus:

$$\text{or } \left. \begin{aligned} p_e &= (x - x_w) / (x_b - x_w), \\ &= (y - y_w) / (y_b - y_w), \end{aligned} \right\} \quad (5)$$

whichever form is determinable with the least rejection error.

Formulas for colorimetric purity and for converting colorimetric purity of spectral colors to excitation purity and the reverse have been derived by Judd [56], Hardy [44], and MacAdam [78, 108].

$$\text{or } \left. \begin{aligned} p_e &= (y_b / y) (x - x_w) / (x_b - x_w), \\ &= (y_b / y) (y - y_w) / (y_b - y_w), \end{aligned} \right\} \quad (6)$$

whichever is determinable with the least rejection error.

$$p_e = \frac{y_w p_c}{y_b + (y_w - y_b) p_c} \quad (7)$$

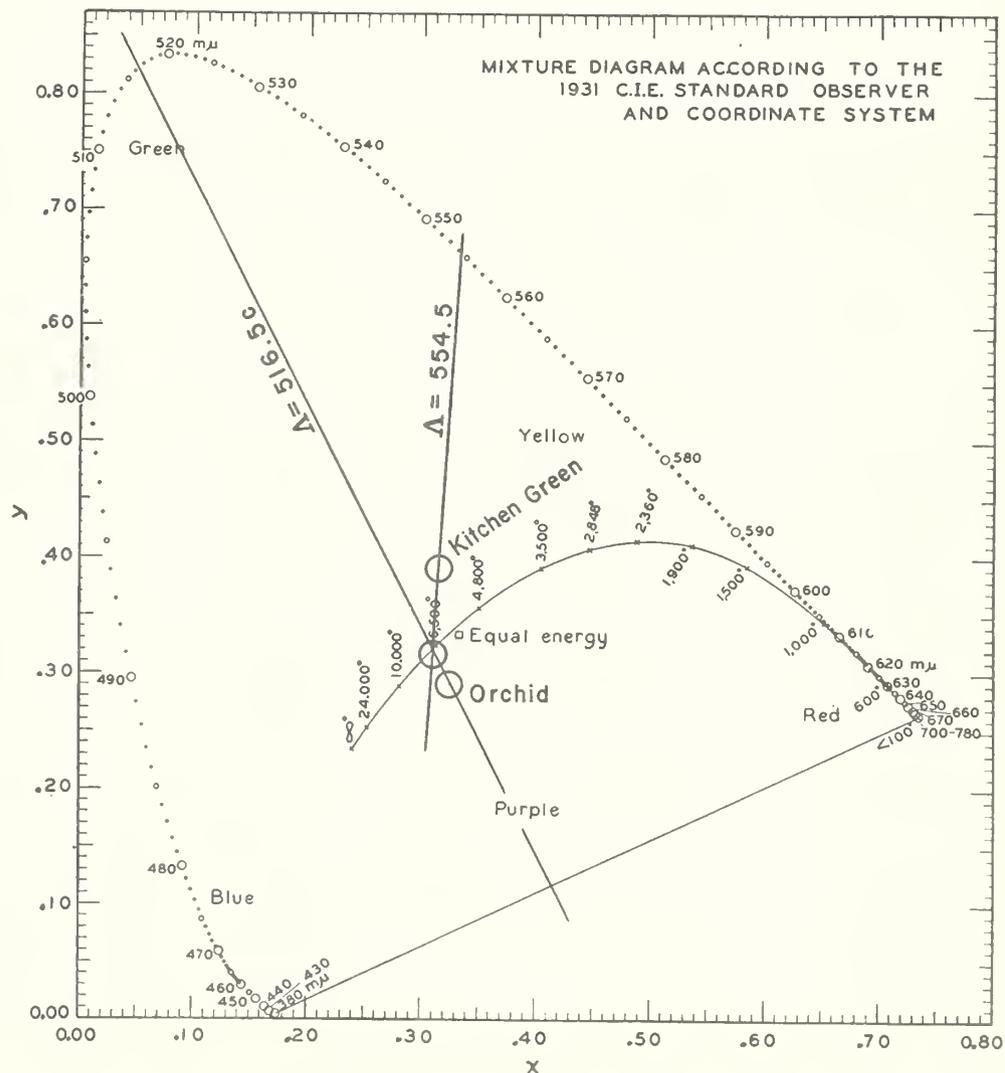
$$p_c = \frac{y_b p_e}{y_w + (y_b - y_w) p_e} \quad (8)$$

Similar formulas may be derived for nonspectral colors, depending upon which definition of colorimetric purity is followed. MacAdam [78, 108] gives tables facilitating these conversions.

Figure 7 indicates how dominant wavelength and excitation purity of the two standards would be found from their chromaticity coordinates, x, y , relative to illuminant C taken as the fixed light. Table 9 gives the dominant (or complementary) wavelength found as in figure 7 by the intersections of the straight lines with the spectrum locus. Table 9 also gives the excitation purities found by dividing the distance from the fixed point (illuminant C) to the

FIGURE 7. Graphical determination of the dominant (or complementary) wavelength and excitation purity of the Kitchen Green and Orchid standards illuminated by standard source C.

The wavelength is found by drawing a straight line between the point representing the chromaticity of the specimen and the point representing the chromaticity of the illuminant and reading the wavelength of the part of the spectrum locus intersected by an extension of this straight line. Excitation purity is the ratio of the distance between the specimen point and the illuminant point to the distance between the illuminant point and the border of the diagram (either spectrum locus or purple border).



specimen point by the total distance from the fixed point to the boundary (spectrum locus plus straight line connecting its extremes), or by eq. 5. Large-scale charts for reading dominant wavelength and purity relative to illuminant C are provided in the Hardy Handbook of Colorimetry [44].

TABLE 9. Dominant wavelengths and excitation purities of the Kitchen Green and Orchid master standards

Color standard	Chromaticity coordinates (from table 5)		Dominant wavelength	Excitation purity
	<i>x</i>	<i>y</i>		
Kitchen Green...	0.315	0.389	mμ 554.5	Percent 21.1
Orchid324	.289	516.5 C	13.4

Apparatus for the direct measurement of dominant wavelength and colorimetric purity has been designed by Nutting [105] and by Priest [115]. The degree of metamerism ordinarily obtained with such apparatus leaves it open to the same objections as have prevented tristimulus colorimeters with single sets of primaries from being useful for production control. There is a further disadvantage in the direct measurement of colorimetric purity that the luminance of the spectrum component has to be determined relative to the luminance of the mixture by separate photometry. Since there is usually a large chromatic difference between these two fields, simple equality-of-brightness settings are not reliable, and an auxiliary flicker photometer, as in Priest's apparatus [115], must be used. This method has been

III. Small-Difference Colorimetry

In the fundamental colorimetry of lights and objects, a single standard is used for each class of specimen. Opaque surfaces are referred either to the ideal perfect diffuser or to a sufficiently thick layer of magnesium oxide deposited from the smoke of magnesium turnings or ribbon burning in air [113]. Transparent objects, such as gelatine films, crystal, or glass plates, are referred to the same thickness of air; transparent solutions, to the same thickness of distilled water or solvent. Self-luminous objects, such as fluorescent lamps, cathode-ray tubes, television screens, and incandescent lamps, are measured relative to one of the standard illuminants, usually illuminant A [20], or they are measured relative to illuminant A viewed through a filter of known spectral transmittance. The colors of specimens closely resembling the respective standards can be evaluated by direct colorimetry quite precisely and accurately; those differing radically in spectral composition, only with relative uncertainty. That is, near-white specimens, nearly clear glass plates, and incandescent lamps nearly equivalent to illuminant A present the simplest colorimetric problem; highly selective absorbers and emitters, like the rare-earth glasses and gaseous discharge tubes, present difficult measurement problems. In general, the greater the deviation in spectral composition between the unknown specimen and the standard, the greater the

found to exaggerate individual-observer differences; oftentimes observers will differ only slightly in the mixtures of spectrum light and fixed light that they find to be equivalent to an unknown color, but they will disagree importantly in their photometry of the components.

(c) Guild Vector Method

By using two known fixed lights and one variable (spectrum) light, Guild [41] was able to determine chromaticity coordinates, *x, y*, without resort to auxiliary photometric devices. A match is first set up for the unknown by a mixture of one fixed light with some portion of the spectrum, the amounts of both components being adjustable by the observer. A straight line is drawn on the chromaticity diagram connecting the points representing the fixed light and the spectrum light. Then the process is repeated with a second fixed light, and a second straight line is drawn on the chromaticity diagram. The intersection of the two straight lines gives the chromaticity coordinates of the unknown. This method was used by Guild chiefly to supplement the tristimulus colorimeter by measuring unknowns whose chromaticities plot outside of the triangle formed by plotting the points representing the primaries of the tristimulus colorimeter. Such unknowns may be evaluated on the tristimulus colorimeter only by the inconvenient expedient of mixing one (or two) of the primaries with the unknown. Since the matches obtained by the vector method are in general highly metameric, a group of observers is required to obtain a representative result.

uncertainty of the result obtained by a visual or a photoelectric colorimeter.

Modern photoelectric spectrophotometers have greatly extended the application of both visual and photoelectric colorimetry. They have supplied a rapid, practical way to calibrate working standards of color. If a fairly large group of specimens is at hand to be measured, say 20 or more, all of similar spectral composition, the most satisfactory way to measure them in the present state of colorimetric science is to evaluate one or two of them carefully by means of the spectrophotometer to serve as working standards, then obtain the color specifications of the remainder by visual or photoelectric determination of the difference between specimen and standard.

Another important application of small-difference colorimetry is the solution of such problems as the color changes produced by various deteriorative agents, or those produced by admixture of a colorant, or toner. In these problems, the color standard is the original unexposed or untuned specimen; and the chief concern is to evaluate character and amount of the color differences, the absolute evaluation of the colors being of secondary interest, or required only approximately.

1. Martens Photometer

One of the most useful visual devices for deter-

mining relative luminance of surfaces of nearly identical spectral selectivity is the Martens photometer [82, 106]. Figure 8 shows the Martens photometer combined with a diffuse illuminator to form the Priest-Lange reflectometer [116]. This reflectometer is intended for the measurement of luminous reflectance of opaque specimens relative to reflecting standards of similar spectral reflectance. It is also adaptable to the measurement of luminous transmittance of transparent plates relative to transmitting standards similar in spectral transmittance to the unknown. Finally the Martens photometer removed from the mounting, shown in figure 8, may be used for the determination of the luminance of an unknown self-luminous surface relative to a spectrally similar standard of known luminance. The superior usefulness of the Martens photometer arises from the convenience of the adjustment for equality of brightness between the two halves of the photometer field and from the fact that in a well-made instrument the dividing line between the half-fields is exceptionally narrow so that it is often invisible when a brightness match has been set. The addition of the simple lens in front of the photometer permits the object viewed

to be thrown into focus and extends the use of the device to the reflectometry* of small specimens.

The Martens photometer is customarily used in one of two ways. In the interchange method the specimen is compared directly with the standard and the angle Θ_1 , required for a brightness match is read from the instrument scale set so that the zero of the scale corresponds to maximum obscuration of the standard side of the field. Then specimen and standard are interchanged, and angle Θ_2 read. The luminance ratio, B/B_s , is found as

$$B/B_s = (\tan \Theta_1)(\cot \Theta_2). \quad (9)$$

In the substitution method the standard is first compared with a comparison surface and the angle Θ_1 required for a brightness match is read. Then the specimen is substituted for the standard and the angle Θ_2 found. The luminance ratio is found as

$$B/B_s = (\tan^2 \Theta_2) / (\tan^2 \Theta_1). \quad (10)$$

If specimen and standard are of considerably different chromaticities, the setting for equality-of-brightness is not adequately reproducible. If the specimen and standard are of considerably different

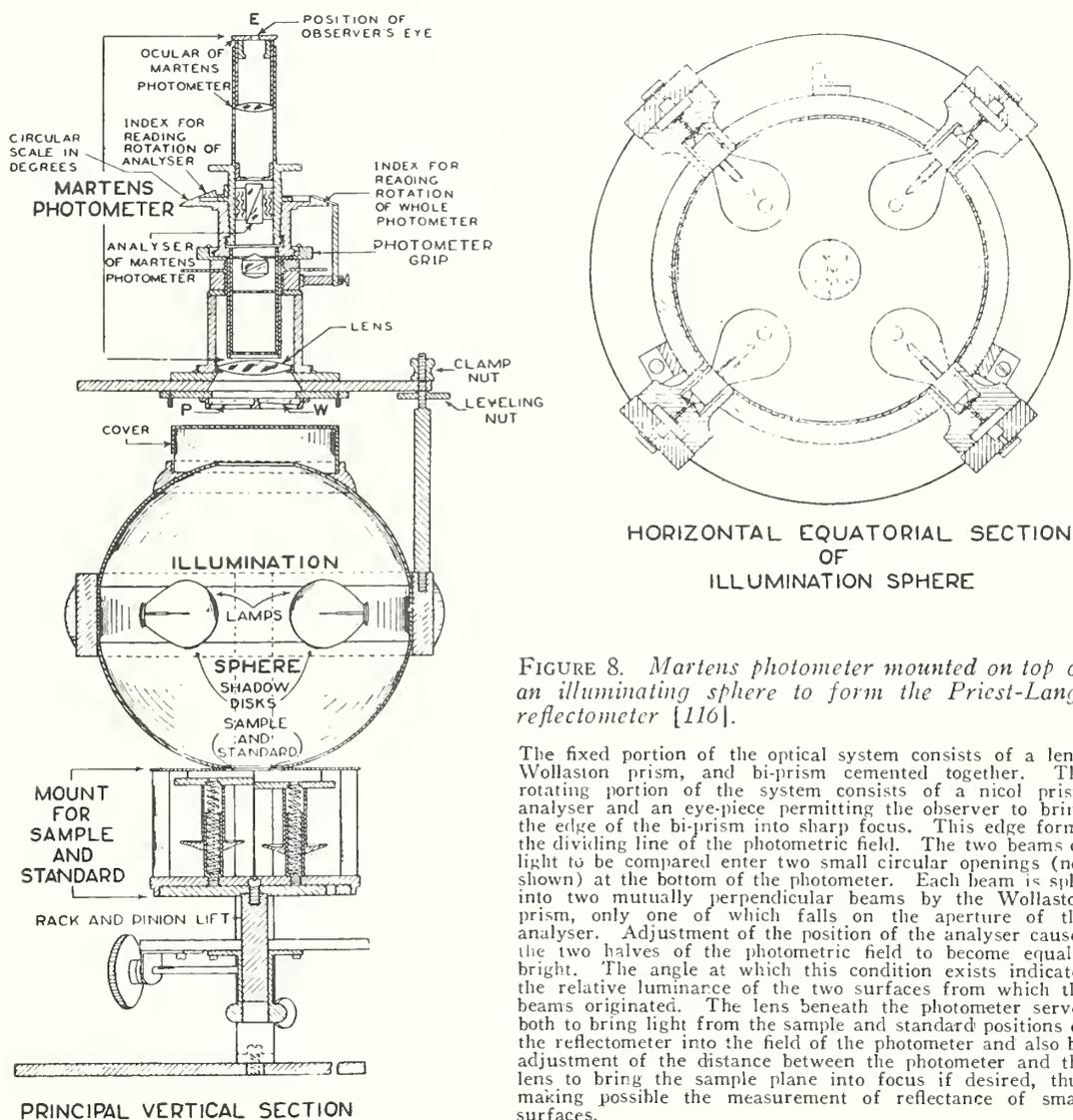


FIGURE 8. Martens photometer mounted on top of an illuminating sphere to form the Priest-Lange reflectometer [116].

The fixed portion of the optical system consists of a lens, Wollaston prism, and bi-prism cemented together. The rotating portion of the system consists of a nicol prism analyser and an eye-piece permitting the observer to bring the edge of the bi-prism into sharp focus. This edge forms the dividing line of the photometric field. The two beams of light to be compared enter two small circular openings (not shown) at the bottom of the photometer. Each beam is split into two mutually perpendicular beams by the Wollaston prism, only one of which falls on the aperture of the analyser. Adjustment of the position of the analyser causes the two halves of the photometric field to become equally bright. The angle at which this condition exists indicates the relative luminance of the two surfaces from which the beams originated. The lens beneath the photometer serves both to bring light from the sample and standard positions of the reflectometer into the field of the photometer and also by adjustment of the distance between the photometer and the lens to bring the sample plane into focus if desired, thus making possible the measurement of reflectance of small surfaces.

spectral compositions but of nearly the same chromaticities, a projection of the macular pigment of the observer appears on the field of the instrument (usually 6° diameter) and renders a reproducible setting very difficult. These limitations apply to all visual photometers.

2. Chromaticity-difference colorimeter

The determination of chromaticity coordinates, x, y , by comparison of the unknown specimen with a working standard of similar spectral reflectance can be carried out visually with high precision by means of a differential colorimeter described by Judd [61]. The adjustment of the chromaticity of the comparison field to match the standard field is by two double wedges, one of greenish and the other of yellowish glass. Since the light from the comparison field must pass through both the yellow and the green wedge, some of the radiant energy being subtracted by each, it is sometimes called a subtractive colorimeter; see figure 9, which gives a schematic diagram. The standard and comparison fields are brought into juxtaposition by means of a Lummer-Brodhun cube having a double-trapezoid pattern subtending $9 \times 13^\circ$ at the observer's eye. The adjustment to near equality of brightness to facilitate detection of chromaticity differences is by movement of the projection lamp that illuminates both standard and comparison surfaces. The comparison is made either by incandescent-lamp light, or by artificial daylight obtained by insertion of Corning Aklo and Daylite glass

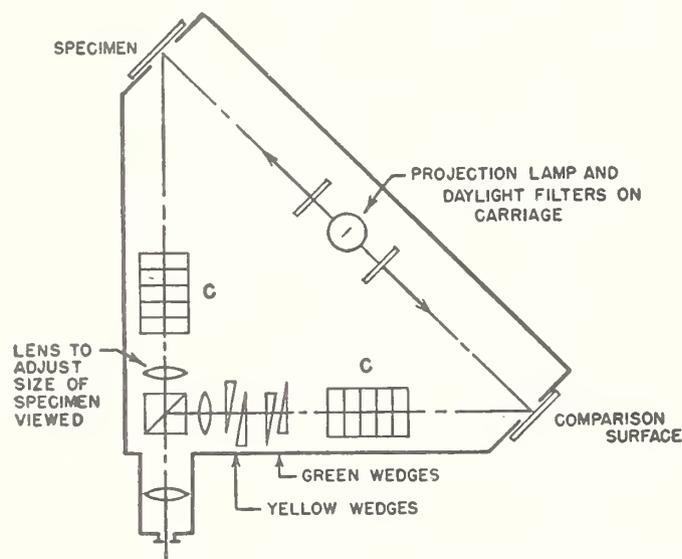


FIGURE 9. Schematic diagram of the chromaticity-difference colorimeter.

Both specimen and comparison surface are illuminated perpendicularly by artificial daylight conveniently variable by movement of the lamp carriage. The specimen is viewed through the Lummer-Brodhun cube and two lenses, the comparison surface is viewed by total reflection in the cube through two double wedges, one of yellowish, one of greenish glass. Auxiliary filters of the same kinds of glass may be inserted either in the specimen beam or the comparison beam. Adjustment of the viewing lens permits the observer to focus approximately on the dividing plane of the Lummer-Brodhun cube, the back half of which has etched on it a double-trapezoid pattern giving the well-known contrast field for enhancement of color differences. The power of the second lens determines the size of specimen area from which light is visible. C represents the containers for the auxiliary yellow and green filters.

found to yield a satisfactory heat-free duplication of illuminant C for this purpose.

A substitution method is usually employed with this colorimeter, a match first being set up between the standard and comparison surfaces by adjustment of the wedges. Then the unknown specimen is substituted for the standard and the wedges readjusted to restore the match. Auxiliary yellow and green filters may be inserted either in the beam reflected from the specimen or in the comparison beam, if the specimen differs sufficiently from the standard to require it. The differences in wedge settings can be calibrated in terms of differences in the chromaticity coordinates, x, y , from the known spectral transmittances of the wedges by computation as in table 3(c), provided the spectral directional reflectances of the comparison surface are known approximately. The calibrations for a comparison surface of magnesium oxide are given in figure 10. Note that, over the range illustrated, there is a nearly linear relation between differences in wedge settings, ΔY , ΔG , and the corresponding differences in chromaticity coordinates Δx , Δy . This calibration has been carried out for about 100 widely differing comparison surfaces. It has been found that the calibration is chiefly a function of the chromaticity coordinates, x, y , of the comparison surface; so calibrations for comparison surfaces intermediate in chromaticity to those already calibrated may usually be found satisfactorily by interpolation.

Because of the large patterned field of high luminance and the convenience of the brightness adjustment, this instrument takes full advantage of the ability of the observer to detect small chromaticity

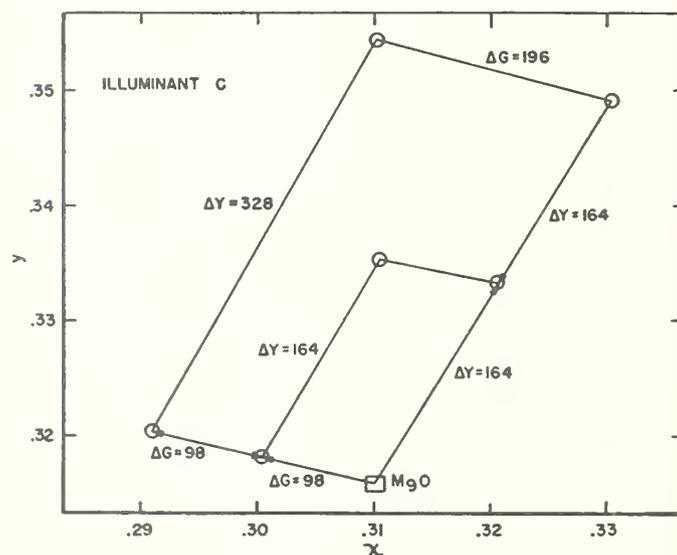


FIGURE 10. Calibration of the yellow and green wedges of the chromaticity-difference colorimeter for near whites and near grays in terms of the (x, y) -chromaticity diagram.

The illuminant is standard source C . Note that in this region of the diagram an increase of 100 units of the yellow wedge corresponds to an increase in x -coordinate by about 0.0060, and an increase in y -coordinate by about 0.0100. An increase of 100 units of the green wedge corresponds to a decrease in the x -coordinate by about 0.0100, and an increase in the y -coordinate by about 0.0020. These calibration coefficients depend somewhat on the spectral composition of the energy reflected from the comparison surface to produce any given chromaticity, but they are determined approximately by the chromaticity itself.

differences. If specimen and comparison surfaces are similar in spectral composition, the settings for match may be repeated generally within 0.001 in chromaticity coordinates, x, y . For chromaticity differences not much larger than this, it is advantageous to use the interchange method instead of the substitution method. In this method the unknown specimen is compared first directly with the standard, then the two are interchanged and a second setting made. No comparison surface is required, and the uncertainty of the determination is one-half that obtained by the more rapid substitution method. If the surfaces be not spectrally similar, or if they be viewed through many yellow and green filters, a faint projection of the macular pigment may appear in the photometric field. Such an appearance lowers the precision of setting somewhat and is also correlated with failure of the result to agree with indirect colorimetry by way of the spectrophotometer [118]. This failure may often be by as much as 0.003 in x or y .

The chromaticity-difference colorimeter has the further disadvantage of requiring a skilled operator. The calibrations, too, are time-consuming; and the instrument is not applicable to some spectrally selective specimens because nearly homogeneous energy is too little changed in spectral composition by passage through the wedges.

This colorimeter has been used in setting up a color standard for ruby mica [63], for inspection of working standards, transparent and opaque, for conformity to a master standard (such as the Kitchen and Bathroom Accessory Colors), and for general

colorimetry by difference both for fluorescent and nonfluorescent specimens [118].

3. Photoelectric tristimulus colorimeters

If three photocells could be adjusted, as by glass filters, so that their responses were proportional throughout the visible spectrum to some linear combination (as in formula 3) of the standard ICI distribution curves (see figure 2), then they could be used to test whether any two light beams have the same color according to eq. 2, and could be made to yield direct measurements of tristimulus values, X, Y, Z [35, 43]. There have been several good attempts to build a photoelectric tristimulus colorimeter suitable for general use [15, 49, 50, 111, 138], but with incomplete success [51, 133]. Figure 11 shows the degree to which filters designed by Hunter [50] to duplicate the ICI standard observer and simultaneously to adjust a projection lamp to ICI illuminant C have succeeded. If A, G , and B represent the settings obtained for a specimen relative to those for a standard magnesium-oxide surface with the amber, green, and blue filters, respectively, approximate tristimulus values, X, Y, Z , may be found as:

$$\left. \begin{aligned} X &\cong 0.80A + 0.18B, \\ Y &\cong 1.00G, \\ Z &\cong 1.18B \end{aligned} \right\} \quad (11)$$

Approximate chromaticity coordinates, x, y , may then be found in the usual way ($x = X/(X+Y+Z)$), $y =$

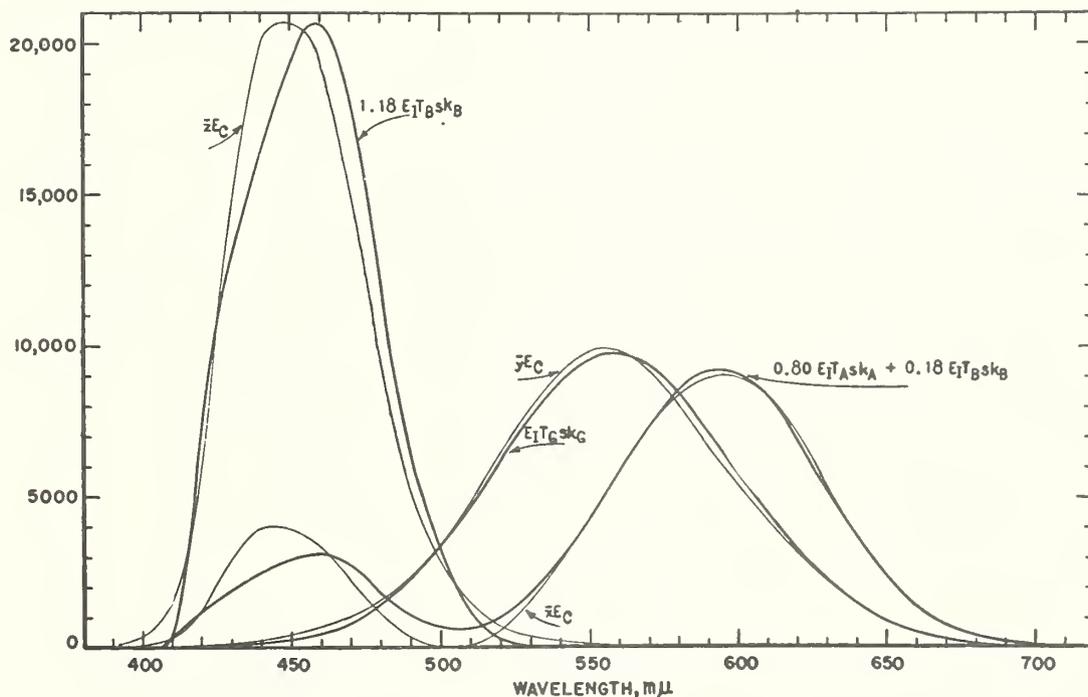


FIGURE 11. The tristimulus values of the spectrum of standard source C according to the ICI standard observer compared to the spectral sensitivities of the source-filter-photocell combinations designed by Hunter [50] for photoelectric tristimulus colorimetry with three filters.

Note that the duplication of the yE_C -function is fairly good, but the short-wave portions of the other two functions are more poorly duplicated. Departures of this amount limit the usefulness of these filters to the measurement of color differences between nonmetameric or slightly metameric pairs.

$Y/(X+Y+Z)$). Figure 12 shows the discrepancies that this use of the Hunter filters introduce. These discrepancies are roughly proportional to the distance from the point representing the magnesium-oxide standard, and are frequently larger than 0.02 in x or y , that is, more than 10 times a reasonable chromaticity tolerance for most colorimetric work.

For the comparison of specimens of similar spectral composition (such as near-white surfaces), however, the degree of duplication of the ICI standard observer shown in figure 11 is sufficient. Figure 13 refers to the small rectangle near the center of figure 12 and indicates the degree of agreement between measurements by means of the chromaticity-difference colorimeter and photoelectric tristimulus colorimetry by

means of the multipurpose reflectometer. Figure 13 indicates that the discrepancies are less than 0.001 in x or y for comparison of near-white surfaces with magnesium oxide. Figure 14 illustrates an application of photoelectric tristimulus colorimetry to near-whites in which the instrument is used to test white casein paints for one of the requirements of Federal Specification TT-P-23a. Another application of considerable importance is evaluation of the yellowness index:

$$\text{Yellowness index} = (A - B) / G. \quad (12)$$

This index yields a scale of yellowness increasing from zero for the standard magnesium-oxide surface or any equally nonselective surface to positive values for yellowish surfaces and negative values for bluish

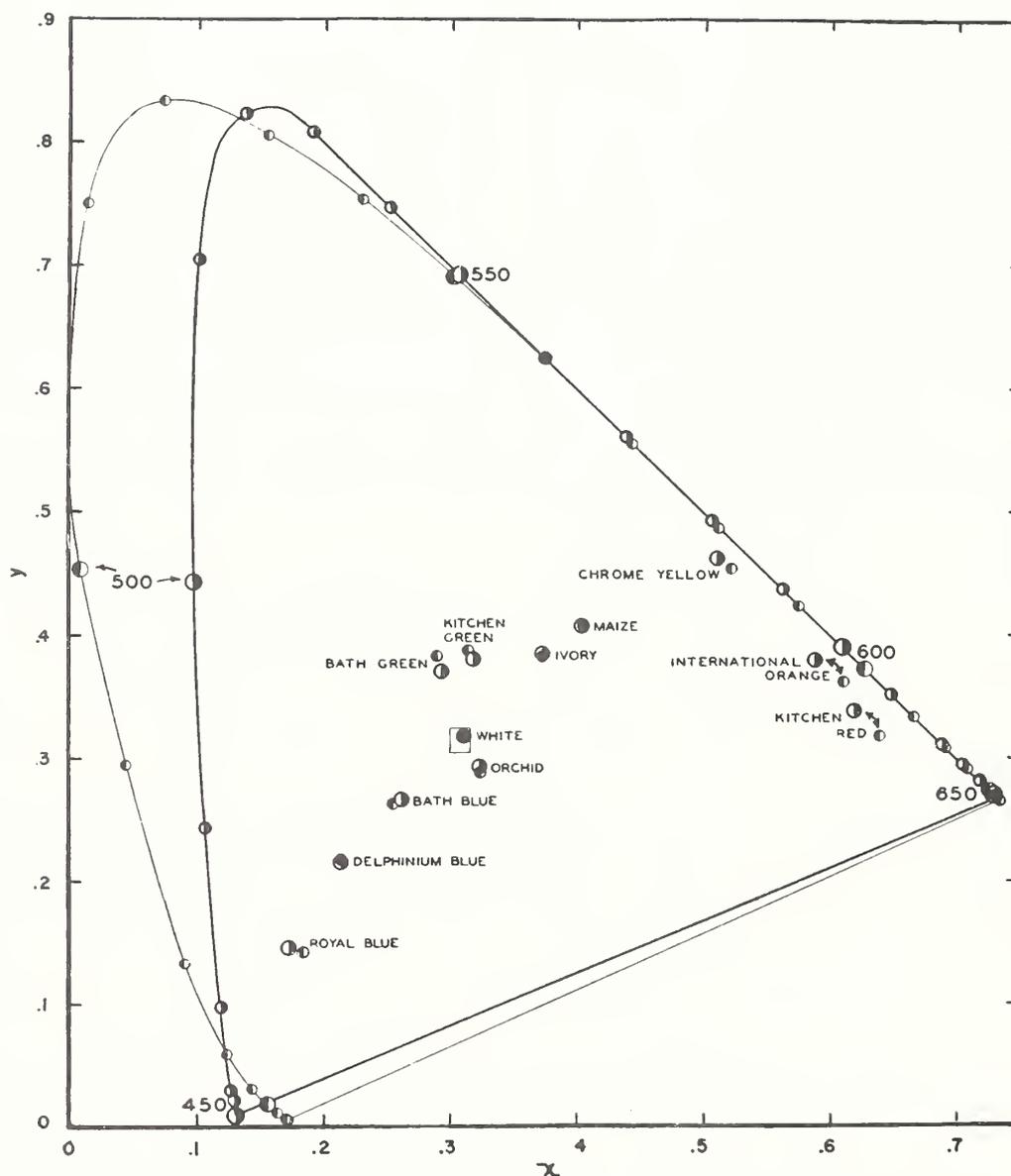


FIGURE 12. Discrepancies in chromaticity introduced by the use of the source-filter-photocell combinations shown in figure 11 (Hunter [150]).

The white (MgO) point is common to both plots, and in general the discrepancies increase with distance from this point. Nearly all of these discrepancies far exceed the least differences tolerable in colorimetry of industrial products.

• From spectrophotometer and ICI observer.

From multipurpose reflectometer.

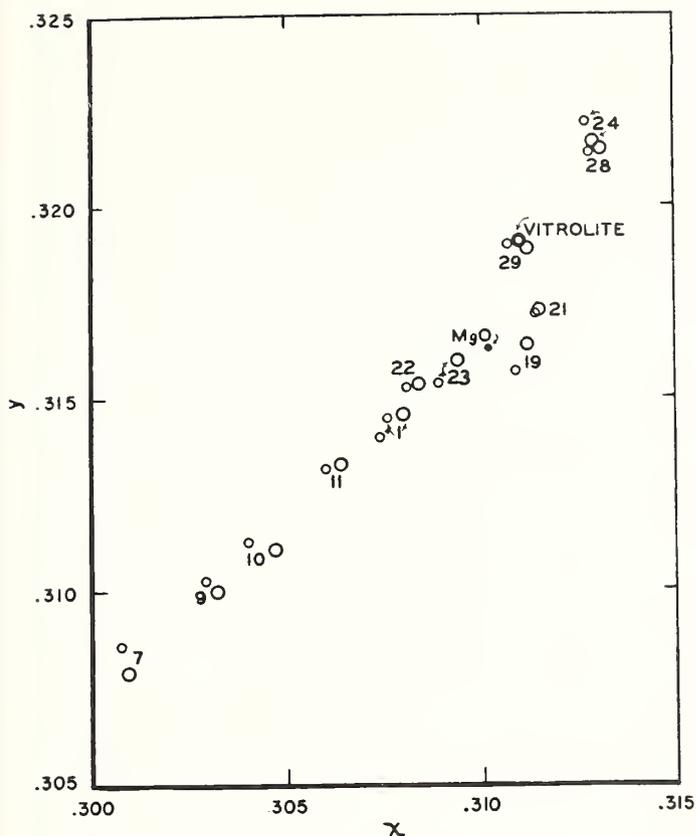


FIGURE 13. Comparison of photoelectric tristimulus colorimetry (fig. 11) of a series of near-white specimens of vitreous enamel with visual colorimetry of them by means of the chromaticity-difference colorimeter (fig. 9).

The point representing the Vitrolite plaque used as standard is common to both plots. Note that for these near whites the discrepancies are smaller than 0.0010 in x - or y -coordinate. Errors of this size are nearly always tolerable in the colorimetry of industrial products.

- From subtractive colorimeter.
- From multipurpose reflectometer.

surfaces. Photoelectric tristimulus colorimetry is listed as Method 425.2 in Federal Specification TT-P-141a dealing with test methods for paint, varnish, lacquer, and related materials.

If the limitations of photoelectric tristimulus colorimetry are appreciated, the method is most useful in product-control colorimetry of nonfluorescent specimens by difference from a working standard. A set of 11 gray standards, ranging from black to white, has been developed at this Bureau for this purpose. These standards apply to the colorimetry of nearly nonselective specimens under the standard angular conditions of 45° illumination and normal viewing, or the equivalent. The precision of the method is comparable, though perhaps not quite equal, to the best that can be done by eye. No unusual qualifications or extended special training is required for an operator, and compared to visual colorimetry, or to indirect colorimetry by the spectrophotometer, the results are obtained very rapidly.

Photoelectric reflectometers and transmissometers of proved practical value are commercially available, as are also the filters separately.² They have been used for the comparison of paint vehicles, solutions,

² Gardner Laboratories, 4723 Elm St., Bethesda, Md. Photovolt Corp., 95 Madison Ave., New York, N. Y.

petroleum products, glasses, painted panels, papers, textiles, ceramics, plastics, and a wide variety of fabricated specimens.

In general, satisfactory agreement can be expected in using such photoelectric tristimulus colorimeters, for the determination of small chromaticity differences between nonmetameric pairs. And even for measurement of fairly sizeable nonmetameric chromaticity differences, such as analyzed spectrophotometrically in the upper portion of figure 15 (BPB 8/2 vs. MgO, BG 7/4 vs. BG 6/4), and small chromaticity differences with a moderate metameric component, such as shown in the lower left portion of figure 15 (Y1 vs. Y2), the discrepancy is in the neighborhood of 0.002 in x or y , which is negligible for many purposes. However, for highly metameric pairs, such as shown in the lower right portion of figure 15, the discrepancy may be expected to be in the neighborhood of 0.02 in x or y , just as it is for large chromaticity differences.

4. Perceptibility of Color Differences

In the interpretation of the importance of chromaticity differences based upon separation of the points representing the two chromaticities in the (x,y) -diagram, a warning is necessary. This diagram is considerably expanded in the green portion relative to the other portions, much as the Mercator projection of the earth's surface is expanded near the poles. Thus, two points separated by a given distance in the green portion of this diagram correspond

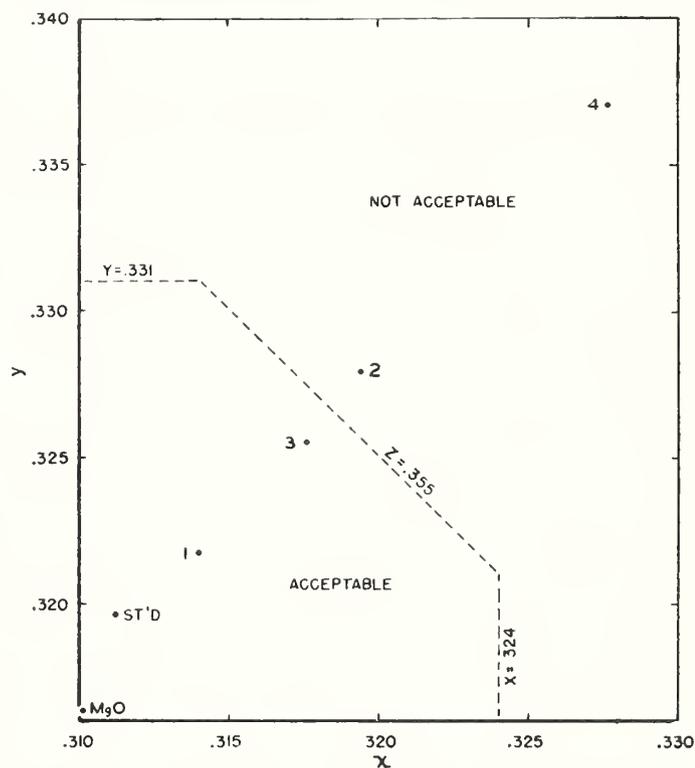


FIGURE 14. Application of photoelectric tristimulus colorimetry to test white casein paints for conformity to the requirements of Federal Specification TT-P-23a (Hunter [50]).

Samples 1 and 3 meet the requirements; samples 2 and 4 do not.

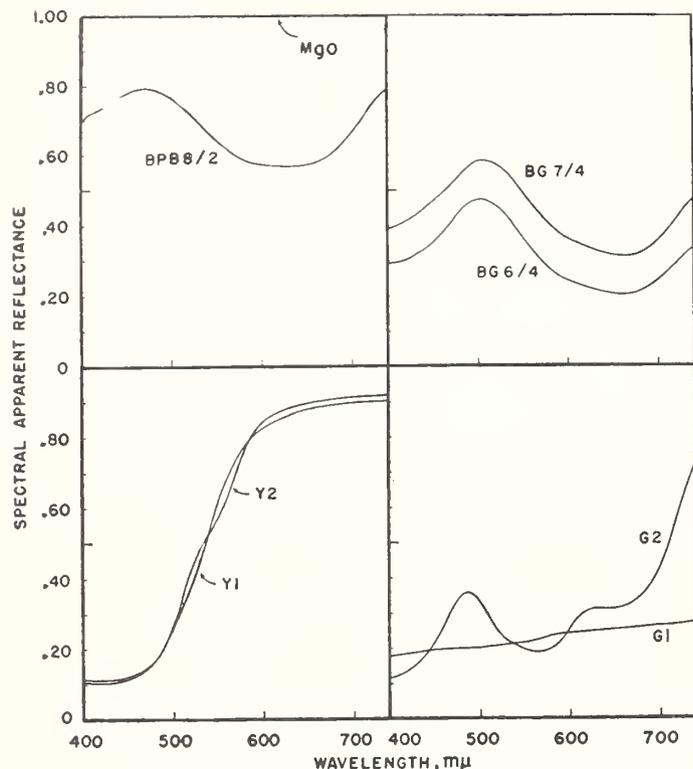


FIGURE 15. Illustration of degrees of metamerism (Hunter [50]).

The upper-right diagram shows the variation of reflectance with wavelength for a pair of Munsell papers (5BG 7/4 and 5BG 6/4) yielding a considerable, though nonmetameric, color difference. The upper left diagram (N 10/, 10B 8/2) also illustrates a color difference of considerable size but slight metamerism. The two lower diagrams refer to small color differences; that on the left, slightly metameric; that on the right, strongly. The presently available filters permit useful application of photoelectric tristimulus colorimetry to moderately large nonmetameric color differences, but not to very large color differences nor to strongly metameric differences.

to chromaticities that are much harder to distinguish under ordinary viewing conditions than two chromaticities separated by the same amount in other portions of the diagram. Furthermore, the bluish purple portion of the diagram is correspondingly compressed. Figure 3 indicates approximately this distortion; for example, the area designated "green" is more than 10 times as large as that designated "purplish blue" but actually refers, within perhaps a factor of 2, to the same number of distinguishable chromaticities. A more detailed and accurate indication of this distortion is given by the system of ellipses shown on figure 16. Under moderately good observing conditions, the distances from the central point of each ellipse to any point on its boundary correspond approximately to one hundred times the chromaticity difference just perceptible with certainty. These ellipses were drawn from a review of the literature in 1936 [57, 60], and subsequent extensive work published by Wright in 1941 [143, 144] and by MacAdam in 1942 [79, 80], has corroborated the essential correctness of the indicated chromaticity spacing. Figure 16 not only indicates approximately the extent to which the green portion of the diagram is expanded, and the bluish purple compressed, but also indicates from the considerable

departures of the ellipses from circles that, in general, the perceptive importance of a distance on the (x,y) -diagram depends not only on the position of the central point but also notably on the direction of the deviation from it.

If the properties of an observer be expressed with reference to other sets of primaries than the ICI standard by transformation in accord with formula 3, the chromaticity spacing yielded by the resulting Maxwell triangles may be made to vary widely. There have been several attempts so to choose primaries that the chromaticity spacing corresponds to its perceptibility, that is, yields uniform chromaticness scales [17, 50, 57, 77, 108, 124]. There have also been a number of fairly successful attempts to produce diagrams to serve the same purposes by other means [3, 28, 88, 89, 122, 125]. Figure 17 shows the uniform-chromaticness-scale (UCS) triangle according to Judd [57]. If the chromaticity coordinates of a color are x,y , in the standard ICI system, the color would have chromaticity coordinates, r,g , in this uniform-chromaticness-scale triangle in accord with eq. 13:

$$\left. \begin{aligned} r &= \frac{2.7760x + 2.1543y - 0.1192}{-1.0000x + 6.3553y + 1.5405} \\ g &= \frac{-2.9446x + 5.0323y + 0.8283}{-1.0000x + 6.3553y + 1.5405} \end{aligned} \right\} \quad (13)$$

On this diagram the ellipses of figure 16 would be equal tangent circles.

Figure 18 shows the rectangular uniform-chromaticness-scale (RUCS) diagram set up by Breckenridge and Schaub [17] by modification of the UCS diagram so as to place the point representing the equal-energy stimulus at the origin of the coordinate system. Since this stimulus under most observing conditions is perceived to have a nearly achromatic or neutral color, the coordinate axes of the RUCS system may be said to divide the chromaticity diagram approximately into four ranges of hues. The first quadrant corresponds to the hue range yellow to green; second quadrant, green to bluish purple; third quadrant, bluish purple to red, and fourth quadrant; red to yellow. The connection of the RUCS coordinates, x'',y'' , to the ICI standard chromaticity coordinates, x,y , is given in two steps by way of intermediate coordinates, x',y' .

$$\left. \begin{aligned} x' &= \frac{0.82303x + 0.82303y - 0.82303}{1.00000x - 7.05336y - 1.64023} \\ y' &= \frac{3.69700x - 5.07713y - 1.36896}{1.00000x - 7.05336y - 1.64023} \end{aligned} \right\} \quad (14)$$

$$\left. \begin{aligned} x'' &= 0.0750 - x' \\ y'' &= y' - 0.5000 \end{aligned} \right\} \quad (15)$$

Tables have been prepared for carrying out this transformation by interpolation with uncertainties less than one in the third decimal [94a]. The RUCS diagram, because of its use of rectangular coordinates, and because the significance of the coordinates in terms of color perception under ordinary condi-

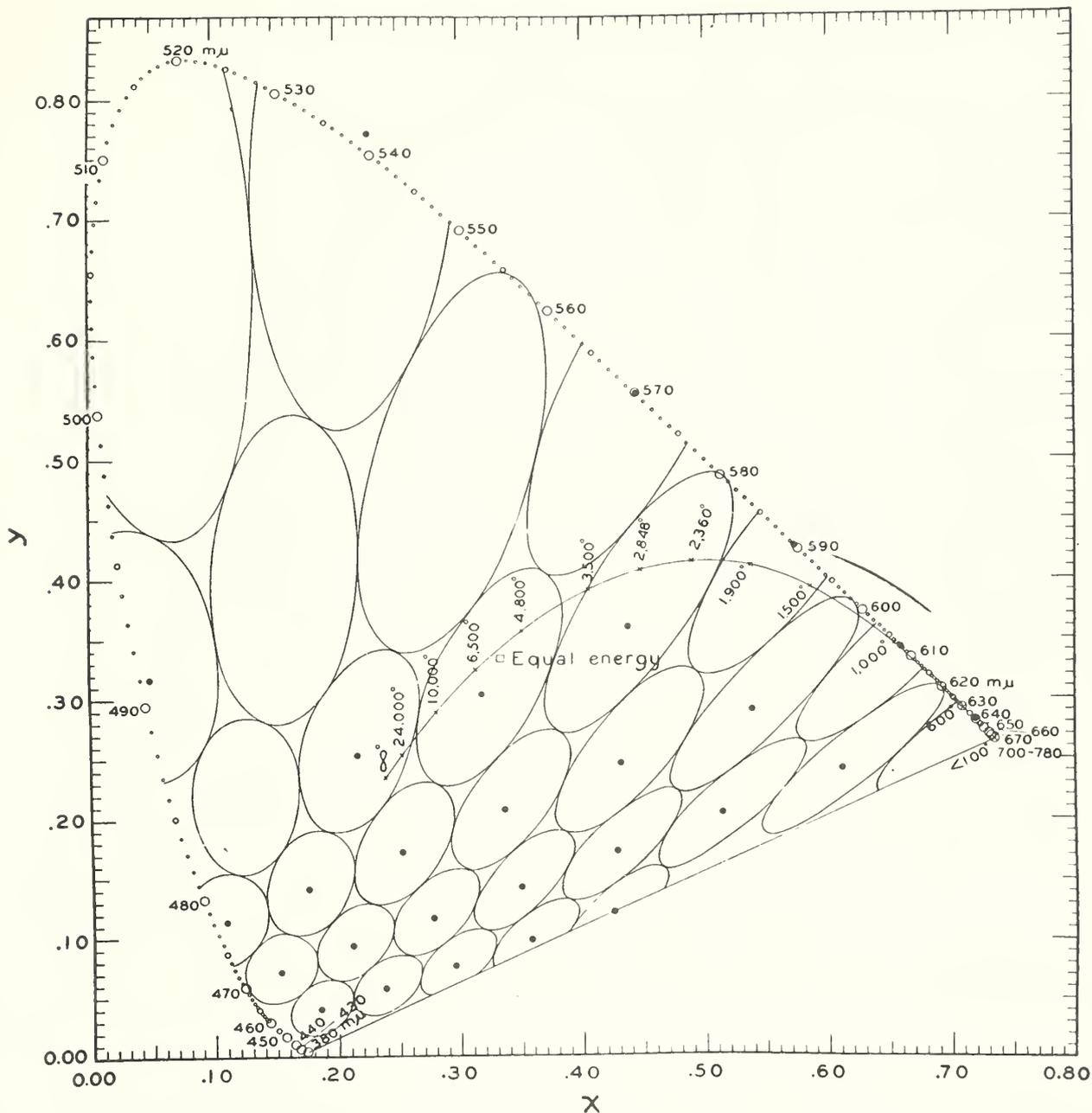


FIGURE 16. Approximate perceptibility of chromaticity differences on the (x,y) -chromaticity diagram.

The distances from points on the boundary of each ellipse to the indicated point within it all correspond approximately to one hundred times the chromaticity difference just perceptible with certainty under moderately good observing conditions.

tions is easy to grasp, has advantages for engineering applications. It was put forward as a suggestion for possible future adoption by international agreement.

Figure 19 shows the "alpha-beta" diagram set up by Hunter [50, 124] by modification of the UCS diagram to adapt it for convenient use with the multipurpose reflectometer and to take into account chromaticity-spacing data collected since 1936 [72, 79, 143]. These new data essentially corroborate the UCS diagram but indicate that the perceptibility of chromaticity differences for purple colors in the sense corresponding to changes parallel to the purple boundary (straight line connecting the extremes of the spectrum locus) is less relative to other differences by about a factor of two than is shown by the

UCS diagram. The connection of the "alpha-beta" coordinates, α, β , with the ICI standard chromaticity coordinates, x, y , is given by formula:

$$\left. \begin{aligned} \alpha &= \frac{2.4266x - 1.3631y - 0.3214}{1.0000x + 2.2633y + 1.1054} \\ \beta &= \frac{0.5710x + 1.2447y - 0.5708}{1.0000x + 2.2633y + 1.1054} \end{aligned} \right\} \quad (16)$$

To a sufficient approximation for small-difference colorimetry these chromaticity coordinates, α, β , can be found from the settings A, G, B , obtained by using the Hunter amber, green, and blue filters, respectively, of the multipurpose reflectometer [49, 50], by the simple relation:

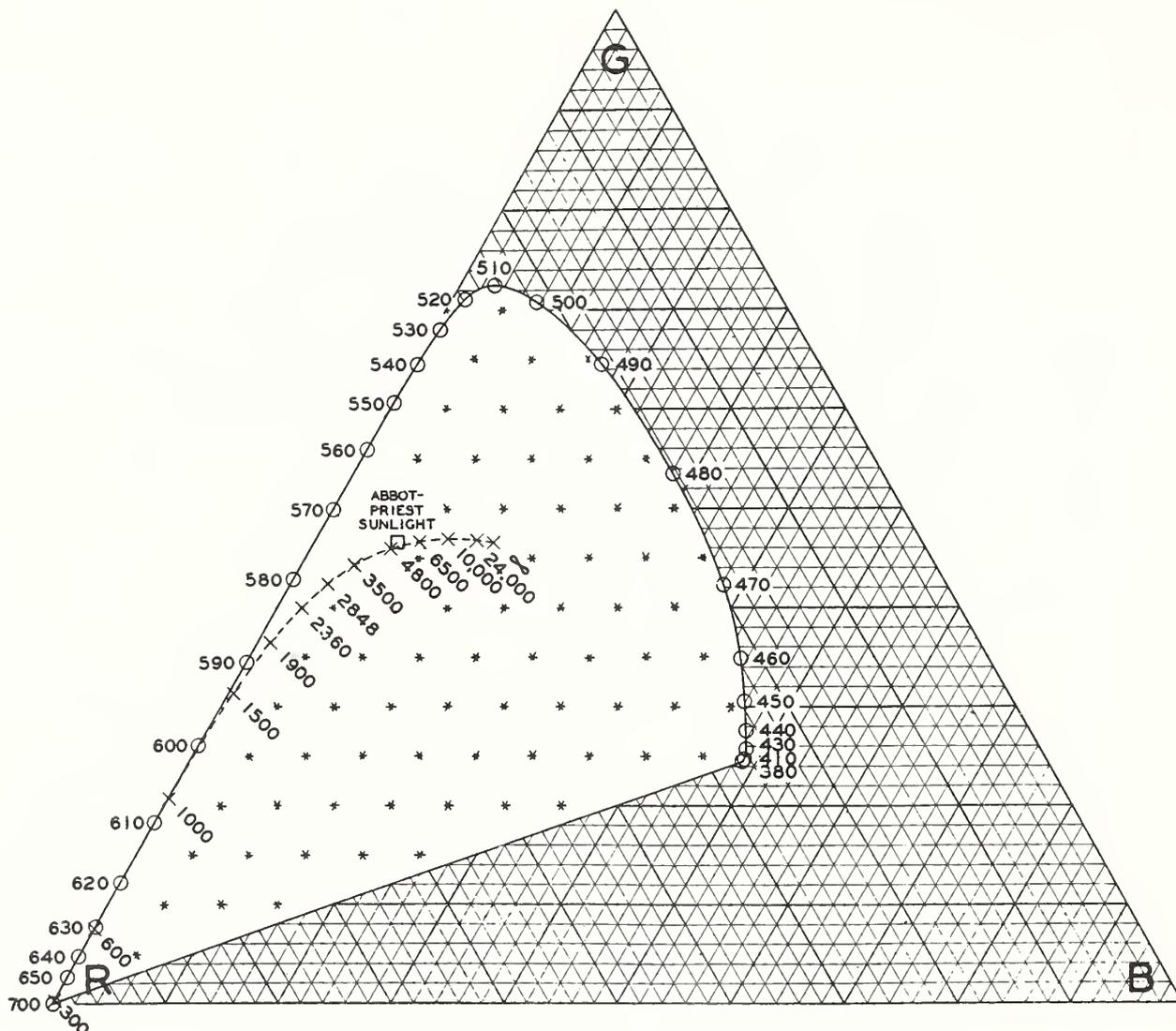


FIGURE 17. The uniform-chromaticness-scale (UCS) diagram.

Points on the spectrum locus are indicated in millimicrons. Points representing the chromaticities of light from a small hole in an enclosure are indicated by the temperature of the walls in degrees Kelvin. The ellipses of figure 16 correspond to a family of equal tangent circles on this diagram. The least perceptible chromaticity difference under the most favorable observing conditions corresponds on this diagram to a change in r , g , or b -coordinate by about 0.0004.

$$\alpha \cong \frac{A-G}{A+2G+B}$$

$$\beta \cong \frac{0.4(G-B)}{A+2G+B} \quad (17)$$

The least chromaticity difference perceptible with certainty under the most favorable observing conditions (luminance 50 or more millilamberts, field size 8° or more, convenient luminance adjustment about the condition of match) corresponds on each of the three uniform-chromaticness-scale diagrams just discussed to about 0.0004.

(a) NBS Unit of Color Difference

An empirical formula for estimating the perceptibility of color differences has been developed [61, 62] on the assumption that surface colors can be

represented by a three-dimensional figure in which length of a straight line is a close measure of the perceptibility of the difference between the colors represented by its extremes. In such a surface-color space the constant-lightness planes (representing colors of surfaces of constant luminous reflectance) would be perpendicular to the lines representing changes in lightness at constant chromaticness. The basic assumption thus permits a calculation of the color difference, ΔE , in terms of the chromaticness and lightness components, $\Delta E_c, \Delta E_l$, by Euclidean geometry, thus:

$$\Delta E = \left[\overline{\Delta E_c^2} + \overline{\Delta E_l^2} \right]^{1/2} \quad (18)$$

The size of the lightness component, ΔE_l , is evaluated by changes in the square-root of the luminous directional reflectance, thus:

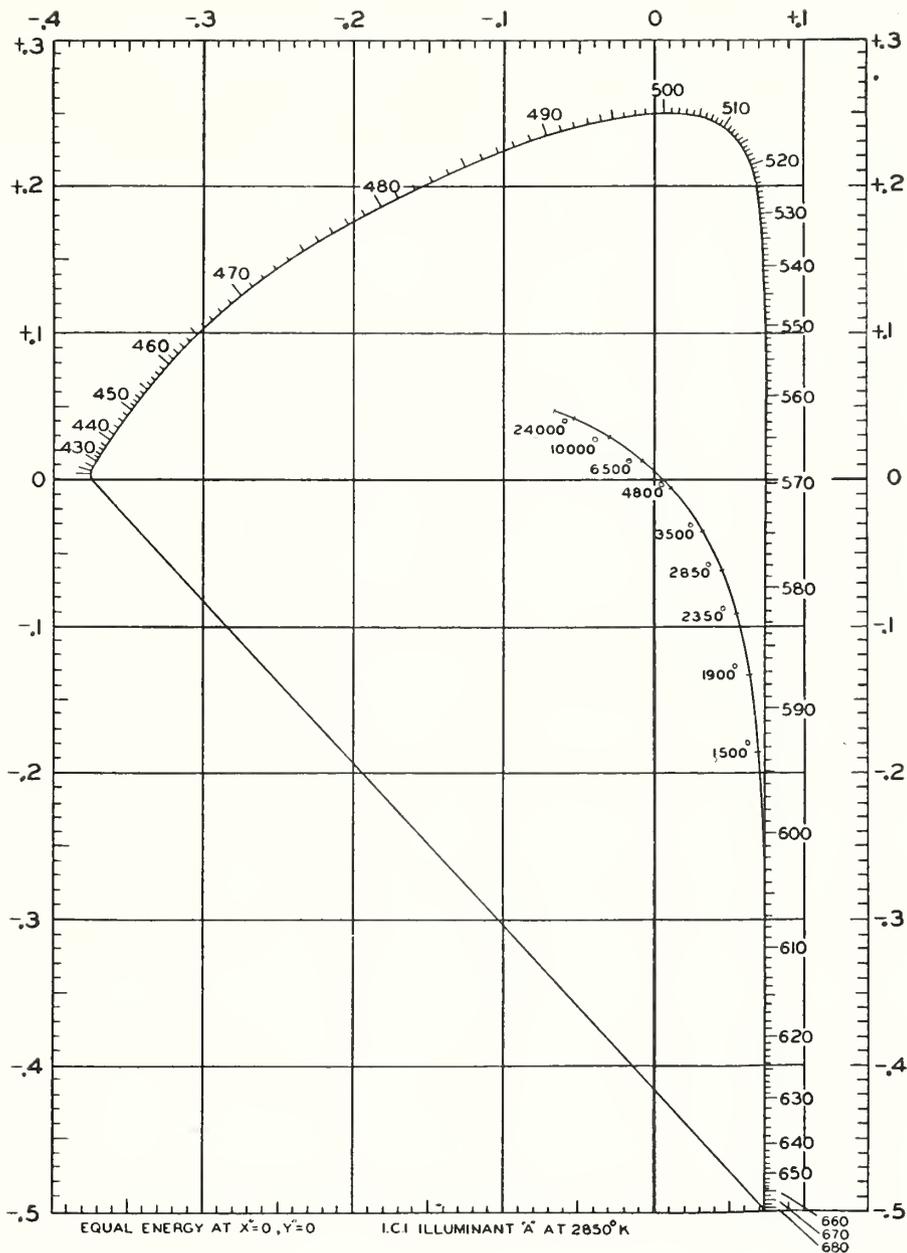


FIGURE 18. The rectangular uniform-chromaticness-scale (RUCS) diagram derived by Breckenridge and Schaub [17].

The chromaticity spacing is very similar to that of the UCS diagram (fig. 17).

$$\Delta E_t = k_1 \Delta(Y^{1/2}). \quad (19)$$

The size of the chromaticness component, ΔE_c , is approximately proportional to distance ΔS on a uniform-chromaticness-scale diagram, except that under usual observing conditions two very dark colors cannot be distinguished as easily as two light colors having the same chromaticity difference. The component is evaluated therefore as:

$$\Delta E_c = k_2 Y^{1/4} \Delta S, \quad (20)$$

where Y is an average of the luminous directional reflectances of the two surfaces, and ΔS is the distance on a uniform-chromaticness-scale diagram between the points representing the two chromaticities.

The constants k_1 and k_2 must conform to the relative importance of the lightness and chromaticness components and also serve to establish the unit of color difference. Preliminary studies have shown that the perceptibility of differences in luminous directional reflectance depends importantly on the conspicuousness of the line dividing the two fields, but the perceptibility of chromaticity differences is comparatively independent of this factor. The constant, k_1 , is therefore known as the "proximity factor". Table 10 gives preliminary values for the proximity factor, k_1 , adjusted so that color differences of less than one unit (known as the "NBS unit") are perceptually unimportant in most commercial transactions. The constant, k_2 , depends importantly on the angular subtense of the specimens to be compared. For ΔS evaluated as

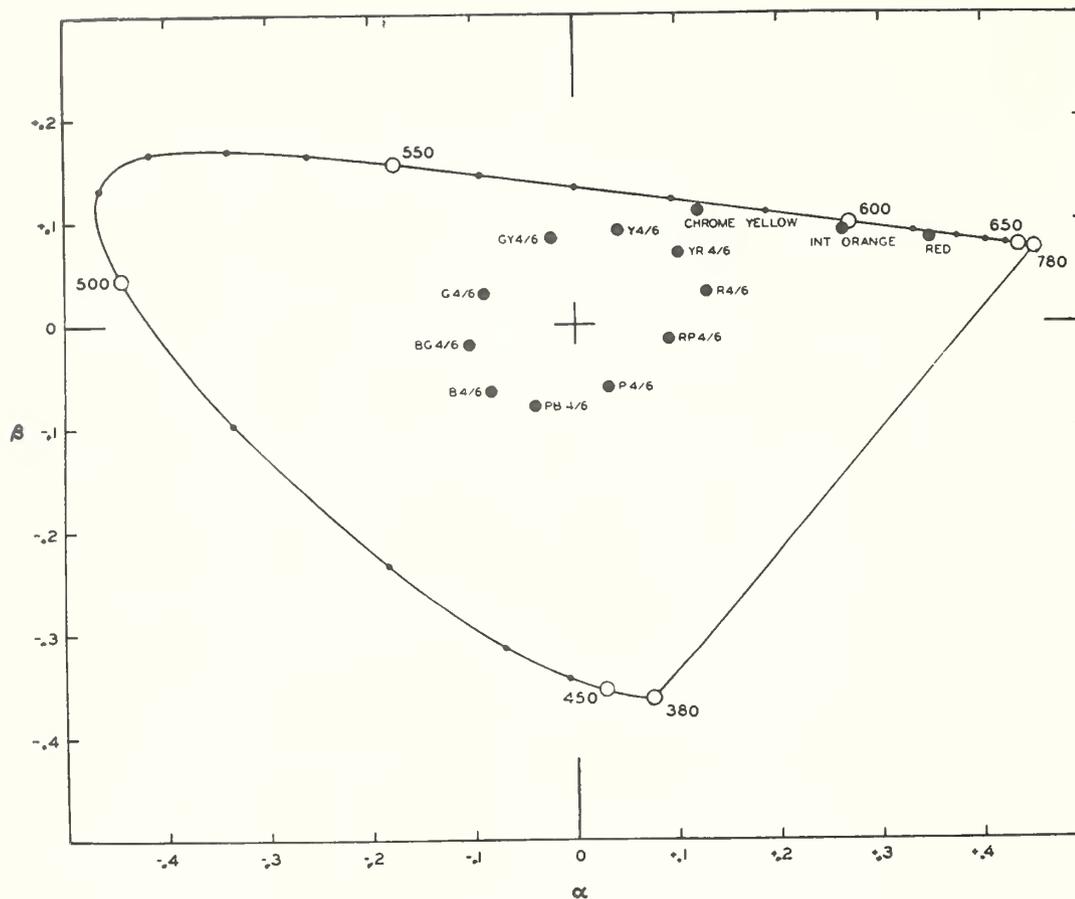


FIGURE 19. The alpha-beta diagram derived by Hunter [50].

The chromaticity spacing of this diagram is in somewhat better correspondence with perceptibility of chromaticness differences between extended surface colors than those of figures 17 and 18. The dots indicate ten members of the Munsell hue scale at value 4 and chroma 6.

$$\left[(\overline{\Delta\alpha}^2 + \overline{\Delta\beta}^2) \right]^{1/2},$$

and for test panels of the usual size (about 3 by 5 in.), this constant, k_2 , should be taken at about 700. The expression for color difference in NBS units may be written:

$$\Delta E = \left\{ \left[7 Y^{1/4} (\overline{\Delta\alpha}^2 + \overline{\Delta\beta}^2)^{1/2} \cdot 10^2 \right]^2 + \left[k_1 \Delta(Y^{1/2}) \right]^2 \right\}^{1/2}. \quad (21)$$

From formula 21 it may be seen that the NBS unit is considerably larger than the color difference just perceptible with certainty. By setting $\Delta E = 1$ for light surface colors ($Y^{1/4}$ approaching 1.00) of the same luminous reflectance ($\Delta Y^{1/2} = 0$), and assuming $\Delta\beta = 0$, we find: $\Delta\alpha = 0.0037$, which is about 10 times the least chromaticity difference perceptible with certainty under the most favorable conditions. By means of the chromaticity-difference colorimeter [61] and the multipurpose reflectometer [49], color differences can be evaluated in NBS units with an uncertainty of about 0.2 or 0.3 of a unit, even though based on single settings on the two specimens. It is customary practice, however, to make a repeat determination chiefly as a check on clerical errors.

TABLE 10. Values of the proximity factor, k_1 , for various observing conditions

Observing conditions	k_1
Specimens separated by a very narrow or nonexistent dividing line	120
Specimens separated by a contrasting, but narrow dividing line	90
Specimens separated by a broad patterned area contrasting with the areas being compared	40
Specimens evaluated for whiteness without visual reference to any standard	20
Convenient value for average condition, specimens separated by a narrow line	100

The NBS unit has been used in the inspection of working standards of color (such as the Kitchen and Bathroom Accessory Colors) for conformity to a master standard [93, 94], in the purchase specification and test of various types of paint, and in studies of the performance of accelerated weathering machines [26]. In general, this unit evaluated by way of the multipurpose reflectometer is in agreement with the results of visual judgment. Isolated disagreements have been traced (a) to failure of the source-filter-photocell combination to duplicate the standard observer (see fig. 11), (b) to failure of the "alpha-beta" diagram to yield perfectly uniform chromaticness scales, (c) to a tendency of some inspectors to pay preponderant attention to hue differ-

ences and neglect lightness and saturation differences, and (d) to the employment by inspectors of angular conditions of illumination and view different from the standard conditions (45°-normal) used in the measurements themselves.

The angular conditions most often used in inspection correspond approximately with the standard conditions (45°-normal) insofar as the light from the window is incident on the specimens, placed horizontal, at angles roughly centering on 45° and insofar as the direction of view is roughly normal. They differ importantly, however, insofar as most inspecting rooms supply a small quantity of diffused illumination in addition to the roughly directional component from the window. If the specimens to be compared are glossy, the color of each is composed of a mixture of the light diffusely reflected in accord with the measurement combined with light reflected specularly from the ceiling of the inspection room. It has been found particularly for dark specimens that better correlation is obtained with visual estimates of color differences made under these usual inspecting conditions if a correction is made for the specularly reflected light. A convenient approximate correction is obtained if the right-hand member of eq 21 is multiplied by a "gloss factor", f_g , computed as:

$$f_g = Y / (Y + 0.025), \quad (22)$$

where Y refers to luminous directional reflectance either of the standard or of the sample, whichever yields the larger value.

(b) Indices of Hue, Lightness, and Saturation

Inspection of manufactured materials and goods and the rejection of some of them as off-color are very frequently carried out on a basis somewhat different from the strict perceptibility of the color difference between specimen and standard. The inspector will say of an accepted specimen that it is off-color considerably more than another that he rejects, but that this kind of color difference is not objectionable. Departures of the color toward middle gray are usually viewed with comparative disfavor. Such colors are called muddy or dirty. Thus a slight departure from a near-white standard toward gray is more likely to result in a rejection than two or three times such a departure in the opposite direction. On the other hand, the lightest of a group of blacks is usually penalized as gray or "rusty" and the darkest thought of as a "good, clean black". If the standard color be highly chromatic, specimens perceived to have colors of slightly less saturation are rejected, but those even considerably more saturated may be accepted. Some kinds of goods (such as raw cotton) are judged almost entirely on the basis of lightness alone, with saturation and hue secondary. Others, such as dyes, whose price varies roughly with the reciprocal of the amount required to produce a standard color, are judged primarily on saturation. Still others, such as goods finished by application of a colorant (pigment or dye), are judged primarily on conformity to the hue of the

color standard. Lightness differences can be overlooked because they appear to the customer to be ascribable to local differences in the illumination. Saturation differences are not too serious, because if the customer looks steadily at the object he will see the saturation of the color decline anyway. Furthermore, rather large changes in lightness and saturation of one element in a design can be tolerated from the standpoint of color harmony, but hue changes are likely to be most injurious. A hue difference also gives the customer the impression that the wrong pigment or dye has been chosen, that some mistake has been made. For these reasons many inspectors are more critical of hue variations than they are of departures from standard lightness and saturation.

In an effort to provide the basis for numerical specification of color tolerances based upon these considerations, indices of hue, lightness, and saturation and their differences in NBS units have been developed [50]. The lightness index, L , is given by eq 23:

$$L = Y^{1/2}. \quad (23)$$

The hue index, H , is given by eq 24:

$$H = \tan^{-1} \beta / \alpha. \quad (24)$$

The saturation index, S , is given by eq 25:

$$S = [(\alpha^2 + \beta^2) Y^{1/2}]^{1/2}. \quad (25)$$

The simplicity of the hue and saturation indices in terms of the "alpha-beta" coordinates arises from the definition (eq 16) which places illuminant C and all blacks, whites, and grays illuminated by it at the origin of the coordinates.

The lightness component of a color difference, ΔE_1 , is given in NBS units by eq 19 and table 10. The saturation component is given in NBS units by eq 26:

$$\Delta E_s = 700 Y^{1/4} \Delta(\alpha^2 + \beta^2)^{1/2}. \quad (26)$$

The hue component of the color difference, ΔE_h , is given in NBS units by eq 27:

$$\Delta E_h = 12.2 Y^{1/4} (\alpha^2 + \beta^2)^{1/2} \Delta H, \quad (27)$$

where ΔH is found in degrees from eq 24. In eq 26 and 27, as in eq 20, the quantities that do not refer to differences are found as the average for the two specimens whose difference it is desired to evaluate.

An example of the application of eq 27 is afforded by purchases of bituminous emulsion paint by the War Department under U. S. Army Tentative Specification No. T-1213, which stated that some lightness and saturation difference could be tolerated on weathering but no hue differences. Paint was ruled not to comply with this specification, if, from a standard exposure test, it changed in hue by more than 1.0 NBS unit, or if the total color change (eq 21) exceeded 4.0 NBS units.

(c) Index of Whiteness

The whiteness of a specimen may be taken to be proportional to the degree to which it is perceived

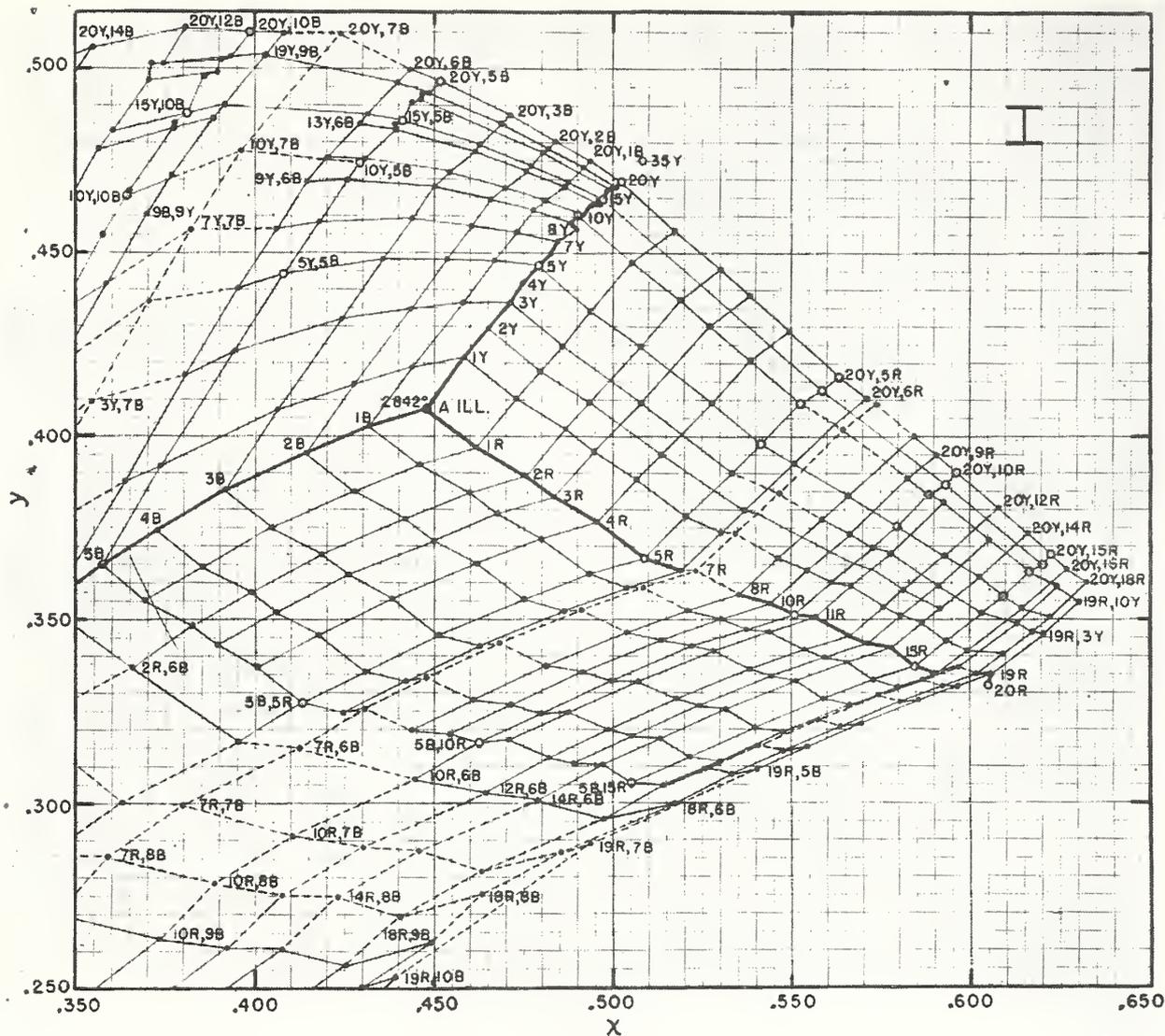


FIGURE 21. Chromaticities of two-part combinations of Lovibond red, yellow and blue glasses from the set (BS9940) kept as a reference at the National Bureau of Standards (Haupt and Douglas [46]).

Combinations judged to be significantly nonrepresentative of the Lovibond system are represented by points joined by dashed lines.

IV. Material Standards of Color

I. Transparent Media

Because of the convenience of material standards of color, they are often used in commerce in preference to specification according to the more fundamental ICI system. Material standards may be carried from place to place and, if the colors are sufficiently closely spaced in the neighborhood of the unknown color, the nearest match may be found by visual comparison. The color specification consists of identifying the particular member of the system yielding a match for the color to be specified. We deal here both with systems of material standards of such scope that a considerable fraction of the colors possible in nonself-luminous objects find in the system a fairly close representation, and with a few special small groups of material standards for particular purposes.

Color systems based upon transparent media take advantage of the fact that it is possible with a fixed illuminant to control the color of the transmitted light over a wide range by introducing varying amounts of three absorbing materials. This is done by permitting the light to pass through two or more elements of the absorbing medium instead of through a single element and is called subtractive combination, or mixture, because the action of each element is to subtract a certain fraction of each part of the spectrum of the incident light. The color specification consists of the number of unit elements required to produce the color match.

(a) Lovibond Glasses

The Lovibond color system consists of three sets

of colored glasses, red, yellow, and blue [72, 73, 74], the principal coloring materials being gold, silver, and cobalt, respectively. These coloring materials are confined to a thin layer of glass flashed onto an essentially clear glass support. The unit of the scale defined by each set is arbitrary, but the three units are related by being adjusted so that for observation by daylight subtractive combination of one unit of each of the red, the yellow, and the blue scales results in a filter perceived as neutral or achromatic. Each scale is exemplified by many glasses, each glass being marked with the number of unit glasses to which it is equivalent. If more than one glass of the same coloring material is used in combination, the number of unit glasses to which the combination is equivalent is found simply by adding the numerals marked on each individual glass of the combination. Although the original purpose of the Lovibond color system was to aid in the color control of beer, these glasses are widely used today for other products such as vegetable oils, lubricating oils, and paint vehicles.

A spectrophotometric analysis of the Lovibond color system was made by Gibson and Harris [33], and a scale of the red glasses used in combination with the 35-yellow glass has been constructed by Priest and Gibson [34, 45], having the same unit as the original Lovibond red scale but embodying a closer approach to the principle that the Lovibond numeral should indicate the number of unit glasses to which the single glass bearing the numeral is equivalent. Red glasses are calibrated in terms of the Priest-Gibson scale at the National Bureau of Standards and at the Electrical Testing Laboratories. The latter has also undertaken to adjust red glasses to slightly lower numerals, if desired, by reducing the thickness of the flashed layer slightly [27]. Tintometer, Ltd., makers of the Lovibond glasses, have computed from the published spectrophotometric measurements [33] the chromaticity coordinates, x, y , of all of the colors of the Lovibond system [123] produced by illuminating the glasses with ICI standard illuminant B. Computation of these colors for illuminants A and C has been carried out at the National Bureau of Standards [46]. Figure 20 shows a plot of these results for illuminant A, and figure 21 shows to a larger scale the most used section of this plot.

(b) Army Solutions

The Army solutions consist of groups of solutions whose concentrations are adjusted to produce the color match. The required concentrations are the specifications of the color. The most-used group is a triad consisting of half-normal aqueous solutions of cobalt chloride (red), ferric chloride (yellow), and copper sulfate (blue) with 1-percent hydrochloric acid. This group produces all colors except deep blue and deep red; it is supplemented by a triad of ammoniated aqueous solutions of cobalt, chromium, and copper salts, and by a pair consisting of aqueous solutions of potassium permanganate and

potassium dichromate [12, 13, 14]. Mellon and Martin [87] have reported the spectral transmittances for a number of solutions for colorimetric standards, including the Army solutions at 3 or 4 concentrations, each for the spectral range 440 to 700 $m\mu$. By extrapolation of these data it would be possible to find approximately the tristimulus values and chromaticity coordinates, x, y , on the standard coordinate system adopted in 1931 for these solutions just as was done by Mellon [86] for the coordinate system used in America before the international agreement. In this way it is possible to transform color specifications from the standard system into the required concentrations of the Army solutions, and the reverse. Kelly [67] has selected groups of Army-solution combinations adapted to color grading because of uniform spacing and variation of one color attribute (hue or saturation) at a time. The Army solutions are used in the 11th edition of the U. S. Pharmacopoeia as standards for the color of cod-liver oil and in carbonization tests with sulfuric acid for 28 organic compounds.

2. Opaque Media

If the set of material standards are pigmented or dyed surfaces, no automatically convenient notation, such as suggested by additive combination of lights or subtractive combination of absorbing elements, is available. Any systematic aspect of the color specification must be derived from the method of identifying the various members of the set of colored surfaces serving as standards.

(a) Color Dictionaries

From a color dictionary are obtained definitions of color names in terms of material standards. The primary aim is therefore to provide an array of named surface colors adequate for the purpose, and any arrangement or organization of the colors serves only the secondary purpose of assisting the user to find the one that most nearly matches.

The Maerz and Paul Dictionary of Color [81] is the foremost authority on color names. It contains about 7,000 different samples of color printed on semiglossy paper, and there are listed about 4,000 color names that are keyed to one or another of the color samples. These names are drawn from usage in many fields: paint, textile, ceramic, scientific, technical, and artistic. The samples are also identified by plate, column, and row, and because of their large number and fairly uniform color distribution it is usually possible to find among them a sample approaching what is called a "commercial color match" for any given uniform opaque surface. On this account the Maerz and Paul Dictionary finds a considerable application as a collection of color standards quite separate from its primary function of defining color names. There are noticeable color differences between corresponding samples in different copies of the dictionary, but the differences have been held to a satisfactorily small amount by exceptional care in the printing process.

The accepted authority for color names in the textile and allied industries is the Textile Color Card Association of the United States. The TCCA has issued nine editions of a standard color card since 1915, the current edition [129] containing 216 color samples of pure-dye silk; furthermore, the TCCA issues to its members several seasonal cards each year. All colors of these standard and seasonal cards are identified by name and cable number. The standard colors have been measured at the National Bureau of Standards by spectrophotometric and colorimetric procedures; and luminous reflectance, Y , relative to magnesium oxide, and chromaticity coordinates, x, y , for illuminant C have been published [118].

A color dictionary much used for the specification of the colors of flowers, insects, and birds was prepared in 1912 by Ridgway [120]. This outstanding pioneer work contains about 1,000 named color samples of paper painted by hand. Each chart shows columns of colors of the same dominant wavelength progressing from each chromatic color at the middle of the column toward white at the top, and toward black at the bottom; and there are five series of such columns, each one encompassing the entire hue circuit, but at different purities. Many of the names were coined at the time of publication to fill in gaps in popular color nomenclature, and so have not much descriptive value. Each sample is arbitrarily identified by column, row, and series, however; and there is an alphabetical list of the color names giving this identification.

(b) Ostwald Color System

The notation of the Ostwald system is based on the properties of idealized pigment surfaces having spectral reflectance constant at a certain value between two complementary wavelengths and reflectance constant at a certain other value at other parts of the spectrum [31]. The *full colors* are those that have the low value of spectral reflectance equal to zero and the high one equal to 100 percent. The difference between these two reflectances for other idealized pigment surfaces is the *fullcolor content*, the value of the low reflectance is the *white content*, and the difference between the high reflectance and 100 percent is the *black content*.

The complete Ostwald notation consists of a number from 1 to 24 and two letters from a to p . The number indicates dominant (or complementary) wavelength on an arbitrary but approximately uniform perceptual scale, and is called *Ostwald hue number*. The first letter indicates white content, a being a white content of 89.13 percent, which is as near to 100 percent as is practicable for usual pigment-vehicle combinations, and other letters in alphabetical sequence indicating decreasing white content on a logarithmic scale. The second letter indicates black content, p being a black content of 10.87 percent, which is as near to zero as is prac-

ticable, and other letters in alphabetical sequence indicating increasing black content on a logarithmic scale. The logarithmic scales were thought by Ostwald to insure uniform color scales, but this is true only to a rough approximation. Since the percentage of white (w) content, black (b) content and full-color (c) content must necessarily add up to 100, no explicit indication of the latter is required. The notation aa thus indicates the practical white of the system ($w=89.13$, $b=10.87$, $c=0.00$). This notation, aa , however, is usually written simply as a ; and notations for grays and black are likewise abbreviated to a single letter. The Ostwald constant-hue triangle has the pigment approximation to white (aa) at the top, the pigment approximation to black (pp) at the bottom, and at the other angle of the equilateral triangle there is the pigment representation of the fullcolor (pa) of that particular Ostwald hue. The colors arranged in slant lines parallel to the top of the triangle have constant black content and are called *isotones*. The colors arranged in slant lines parallel to the bottom of the triangle have constant white content and are called *isotints*. The colors arranged in vertical lines parallel to the black-white series have constant fullcolor content and are called *isochromes*. The isochromes are colors of constant dominant wavelength and purity. Thus the isochromes vary in luminous reflectance but are of constant chromaticity; on this account they are said to form *shadow series*.

The Ostwald ideas have been a considerable aid in thinking about color relationships on the part of those who duplicate colors by mixtures of chromatic pigments with white and black pigments, and they have served as a guide in the selection of combinations of such colors to produce pleasing effects. However, the use of these idealized pigment surfaces as a basis for a system of colorimetry has been hampered by the fact that actual pigment surfaces approximate them rather poorly, and by the fact that not all actual pigment surfaces can be color matched by one of these ideal surfaces. Still, color charts made up more or less in accord with the Ostwald principles have been widely used for color standards and for the selection of harmonizing colors [54, 109, 110, 128]. Of these the Jacobson Color Harmony Manual [54] is preeminent not only because of its technical excellence, but also because Foss [31] has given a clear statement of which of the somewhat contradictory Ostwald principles were followed in its construction, and Granville and Jacobson [38] have made a spectrophotometric study of the color chips, and have published luminous reflectance, Y , and chromaticity coordinates, x, y , for every chip. These chips are therefore valuable for use in colorimetry by difference from a working standard, and the fact that the chip is a member of an orderly arrangement of colors facilitates the selection of a working standard for any particular purpose. The master standards of this collection measured by Granville and Jacobson have been deposited at the National Bureau of Standards.

(c) Munsell Color System

The basis of the Munsell system is description of colors perceived to belong to surfaces in terms of hue, lightness, and saturation. Each such tridimensional description can be represented by a point plotted in a space diagram known as the *surface-color solid*; see figure 22. In the surface-color solid,

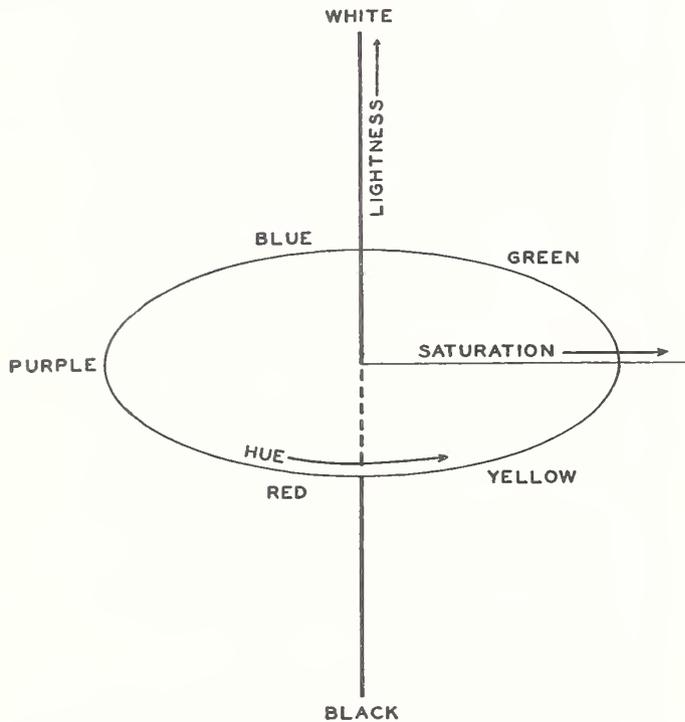


FIGURE 22. Dimensions of the psychological surface-color solid.

In this solid each point represents the color perception of a surface. White is at the top, black at the bottom, with grays intermediate forming the black-white axis. Variations parallel to this axis are lightness variations. Hue is represented by angle about this axis. Saturation is represented by perpendicular distance from the axis.

the central axis represents the grays extending from black at the bottom to white at the top. *Lightness* of a chromatic (nongray) color determines the gray to which it is equivalent on this scale. Lightness is represented in the color solid by distance above the base plane. *Hue* determines whether a color is per-

ceived as red, yellow, green, blue, purple, or some intermediate; it is represented in the color solid by angle about the central axis. *Saturation* indicates the degree of departure of a surface-color perception from the gray of the same lightness; it is represented by perpendicular distance from the central gray axis.

The Munsell color system specifies a surface color by giving for usual viewing conditions its position on more or less arbitrary hue, lightness, and saturation scales having perceptually nearly uniform steps [92]. The Munsell term corresponding to lightness is Munsell value; that for saturation is Munsell chroma, and that for hue is Munsell hue.

Munsell value is zero for the ideal black surface having luminous reflectance equal to zero, and it is ten for the ideal white diffusing surface having luminous reflectance equal to one. Munsell chroma is expressed in arbitrary units intended to be perceptually of the same size regardless of value and hue. The strongest known nonfluorescent pigment colors have chromas of about 16; neutral grays have zero chroma, as do black and white. Munsell hue is expressed on a scale intended to divide the hue circuit (red, yellow, green, blue, purple, back to red) into 100 perceptually equal steps. According to one convention, the 100 Munsell hues are identified simply by a number from 1 to 100, and on this scale hues that differ by 50 are nearly complementary; that is, combine by rotary mixture to yield near gray. The most common convention, however, is to divide these 100 hues into 10 groups of 10 hues each, and to identify each group by initials indicating the central member of the group, thus: red R, yellow red YR, yellow Y, green yellow GY, green G, blue green BG, blue B, purple blue PB, purple P, and red purple RP. The hues in each group are identified by the numbers 1 to 10. Thus, the most purplish of the red hues is designated as 1R, the most yellowish as 10R, and the central hue as 5R, or often simply as R; see figure 23. The transition points (10R, 10YR, 10Y, and so on) between groups of hues are also sometimes designated by means of the initials of the two adjacent hue groups, thus R-YR=10R, YR-Y=10YR, Y-GY=10Y, and so on (see figures 23 and 24).

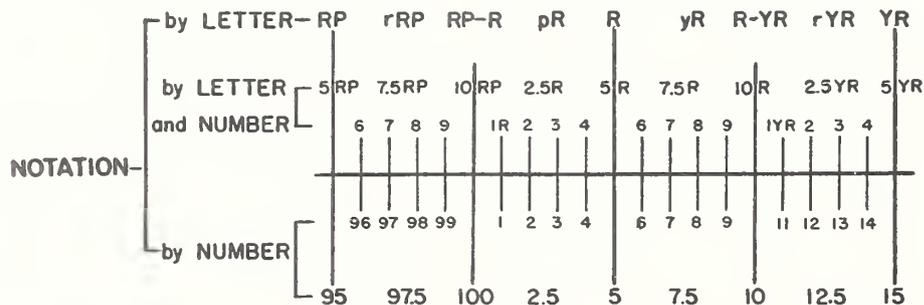


FIGURE 23. Hue notation in the Munsell system.

The relation between the letter, the number, and the combination letter and number designations of Munsell hue are shown here for a portion of the hue circuit. By means of the combination letter-number method, more widely used than the other designations, the third intermediate set of hues are designated: 7.5RP, 2.5R, 7.5R, 2.5YR, and so on.

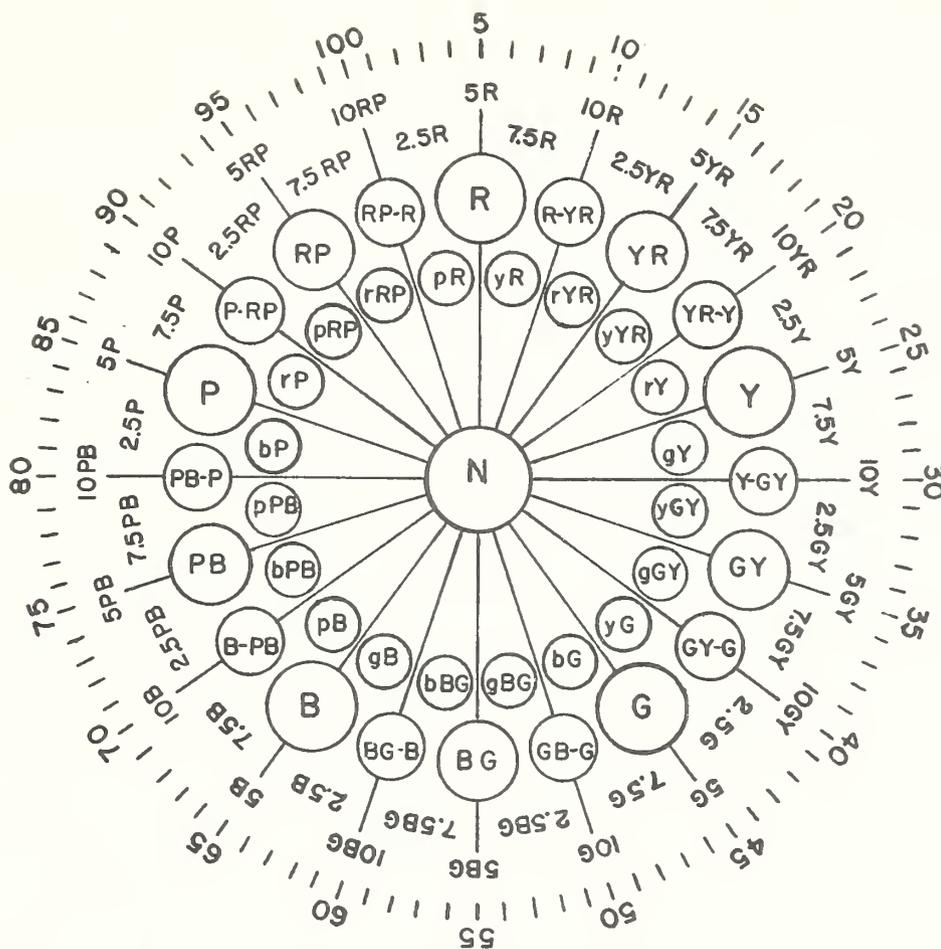


FIGURE 24. Designations of Munsell hue.

The Munsell hue scale is based upon five principal hue names: red, yellow, green, blue, and purple, and it makes use also of the intermediates: yellow-red, green-yellow, blue-green, purple-blue, and red-purple. A second set of intermediates enlarges the hue scale to 20; and a third to 40. The system of letter abbreviations for these 40 hues, shown here, is cumbersome and confusing and has largely been supplanted by a combination number and letter designation (5Y, 10Y, 5GY, 10GY, and so on). A purely numerical system has also been proposed (outer circle) but is little used.

The Munsell notation is commonly written: Hue Value/Chroma, that is, the hue notation, such as 6R, then the value, such as 7, and finally the chroma, such as 4, the latter two being separated by a slurring mark: 6R 7/4. More precise designations are given in tenths of the arbitrary steps of the scales, thus: 6.2R 7.3/4.4. The grays are indicated by the symbol N for neutral followed by the value notation, thus: N 7/ or N 7.3/; the chroma, being zero for neutrals, is not specifically noted.

There have been two representations of the Munsell system published, the original Atlas in 1915 [90], now chiefly a collector's item, and the Munsell Book of Color [91]. This book consists of rectangles of hand-painted paper mounted on charts in a loose-leaf binding. The neutrals form a one-dimensional color scale extending from N 1/ to N 9/. Each chromatic sample, of which there are about 1,000, takes its place on three color scales: a hue scale, a value scale, and a chroma scale; and the spacing of these scales is intended to be perceptually uniform. The pocket edition, adapted for determining Munsell notation of unknown colors by visual comparison, consists of 40 constant-hue charts, so called because all of the samples on each chart have the same Munsell hue,

and are chosen with the intention of yielding under usual observing conditions colors all perceived to have the same hue. These samples are arranged in rows and columns, the rows being chroma scales at constant Munsell value, the columns being value scales at constant Munsell chroma. Comparison of an unknown color with these two families of scales gives by interpolation the Munsell value and Munsell chroma of the unknown. Interpolation between adjacent constant-hue charts gives the Munsell hue. Table 11 gives the Munsell book notations of the Kitchen Green (SKC-15) and the Orchid (SBC-20) master standards found in this way. Unknowns not too far outside the range of the Munsell charts may be evaluated with some reliability by extrapolation along the value and chroma scales.

To facilitate the comparison of the unknown color with those of the paper rectangles on the Munsell charts, either the unknown color must be brought into juxtaposition with the rectangle and held in nearly the same plane, or, if the form of the unknown prevents such juxtaposition, two masks of thin cardboard having rectangular openings to fit the paper rectangles should be used. One mask should be placed over the unknown color; the other over one

or another of the Munsell colors in succession to obtain the interpolated Munsell book notation. Kelly [64, 65] made effective use of a form of mask with three rectangular openings particularly adapted to comparisons involving powdered chemicals and drugs viewed through a cover glass. It is advantageous to have the color of the mask fairly close to that of the unknown, particularly in Munsell value; that is, if the unknown color is dark, the mask should be of a dark color also. Use of a light mask for a dark color prevents the observer from making as precise a visual estimate as he can make with a mask more nearly a color match for the unknown.

Because of the visual uniformity of the scales, the estimates of Munsell notation for unknown colors within the color range of the charts have a reliability corresponding to the use of a much larger collection of unequally spaced color standards. On this account the pocket edition of the Munsell Book of Color is widely used as a practical color standard for general purposes.

The standard, or library, edition shows the same colors as the pocket edition, but it shows them on constant-value charts in a polar coordinate system, and on constant chroma charts in a rectangular coordinate system, as well as on constant-hue charts. This edition has full explanatory matter in the text and is adapted particularly for teaching color. It is too bulky for convenient practical use in determining the Munsell notation of an unknown color.

The samples of the 1929 Munsell Book of Color have been measured by means of the spectrophotometer twice independently with generally concordant results [36, 68]. In both of these studies luminous reflectance, Y , relative to magnesium oxide and chromaticity coordinates, x, y , of the roughly 400 samples were computed for illuminant C. Glenn and Killian [36] have also published the dominant wavelength and purity for each of the colors; and Kelly, Gibson, and Nickerson [68] have published specifications ($X, Y, Z; x, y, z$) for three additional illuminants (illuminant A, Macbeth daylight [108], and limit blue sky). Furthermore, they have published a series of (x, y)-chromaticity diagrams showing the position of the Munsell colors for each of the Munsell values from 2/ through 8/. Figure 25 is their diagram for Munsell value 6/. From these diagrams, it is possible to find the Munsell book notation corresponding to any given values of luminous reflectance and chromaticity coordinates (Y, x, y). Table 11 shows such values for the Kitchen Green and Orchid master standards. The samples of the 1942 supplement to the Munsell Book of Color together with many special standards put out by the Munsell Color Company, have been measured spectrophotometrically by Granville, Nickerson, and Foss [37]. These Munsell standards, together with those of the 1929 Munsell Book of Color, number over 1,000, and comprise the largest systematic set of color standards of known luminous reflectance and chromaticity coordinates ever made. These standards are commercially available separately in disk form and on large sheets, and they make practical the general colorim-

etry of opaque specimens not only by disk mixture (section II, 2 (a)) but also by difference from a standard (section III).

Master standards of the Munsell Book of Color are on file at the National Bureau of Standards, and as repaintings of the various colors are issued this file is kept up to date.

TABLE 11. *Munsell book notations of the Kitchen Green and Orchid master standards determined, (1) by visual interpolation along the color scales of the Munsell Book of Color, and (2) by interpolation from the chromaticity coordinates (x, y) and luminous reflectance (Y) given in Table 5*

The Munsell renotations found from Y, x, y , are also shown

Color standard	Munsell book notation		Munsell renotation
	From visual interpolation	From chromaticity coordinates and luminous reflectance of table 5	
Kitchen Green	9GY 6.2/3.8	9.2GY 6.2/4.0	9.6GY 6.3/4.4
Orchid	2RP 6.1/3.6	2.1RP 6.0/3.6	1.3RP 6.2/4.2

From reverse application of the charts, luminous reflectance and chromaticity coordinates of an unknown color may also be found quickly and with an accuracy sufficient for many purposes by obtaining first the Munsell book notation of the unknown by visual interpolation and then transforming it by reference to (x, y)-interpolation charts (like figure 25) based on the complete set of standards.

The Munsell color standards may also be used, though less conveniently, in the colorimetry of light-transmitting elements (gelatine films, crystal and glass plates, solutions, and so on); and, conversely, such elements may be given Munsell book notations from their ICI specifications by means of interpolation charts like figure 25. In the application of these charts to light-transmitting specimens, Munsell value is found from luminous transmittance in a way analogous to its usual dependence on luminous reflectance. Table 18, to be discussed presently in another connection, shows Munsell book notations so derived from the luminous transmittances, T , and chromaticity coordinates, x, y , of the glass standards of the ASTM Union colorimeter. The last four book notations given are relatively uncertain, because the colors to be specified are far outside the range of the Munsell standards.

The spacing of the Munsell colors has been examined in detail by a subcommittee of the Colorimetry Committee of the Optical Society of America [96, 97]. This Committee work confirmed the many local irregularities in spacing revealed by the spectrophotometric studies (see figure 25) and established the need for some more general but minor adjustments to make the colors of the Munsell charts correlate more perfectly under ordinary observing conditions (adaptation to daylight, gray to white surrounding field, and so on) with the surface-color solid. The subcommittee found it possible from this study to recommend specifications (Y, x, y) on the

TABLE 12. Luminous reflectance (Y_v) corresponding to various Munsell renotation values (V)

V	Y_v	V	Y_v	V	Y_v	V	Y_v	V	Y_v
	Percent		Percent		Percent		Percent		Percent
0.0	0.000	2.0	3.126	4.0	12.00	6.0	30.05	8.0	59.10
.1	.120	2.1	3.391	4.1	12.66	6.1	31.23	8.1	60.88
.2	.237	2.2	3.671	4.2	13.35	6.2	32.43	8.2	62.71
.3	.352	2.3	3.968	4.3	14.07	6.3	33.66	8.3	64.57
.4	.467	2.4	4.282	4.4	14.81	6.4	34.92	8.4	66.46
.5	.581	2.5	4.614	4.5	15.57	6.5	36.20	8.5	68.40
.6	.699	2.6	4.964	4.6	16.37	6.6	37.52	8.6	70.37
.7	.819	2.7	5.332	4.7	17.18	6.7	38.86	8.7	72.38
.8	.943	2.8	5.720	4.8	18.02	6.8	40.23	8.8	74.44
.9	1.074	2.9	6.128	4.9	18.88	6.9	41.63	8.9	76.53
1.0	1.210	3.0	6.555	5.0	19.77	7.0	43.06	9.0	78.66
1.1	1.354	3.1	7.002	5.1	20.68	7.1	44.52	9.1	80.84
1.2	1.506	3.2	7.471	5.2	21.62	7.2	46.02	9.2	83.07
1.3	1.667	3.3	7.960	5.3	22.58	7.3	47.54	9.3	85.33
1.4	1.838	3.4	8.471	5.4	23.57	7.4	49.09	9.4	87.65
1.5	2.021	3.5	9.003	5.5	24.58	7.5	50.68	9.5	90.01
1.6	2.216	3.6	9.557	5.6	25.62	7.6	52.30	9.6	92.42
1.7	2.422	3.7	10.134	5.7	26.69	7.7	53.94	9.7	94.88
1.8	2.642	3.8	10.734	5.8	27.78	7.8	55.63	9.8	97.39
1.9	2.877	3.9	11.356	5.9	28.90	7.9	57.35	9.9	99.95
								10.0	102.56

luminous transmittance relative to the same thickness of distilled water or solvent. For any of these objects, Munsell renotation value, V , may be found from Y in accord with table 12. Because of their close correlation with the color solid, Munsell renotations are capable of being quickly understood. Thus the renotation 1.3 RP 6.2/4.2 for Orchid indicates from the letters *RP* that the specimen is a red-purple, from the value 6.2 that it is somewhat lighter than middle gray, and from the chroma 4.2 that it is a moderately saturated color inclining somewhat toward gray. Munsell renotation hue and chroma serve more adequately for object colors the purposes formerly served by dominant wavelength and purity. Munsell renotation hue correlates significantly better under ordinary conditions of daylight observation with the hue of the perceived color than does dominant wavelength; and Munsell renotation chroma is by far superior to purity in its correlation to saturation of the perceived color.

This correlation with the color-perception solid does not, however, necessarily hold under all observing conditions. Ordinarily this Orchid standard will be perceived to have a moderate to weak reddish

purple color, but it is not so perceived under all conditions. If this specimen be viewed next to a brilliant magenta color, such as may be provided by a fluorescent fabric, it will be perceived to take on a color of different hue and much reduced saturation, such as might be described as a bluish gray. Thus the lightness, hue, and saturation of the color perception depend upon the surroundings and upon the adaptive state of the eye; and lightness, hue, and saturation are taken correctly to be psychological terms. But the Munsell renotation refers only to the light that is reflected from the specimen, and stays constant regardless of these changes in observing conditions. It is therefore a psychophysical characterization of the specimen according to the light reflected from it just as are luminous reflectance, Y , and chromaticity coordinates, x, y , from which it can be derived, and dominant wavelength and purity, which it is coming to supplant.

Another advantage of expressing spectrophotometric results in the form of the Munsell renotation is that the amount and kind of the color difference between two specimens can be found immediately from the two renotations in an easily understandable form. One NBS unit of hue difference from eq 27 is equivalent to approximately 0.25 Munsell hue step at chroma 10, or to 2.5 Munsell hue steps at chroma 1. One NBS unit of saturation difference from eq 26 is equivalent to about 0.15 Munsell chroma step; and one NBS unit of lightness difference from eq 19 corresponds to about 0.10 Munsell value step. If the two colors differ in more than one dimension, the number of NBS units may be estimated by taking the sum of the NBS units in each dimension, thus:

$$\Delta E = (C/5)(2\Delta H) + 10\Delta V + 6\Delta C. \quad (29)$$

Or if, as often happens, hue differences are of greater concern than lightness and saturation differences, the Nickerson [101, 103] index of color difference, I , may be used

$$I = (C/5)(2\Delta H) + 6\Delta V + 3\Delta C, \quad (30)$$

where C is Munsell chroma, and ΔH , ΔV , ΔC , are the differences between the two colors in Munsell hue, value, and chroma respectively.

V. One-Dimensional Color Scales

There are many tests analogous to the comparison of a solution of an unknown amount of a constituent with a series of suitably prepared standard solutions to find the concentration of the specimen. In these tests the colors of the unknowns exhibit a one-dimensional change with concentration, and although this change may be complicated in terms of luminous transmittance and chromaticity coordinates [86], a suitably spaced series of standards over this range of colors will yield the desired concentration either by actual match with one of the standards, or by visual interpolation among them. Such a series of standards is said to constitute a *color scale*. The ideal material from which to make the standards is the unknown constituent itself; in this way there is guaranteed not only a perfect color match at some

point along the scale, but also a perfectly nonmetameric match so that variations of the illuminant or individual-observer variations are generally unimportant.

However, if the unknown is impermanent, it may become necessary to try to duplicate the desired colors in a more permanent medium. Glass is a frequent choice because of its generally superior permanence. Some degree of metamerism has then to be tolerated; because the standards have coloring constituents not a perfect spectral match for the unknown. It is also rare that a perfect job of color matching for any standard illuminant and observer is done. The observer is then faced with what is often a difficult, and sometimes an impossible, task.

He must estimate the position of the unknown color on the scale, and oftentimes it will seem to him that the unknown color is not equal to any of the standard colors nor intermediate between any two of them. The concepts in terms of which the observer perceives these color differences then come into play. If he judges the color difference between the two luminous areas presented to him in terms of hue, brightness, and saturation, as is fairly common, he could estimate the position of the unknown on the color scale as the point on the scale yielding the same hue, or as that yielding the same brightness, or the same saturation; or he could try to estimate the point on the scale yielding the closest color match, or he could disregard brightness differences and try to estimate the point on the scale yielding the closest chromaticity match. The determination becomes an estimate based on what criterion of equivalence is used by the observer, and it depends upon his mental furniture in an essentially indescribable way. In spite of these drawbacks, a good color scale is a useful timesaver, as long as it is not used in attempts to provide a one-dimensional solution to what is essentially a multi-dimensional problem.

Judgments of position on the color scale according to equality of brightness can be expected to correspond to luminous transmittance. Judgments according to equality of hue agree well with Munsell renotation hue for ordinary conditions of observation; see curved lines of figure 3 separating the areas corresponding to the various hue names, also figure 26. Judgments according to equality of saturation agree well with Munsell renotation chroma; see figure 26. If the brightness variation along the scale is slight compared to the chromaticity variation, judgments are likely to be according to nearest chromaticity match. Such judgments agree well with the point on the scale found by taking the shortest distance on a uniform-chromaticity-scale diagram between the point representing the unknown color and the line representing the scale; see figures 17, 18, and 19. If there is primary variation of both luminance and chromaticity, no reliable way of estimating the nearest color match has yet been developed. According to the OSA Colorimetry Committee [108] "the complete experimental clarification of this problem is one of the major programs yet to be undertaken in the field of colorimetric research".

1. Color Temperature

Perhaps the most widely used one-dimensional color scale is that of color temperature for classifying light sources. The *color temperature* of a light source is the temperature at which the walls of a furnace must be maintained so that light from a small hole in it shall yield the chromaticity of the source to be specified. The color scale thus consists of the series of lights producible by closed-cavity radiation and is specified by temperature on the absolute scale (degrees Kelvin). Working standards of color temperature may consist of an incandescent

lamp operating at a fixed voltage combined with a series of amber or blue filters, like the Davis-Gibson [22] liquid filters or the Lovibond blue glasses; but by far the most common way of producing these chromaticities over moderate ranges of color temperature is by variation of the voltage applied to an incandescent lamp. The locus of these chromaticities (the so-called Planckian locus) is shown on figures 3a, 5, 7, 17, and 18. If the chromaticity of the light source is close to, but not exactly equal to, any of the Planckian chromaticities, still it is possible to correlate a color temperature with the source by taking the nearest chromaticity match. Figure 27 shows this correlation [60]. The isotherm lines, which cut the Planckian locus at varying angles, are all such as to be perpendicular to this curve on figure 17. Since color temperature specifies only the chromaticity of a light, there are many spectral compositions corresponding to the same color temperature. Color temperature of an illuminant is therefore an incomplete and unreliable indication of the rendering of the colors of objects illuminated by it, or of the photographic effect of the illuminant. To make color temperature a perfectly valid basis for comparing two lights, it must also be shown that they are spectrally similar; thus, incandescent lamps may be usefully compared by means of color temperature, and fluorescent lamps with about the same admixture of mercury spectrum may also be so intercompared, but comparison of incandescent lamps with fluorescent, by color temperature, is often not valid.

2. Lovibond Grading of Vegetable Oils

The Lovibond glasses are used extensively in the grading of vegetable oils in this country. McNicholas [85] has shown from the spectral transmittance of representative samples of refined cottonseed oils, also oil of sesame, peanut, soybean, corn, rape, and olives, that these oils owe their colors to independently varying amounts of two groups of coloring matters commonly found in plants, a group of brown pigments, and a group of green (chlorophyll compounds). For commercial purposes the colors of these oils are mostly graded by the number of Lovibond red units required in combination with Lovibond 35-yellow to color match a 5/4-in. layer of the oil. Figure 28 shows the spectral transmittance of a group of cottonseed oils (solid curves) and one oil of other plant origin (dotted curve), all of which have color grades between 11.0 and 13.0 Lovibond red units on the 35-yellow plus red scale. Figure 28 also shows the spectral transmittances of these limiting Lovibond glass combinations (solid curves with solid circles). It will be noted first that the oils all make metameric pairs with the Lovibond glass combinations. A standard light source is thus required, and a certain amount of individual-observer difference may be expected in grading oils by this method. Since there are two main groups of pigments in the oils, it is also to be expected that no one-dimensional grading system, like the scale of 35-yellow plus some

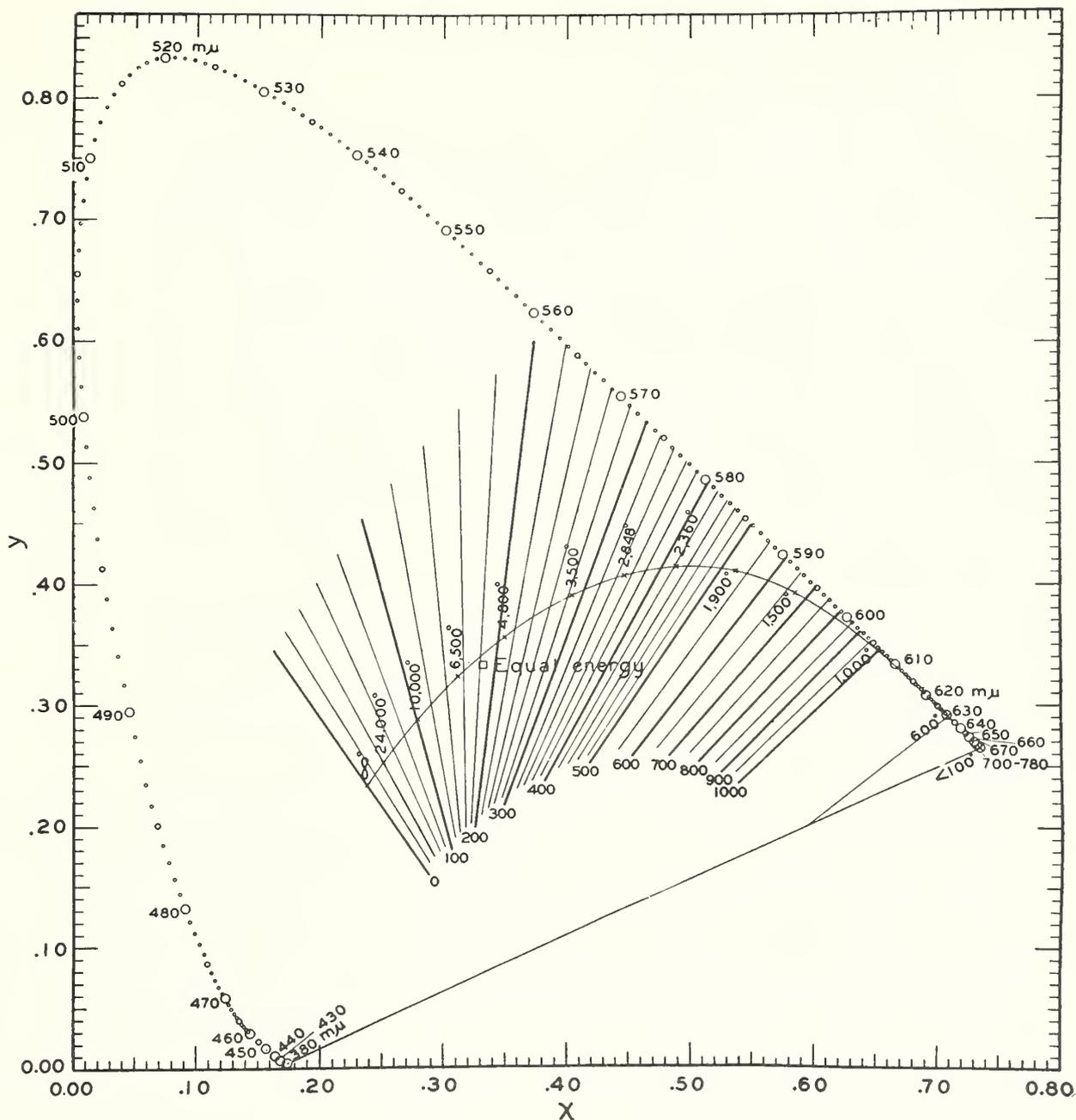


FIGURE 27. Correlated color temperature for chromaticities represented near the Planckian locus on the (x,y) -chromaticity diagram.

The straight lines intersecting the Planckian locus are identified by the reciprocal color temperature in microreciprocal-degrees (μrd) of their intersection with the Planckian locus. Points on the locus, itself, are identified by color temperature in degrees Kelvin. The straight lines, if transformed to the UCS triangle (fig. 17), would be orthogonal to the Planckian locus.

amount of red, will yield color matches. Computation of the tristimulus values from the spectral-transmittance curves and plotting on the (r,g) -Maxwell triangle (see fig. 29 due to McNicholas [85]) reveals this spreading of the chromaticity points over an area instead of along a single line, and it shows further that the Lovibond 35Y plus R locus is too far from the spectrum locus to be a good match for the center of this area except for Lovibond red less than 3 units. There is some doubt, therefore, what Lovibond grade corresponds to a given spectral-transmittance curve of figure 28. McNicholas chose to base the decision on nearest chromaticity match, and made use of the uniform-chromaticity-scale dia-

gram shown in figure 17. The short straight lines on figure 29 intersecting Lovibond 35Y plus R locus at somewhat oblique angles would be perpendicular to the corresponding locus on figure 17.

In spite of the difficulties of determining the Lovibond grade of a vegetable oil, the method is well suited to indicate quality and salability, and it has been used for more than 30 years. Because of the difficulties, however, and the further disadvantage of being dependent upon a foreign source of glass standards, there has been fairly continuous agitation and research work intended to lead to another, perhaps photoelectric, method. But the Lovibond color scale, 35Y plus R, is so ingrained in the purchase

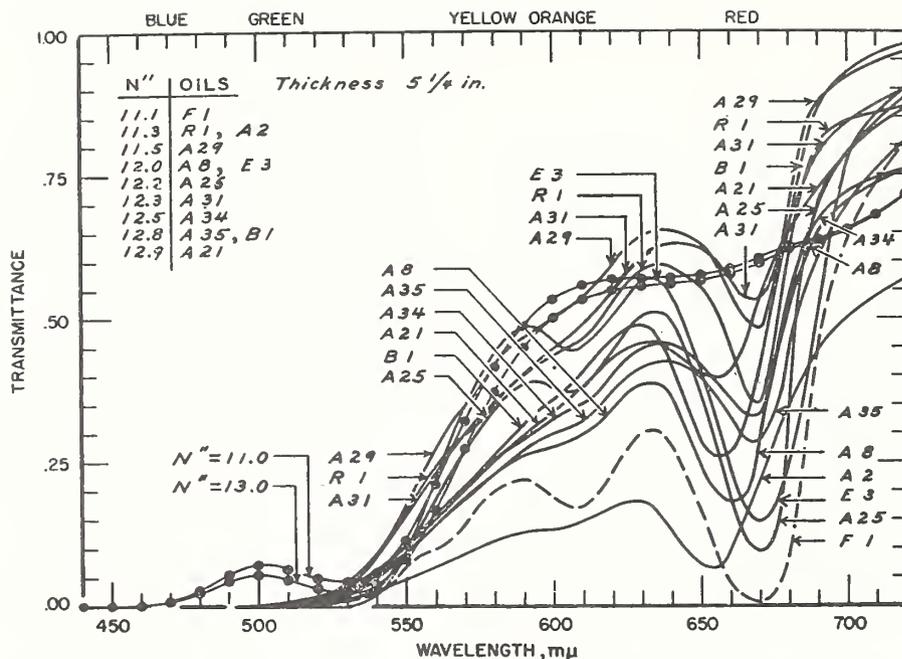


FIGURE 28. Spectral transmittance of a group of vegetable oils (solid and dashed curves) compared to the spectral transmittance (solid circles) of combinations of Lovibond yellow and red glasses (35Y 11R and 35Y 13R) to which they are closely equivalent in trade practice (McNicholas [85]).

It will be noted that each oil combined with each glass combination makes a rather strongly metameric pair; that is, the brown and green pigments in the oils are spectrally quite dissimilar to the colorants (silver, yellow, gold ruby) of the glasses.

and sale of vegetable oil in this country that it may well be that a substitute method will still have to give results expressed on that scale.

3. Union Colorimeter Grading of Lubricating Oils

For more than 20 years the color of lubricating oils and petrolatum has been graded by comparison with the colors of 12 glass standards [10]. The petroleum product in a 33-mm layer and the standard are illuminated by artificial daylight produced by combining an incandescent lamp of color temperature approximately 2,750°K with a filter of Corning Daylight glass specially selected to have spectral transmittances within specified tolerances and further such as to have, for standard illuminant A, luminous transmittance, T_w , and chromaticity coordinates x, y, z , within the limits:

T_w	0.107 to 0.160
x314 to 0.330
y337 to 0.341
z329 to 0.349

The specimen holder, the magazine containing the glass color standards, the artificial daylight assembly, and a viewing diaphragm defining the direction of view are mounted together to form a portable instrument known as the Union colorimeter.

Table 13 gives the Lovibond analysis of the glass color standards [10], the luminous transmittance, T_w , and chromaticity coordinates, x, y , for illuminant

C [124], and the color names used by the National Petroleum Association. Figure 30 shows by large circles on the (x, y)-diagram the chromaticities of these glass color standards compared to those of 22 petroleum products (small circles) measured by Diller, De Gray, and Wilson [24]. It will be noted from figure 30 that the Union colorimeter glasses yield good chromaticity matches for the petroleum products represented except for ASTM color numbers 1½ to 3½, in which range the oil points fall between the spectrum locus and the Union locus. Note also that the oil points and the Union locus above ASTM color number 5 fall on the spectrum locus; that is, the standard observer would find that each such glass standard and each such oil color matches some part of the spectrum between 605 and 640 mμ. It is possible that some improvement could be achieved by choosing glass standards of higher excitation purity for the color numbers 2, 2½, 3, and 3½; such a change would make it easier for an observer to decide which glass standard is closest in chromaticity to such oils as are represented on figure 30. However, the difficulty that has probably prevented the Union colorimeter from becoming the basis of a standard method is that the luminous transmittances of lubricating oils fail to correlate well with their chromaticities; that is, no single line in the color solid can represent these colors. This essential difficulty would not be overcome by any choice of standards in a one-dimensional series. If, as seems probable from the words "lighter" and "darker" used in describing the method [10] and

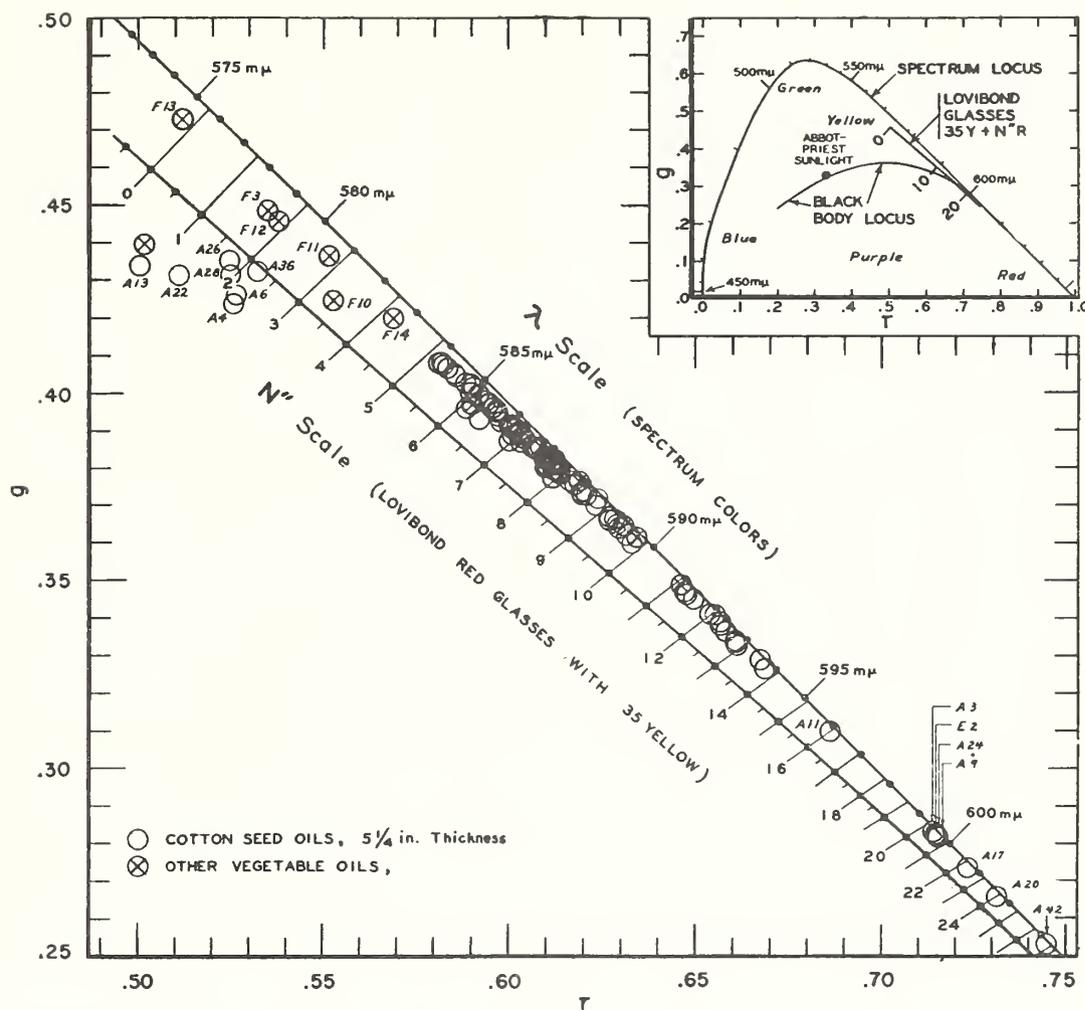


FIGURE 29. Chromaticities of vegetable oils compared to those of the combinations of Lovibond yellow and red glasses, by means of which they are graded.

The insert shows the entire (r,g) -chromaticity diagram used by McNicholas [85]; the main diagram shows only the chromaticity range covered by the vegetable oils. Note that the chromaticities of the oils are represented between the spectrum locus and the locus of 35-yellow plus N'' -red Lovibond glasses by which they are graded. The slightly oblique lines extending from the spectrum locus through the 35-yellow N'' -red locus indicate the nearest chromaticity matches to the latter. These lines if plotted on the UCS triangle (fig. 17) would be perpendicular to the 35-yellow N'' -red locus.

from a proposed photoelectric method of test [7], the chief intent is to specify the lightness or darkness of the oil color, it would be more helpful to bring together the photometric fields to be compared by a suitable prism eye-piece so that brightness differences could be more readily detected; or even use a photometer such as the Martens (fig. 8) to determine by comparison with the glass standards the luminous transmittance of the oil. The Union colorimeter, however, in spite of occasional important ambiguity of setting is very useful, and the Union color scale is well known in the petroleum industry.

4. Saybolt Chromometer Grading of Refined Petroleum

The grading of naphthas, kerosines, and so on, has for many years been carried out by comparison of the color of rather thick layers (up to 20 in.) of the refined oil with the colors of a set of three color standards made of yellowish glass. The Saybolt chromometer [8] is a device for carrying out this comparison. It

TABLE 13. ASTM Union Colorimeter Standards, Lovibond analysis, luminous transmittance, T_w , and chromaticity coordinates, x , y , for standard illuminant C, and NPA color names

ASTM color number	Lovibond analysis			Luminous transmittance, T_w	Chromaticity coordinates		National Petroleum Association names
	Red	Yellow	Blue		x	y	
1 ...	0.12	2.4	0.751	0.349	0.382	Lily white.
1½60	8.0654	.400	.446	Cream white.
2 ...	2.5	26.0443	.472	.476	Extra pale.
2½ ...	4.6	27.0365	.498	.457	Extra lemon pale.
3 ...	6.9	32.0287	.525	.440	Lemon pale.
3½ ...	9.4	45.0211	.556	.423	Extra orange pale.
4 ...	14.0	50.0	0.55	.096	.591	.400	Orange pale.
4½ ...	21.0	56.0	.55	.065	.620	.376	Pale.
5 ...	35.0	93.0036	.653	.347	Light red.
6 ...	60.0	60.0	.55	.017	.676	.323	Dark red.
7 ...	60.0	106.0	1.8	.0066	.684	.316	Claret red.
8 ...	166.0	64.00020	.714	.286

consists of an artificial daylight lamp meeting the same requirements given above for the ASTM Union colorimeter, a graduated tube for the oil specimen, a holder for the glass color standards, and a prism eyepiece to

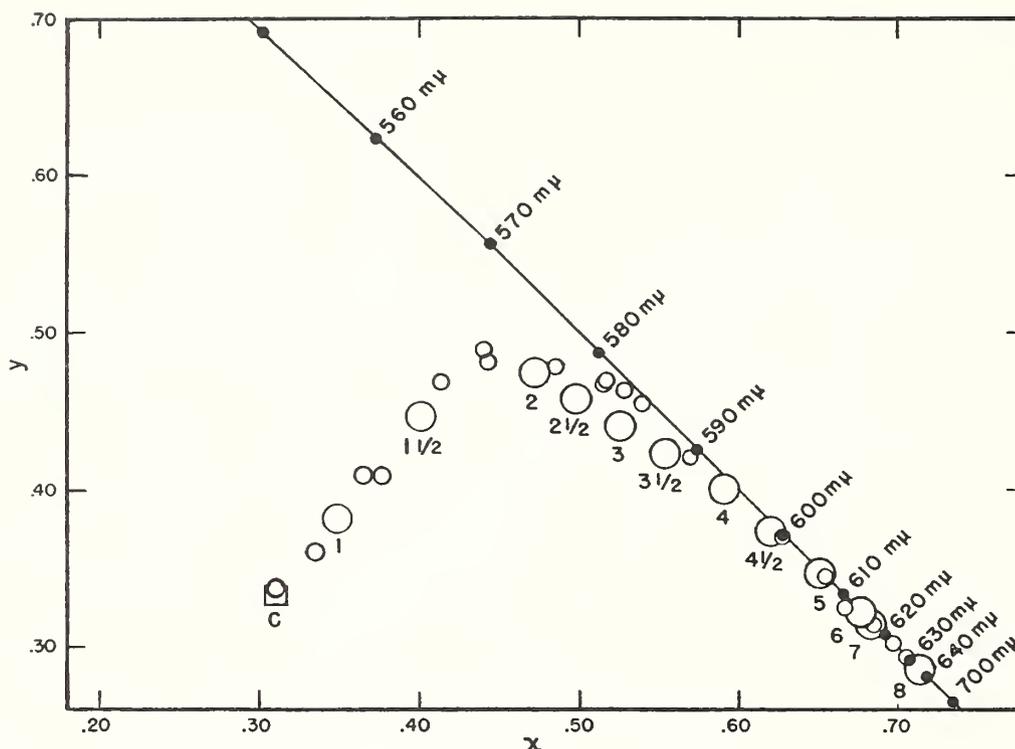


FIGURE 30. Chromaticities of the ASTM Union colors [10] compared to those of a group of petroleum products measured by De Gray, Diller, and Wilson [24].

A portion of the spectrum locus (550 to 700 $m\mu$) of the (x,y) -chromaticity diagram is also shown. It will be noted that between Union 1½ and 3½ the chromaticities of these petroleum products tend to be represented by points falling between the Union color locus and the spectrum locus.

bring into juxtaposition the two fields to be compared. There is an open ungraduated tube beneath the holder for the glass standards that serves to duplicate to a degree on the standard side of the instrument the effect of light multiply reflected within the specimen tube. The glass standards consist of two whole disks, and one "one-half" disk; these standards must have luminous transmittances, T_w , and chromaticity coordinates, x,y , for illuminant C, as follows:

	Whole disks	One-half disks
T_w	0.860 to 0.865	0.888 to 0.891
x342 to 0.350	.327 to 0.331
y367 to 0.378	.344 to 0.350
z272 to 0.291	.319 to 0.330

Oils having colors closely resembling that of distilled water are graded on the Saybolt chromometer by comparison with the half disk, more yellowish oils by one or two whole disks. The depth of oil yielding the closest color match with the glass disk is found by a prescribed procedure [8], and the color of the oil specimen is designated by a number defined from the disk used and the required depth of oil. Table 14 gives this definition.

In spite of the fact that the prism eyepiece brings the fields to be compared into juxtaposition, and so facilitates the detection of differences in brightness, the change in luminous transmittance with thickness of these refined oils is so slight that it is much less easily detected than the corresponding chromaticity change. The settings of the Saybolt chromometer therefore

probably depend essentially on nearest chromaticity match. If the oil sample is turbid, however, not even an approximate match can be obtained, and the method may be inapplicable. In these cases a thickness of the specimen yielding a chromaticity match is much darker than the standard, and no reliable setting of depth of sample to yield nearest color match can be found.

TABLE 14. Definition of Saybolt chromometer numbers from the depths of the specimen and the number of disks used to produce the nearest color match

Number of disks	Depth of oil	Saybolt number	Number of disks	Depth of oil	Saybolt number
	<i>in.</i>			<i>in.</i>	
½	20.00	+30	2	5.75	+5
½	18.00	+29	2	5.50	+4
½	16.00	+28	2	5.25	+3
½	14.00	+27	2	5.00	+2
½	12.00	+26	2	4.75	+1
1	20.00	+25	2	4.50	0
1	18.00	+24	2	4.25	-1
1	16.00	+23	2	4.00	-2
1	14.00	+22	2	3.75	-3
1	12.00	+21	2	3.625	-4
1	10.75	+20	2	3.50	-5
1	9.50	+19	2	3.375	-6
1	8.25	+18	2	3.25	-7
1	7.25	+17	2	3.125	-8
1	6.25	+16	2	3.00	-9
2	10.50	+15	2	2.875	-10
2	9.75	+14	2	2.75	-11
2	9.00	+13	2	2.625	-12
2	8.25	+12	2	2.50	-13
2	7.75	+11	2	2.375	-14
2	7.25	+10	2	2.25	-15
2	6.75	+9	2	2.125	-16
2	6.50	+8
2	6.25	+7
2	6.00	+6

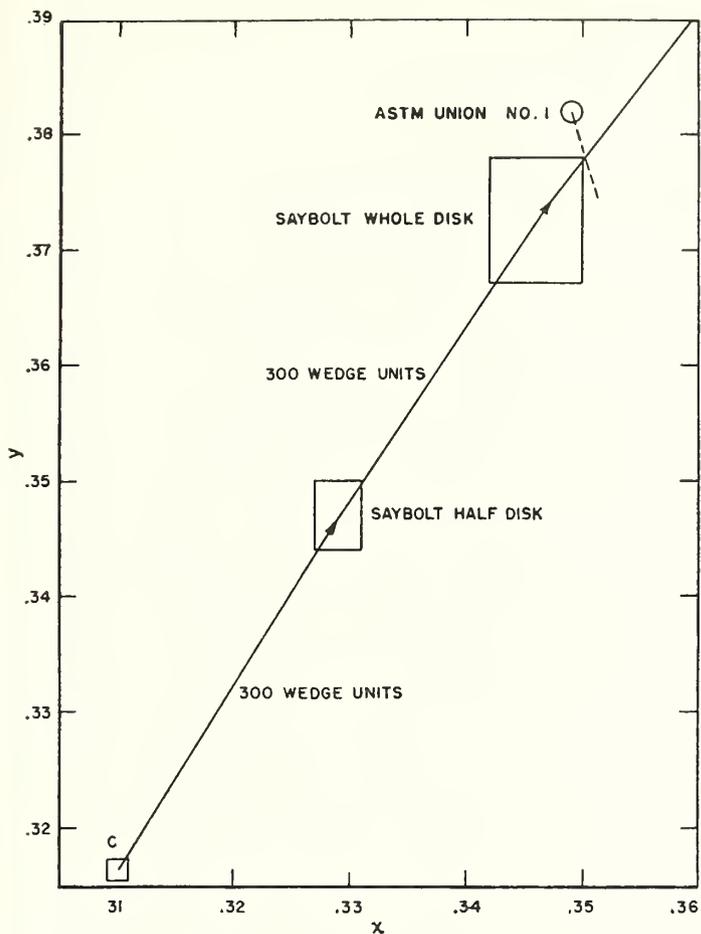


FIGURE 31. Chromaticities of the half disk and whole disk of the Saybolt chromometer compared with that of Union color 1 and with those produced by the yellow wedge of the chromaticity-difference colorimeter (see fig. 10).

The dotted line serving to evaluate the Saybolt color corresponding to Union color 1 would be perpendicular to the solid line that it intersects if they were plotted on the UCS triangle (fig. 17).

Figure 31 shows a portion of the (x,y) -chromaticity diagram on which have been plotted the rectangles corresponding to the chromaticity tolerances for Saybolt half disks and whole disks (see above). It will be noted that the tolerances are fairly wide so that variations of plus or minus 10 percent of the chromaticity change caused by introduction of the disk into the daylight beam are permissible. From table 14 it may be seen that a whole disk is never equivalent to as much as a difference of 10 in Saybolt color; so if, as seems likely, the criterion of match is nearest chromaticity, the tolerances are equivalent to less than 1 in Saybolt color.

On figure 31 is shown by a circle the point representing Union Color No. 1. It is of interest to inquire what would be the Saybolt color of an oil having ASTM Color No. 1, because we could then write down an interconversion of the two color scales. This may be done on the assumption that the settings are made by the criterion of nearest chromaticity. Since the Saybolt chromometer makes use of variable depths of oil specimen, we must first find out how the chromaticity of an oil specimen expressed on the (x,y) -diagram varies with its thick-

ness. An approximate answer is supplied by the calibration of the yellowish wedge of the chromaticity-difference colorimeter (fig. 9). The spectral transmittances of this wedge resemble fairly well those of refined petroleum oils. Figure 31 shows two vectors each corresponding to 300 yellow wedge units on the chromaticity-difference colorimeter and the beginning of a third. The first vector starts at the point representing illuminant C, and it is seen that a difference of 300 wedge units is equivalent within the prescribed tolerances to the Saybolt half disk. The second vector starts at the end of the first and indicates that adding 300 additional wedge units bridges the chromaticity differences between the Saybolt half disk and the Saybolt whole disk. The third vector commences at the end of the second and shows, in agreement with figure 30, that increasing thickness of a light-absorbing medium like refined petroleum oil gives a slightly curving line on the (x,y) -diagram with a decreasing scale as thickness is increased. The point representing Union Color No. 1 does not fall exactly on this line, but the dotted line drawn from this point intersecting the third vector corresponds to the perpendicular to the vector plotted on a uniform-chromaticity-scale triangle (fig. 17) and indicates the closest chromaticity match. The direction of the dotted line may also be found with good approximation from the family of ellipses shown on figure 16; it is the direction of the line connecting the center (Union Color No. 1) of an ellipse of this family with the point of tangency to the vector. From the intersection of the dotted line with the vector it may be seen that the chromaticity of Union Color No. 1 approaches most closely to that of about 1.10 Saybolt disk. An oil of Union Color No. 1 will therefore normally match 1.00 Saybolt disk in a thickness of $33.0/1.10=30.0$ mm, and it will match two whole disks in a thickness of 60.0 mm or 2.365 in. Reference to table 14 shows that such an oil would have Saybolt color equal to about -14; this corresponds nearly to the yellowest oil indicated on the Saybolt scale. Thus, the Saybolt scale serves to fractionate the first unit of the Union colorimeter scale. If it is permitted us to evaluate colors on the Union scale in accord with the fraction, f , of the depth of a standard solution such that unit depth matches Union Color No. 1, then we may find the Union Color, f , corresponding to any Saybolt number from the number, n , of disks used and the depth, D , defining the Saybolt number (table 14) by the relation

$$f=2.365n/2D=1.182n/D. \quad (31)$$

Table 15 shows the Union Color, f , corresponding to each Saybolt color in accord with eq 31. Correlations obtained from actual comparison of specimens to the two sets of standards may differ somewhat from that indicated in table 15 if the criterion of nearest chromaticity match be not followed exactly, or if the observer departs considerably from the ICI standard.

TABLE 15. Union color, *f*, corresponding to Saybolt color number from eq 31

Saybolt number	Union color, <i>f</i>	Saybolt number	Union color, <i>f</i>	Saybolt number	Union color, <i>f</i>
+30	0.030	+15	0.225	0	0.526
+29	.033	+14	.243	-1	.557
+28	.037	+13	.262	-2	.592
+27	.042	+12	.286	-3	.631
+26	.049	+11	.305	-4	.652
+25	.059	+10	.326	-5	.677
+24	.066	+9	.350	-6	.701
+23	.074	+8	.364	-7	.727
+22	.084	+7	.376	-8	.758
+21	.098	+6	.393	-9	.789
+20	.110	+5	.411	-10	.823
+19	.124	+4	.430	-11	.860
+18	.143	+3	.450	-12	.901
+17	.163	+2	.474	-13	.947
+16	.189	+1	.498	-14	.996
.....	-15	1.05
.....	-16	1.11

5. U. S. Color Standards for Rosin

Gum rosin has been graded by color for more than 50 years. Up to 1914 the color standards were made of rosin itself in spite of the relative impermanence of its color, and from 1914 to 1936 standards composed of combinations of Lovibond glasses were used. Brice [18] has described the selection of the present 12 official standards composed of two components of colored glass combined with one component of clear glass, all three cemented together with Canada balsam. The various combinations are given letter designations denoting the grades of rosin delimited by them and have legal status under the Naval Stores Act. The cemented face of the clear glass in each combination is fine ground so as to duplicate the slight turbidity characteristic of molded samples of rosin that commonly contain traces of fine dirt. The chromaticities are shown by solid circles on figure 32, and it will be noted that they cover approximately the same chromaticity range as ASTM Union Color

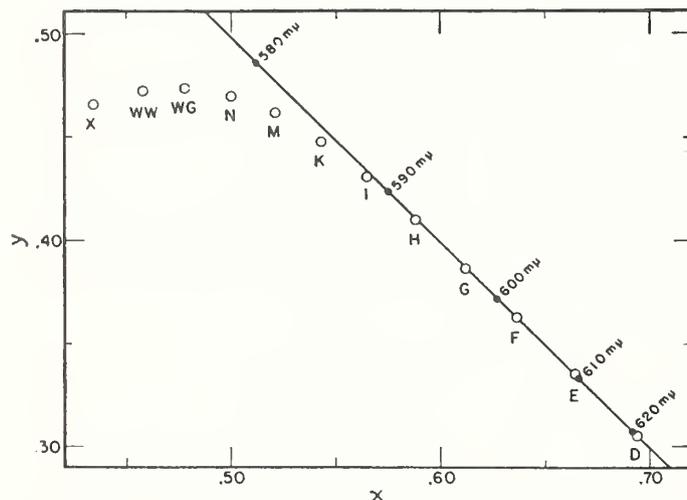


FIGURE 32. Chromaticities of the US Color Standards for Rosin according to Brice [18].

A part of the spectrum locus (580 to 620 $m\mu$) of the (*x,y*)-chromaticity diagram is also shown. This series of chromaticities is similar to that of the Union colors (fig. 30), except that it has been adjusted for progressively greater spacing toward the red end of the scale by making use of the UCS triangle (fig. 17).

1½ to 8. The chromaticity spacing was adjusted by means of the UCS triangle of figure 17 so as to progress regularly from small steps for yellow rosins to steps of about four times the initial size for reddish orange rosins.

6. Color Standards for Paint Vehicles

Many special color scales have been set up for the specification of paint vehicles (varnishes, linseed oil, tung oil, and so on). A solution of nickel sulfate and iodine [19] is used in Great Britain to define the darkest color permissible for spar varnish. A color comparator having 18 glass color standards made by Hellige, Inc., has been used for similar purposes. The Pfund color grader, made by the Munsell Color Company, compares a variable thickness of the unknown specimen with a variable thickness of a carbon-yellow glass [113]. The standard is wedge-shaped, and the cell for the specimen is likewise wedge-shaped. The Parlin (or Cargille) color standards consist of a set of 35 solutions. The first ten are Hazen platinum-cobalt solutions [47] developed originally to measure the color of natural waters and still used for that purpose under the name of APHA (American Public Health Association) standards [6]. The remainder of the Parlin color standards are caramel solutions. They have been adopted tentatively by the ASTM (Designation D 365-39T) for testing the color of soluble nitrocellulose base solutions. The Pratt and Lambert Color Standards are varnish mixtures calibrated against the Pfund color grader. The DuPont colorimeter employs six glass plates as color standards, together with a wedge of the same glass permitting a continuous variation of color between the standards. The Hellige-Stock-Fonrobert colorimeter has three sets of nine glass disks each. The unit of color is 1 mg of iodine in 100-ml of potassium iodide solution [32]. The Gardner color standards consist of 18 combinations of red and yellow Army solutions. Gardner has determined the Army and Lovibond specification for the nearest matches for all of the above-mentioned sets of color standards and has obtained also the nearest equivalents in terms of potassium dichromate solutions [32]. From these nearest equivalents it is possible to express color specifications given by any of these means in terms of any other of them.

The British Paint Research Station has recommended [130] combinations of Lovibond glasses for color grading oils and varnishes in 1-in. thickness. Some of the combinations involve colorless (or blue) glasses to be combined with the varnish or oil to match red and yellow glasses, and a device facilitating the setting up of such combinations is also recommended. The Lovibond glasses are mounted in a slide, and the two photometric fields to be compared are brought into juxtaposition by mirrors. For oils two scales, a reddish and a greenish, are provided; and for both oils and varnishes care has been taken to find glass combinations that yield a luminance as well as a chromaticity match for the average commercial product.

VI. General Methods

Standard methods of testing, specifying, and describing colors have been adopted by various organizations. We give here summaries of most such methods intended to apply to color in general.

1. TAPPI Standards T 216 m-47 and T 442 m-47

The Technical Association of the Pulp and Paper Industry has used since January, 1940, the Official Standards T 216 m-47, Spectral Reflectivity and Color of Pulp, and T 442 m-47, Spectral Reflectivity and Color of Paper [127]. The method is to determine by means of a spectrophotometer the spectral directional reflectance relative to magnesium oxide of a sufficient number of thicknesses of the pulp or paper sample that doubling the number of thicknesses will not cause appreciable change in the reflectance at any wavelength. This quantity for brevity is termed spectral reflectivity, and serves to specify the optical properties of pulp and paper that are responsible for the color, subject to the limitation that the color may depend upon the manner of illumination and viewing, which must be made a part of the report. The tristimulus specification of the color computed from the spectral reflectivity according to the ICI standard observer and colorimetric coordinate system is used. The 30 selected-ordinate method of calculation and luminous reflectivity (called visual efficiency) and chromaticity coordinates (called trichromatic coefficients) are suggested.

2. ASTM Standard D 307-44

The American Society for Testing Materials has used since 1944 the Standard Method of Test for Spectral Characteristics and Color of Objects and Materials, D 307-44 [9]. The method consists of spectrophotometry plus computation based upon the 1931 ICI standard observer and colorimetric coordinate system. Three kinds of test specimens are dealt with (a) light transmitting, (b) nonopaque reflecting, and (c) completely opaque reflecting. For these three kinds of test specimens the spectrophotometer yields (a) spectral transmittance (called spectral transmission), (b) spectral directional reflectance (called spectral apparent reflectance), and (c) spectral directional reflectivity (called spectral apparent reflectivity), respectively. Application of the standard luminosity function (y_λ of the standard observer) to these data yields respectively: (a) luminous transmittance, (b) luminous directional reflectance, and (c) luminous directional reflectivity. For transparent specimens the incident light is used as a standard relative to which the transmitted light is evaluated; for reflecting specimens the standard is either the ideal completely reflecting completely diffusing surface, or it is an opaque layer of magnesium oxide prepared by collecting the smoke from magnesium burning in air. The chromaticities of all three types of specimens are specified by the chromaticity coordinates (called trichromatic coefficients), x and y . The manner of illumination and

viewing must also be included in the report, together with an identification of the illuminant to which the color designation of the test specimen applies; that is, whether it is the standard illuminant A, B, or C, or some other illuminant.

3. Federal Specification TT-P-141a

Since June 16, 1944, there has been in force Federal Specification TT-P-141a for sampling and test methods for paint, varnish, lacquer, and related materials [30]. Three general methods of color measurement are included.

(a) Method 425.1, Color specified by trichromatic coefficients (from spectrophotometric curve). This method is identical with ASTM Standard D 307-44 except for some abridgment and inclusion of recommended angular conditions of illumination and view. For transmission measurements illumination at 0° and viewing at 180° is specified. For reflection measurements, either the illumination should be at 45° from the perpendicular and viewing at 0° or the illumination should be at 0° with diffuse viewing. The standard for reflection measurements is an opaque layer of magnesium oxide, use of the ideal perfectly reflecting perfectly diffusing surface not being permitted.

(b) Method 425.2, Color specified by trichromatic coefficients (from photoelectric tristimulus settings). In this method the unknown specimen is compared to a standard known to be permanent and spectrally similar to the specimen. From previous spectrophotometric examination by method 425, the tristimulus values, X, Y, Z , of the standard are also known. From the settings, A, G , and B , obtained for the specimen relative to those for a standard magnesium oxide surface with the amber, green, and blue filters, respectively, of the photoelectric colorimeter, the tristimulus values of the specimen are obtained from eq 11, and from these are then obtained the chromaticity coordinates, x, y (called trichromatic coefficients). The Hunter multipurpose reflectometer and Photovolt Lumetron colorimeter are mentioned as suitable instruments.

(c) Method 623, Color difference (or color change) from photoelectric tristimulus measurements. Settings A, G , and B are made in accord with method 425.2 for both of the specimens whose color difference it is desired to evaluate. The color difference is found from a modification of eq 21 as follows:

$$\Delta E = [(L_t - L_s)^2 + (a_t - a_s)^2 + (b_t - b_s)^2]^{1/2}, \quad (32)$$

where for each panel

$$\begin{aligned} L &= 10G^{1/2} \\ a &= 70G^{1/2}(A - G)/(A + 2G + B), \\ b &= 28G^{1/2}(G - B)/(A + 2G + B). \end{aligned}$$

This method applies only to panels of similar spectral characteristics. However, it is not necessary that the standard used have spectral characteristics similar to either.

4. ISCC-NBS Method of Designating Colors

A method devised at the request of the American Pharmaceutical Association and the U. S. Pharmacopoeial Convention for designating the colors of drugs and chemicals is also used for general purposes. The plan of the method was worked out by the Inter-Society Color Council, and the details were developed at the National Bureau of Standards; the method is

therefore referred to as the ISCC-NBS method of designating colors [64, 65]. This method provides a designation for every color perceived as belonging to an object (either an opaque surface, or a light-transmitting layer), and it has been extended to the colors of self-luminous areas by Kelly [66]; see figure 3. The number of color designations was purposely made small (slightly over 300) for the sake of simplicity. Since about ten million surface

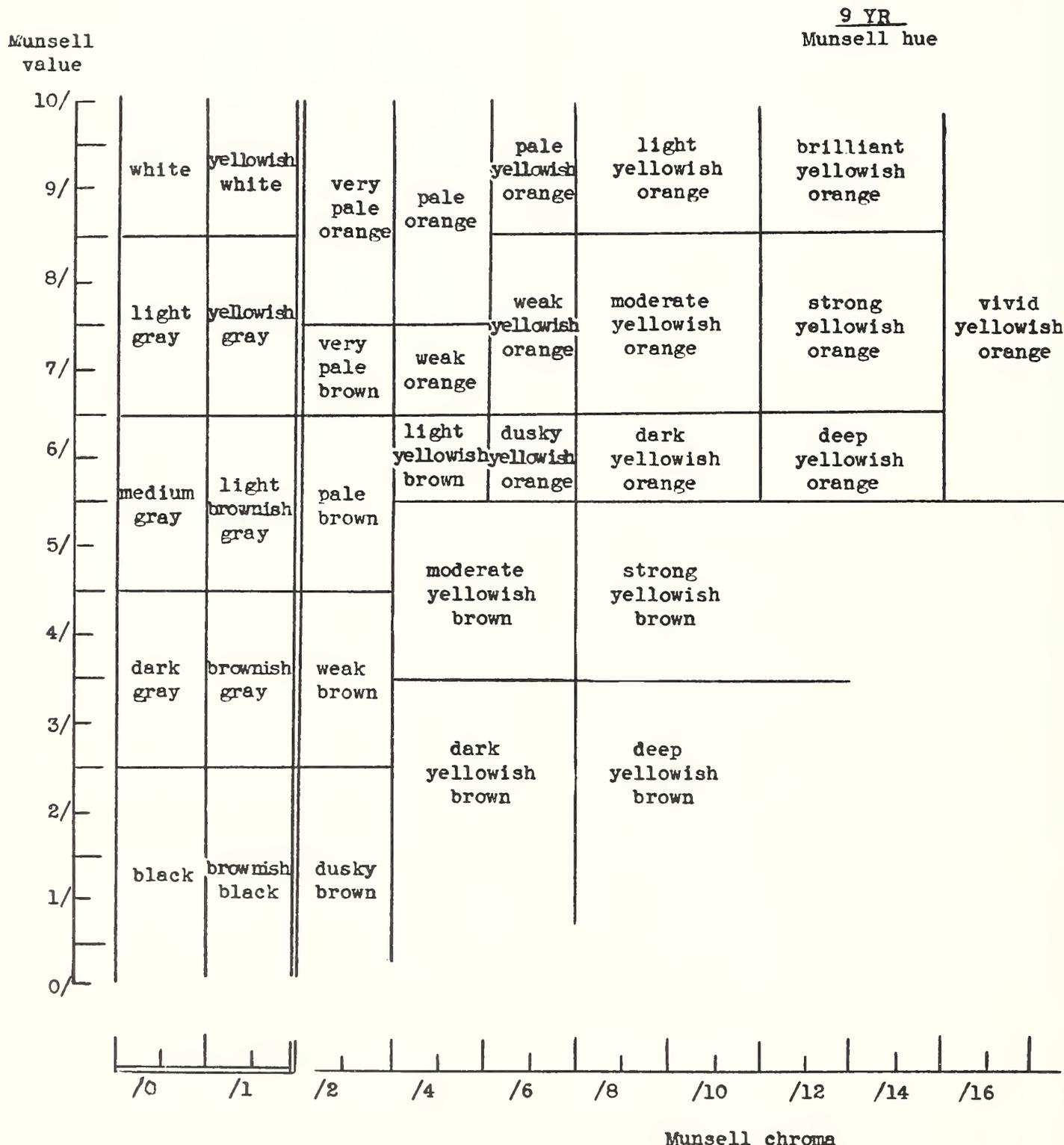


FIGURE 33. Illustration of method of defining color designations in terms of Munsell book notations. The designations for all colors of Munsell book notation hue 9YR are shown in terms of Munsell value and chroma.

colors can be distinguished by the normal human eye with optimum observing conditions, the ISCC-NBS method falls far short of supplying a different designation for each distinguishable color, or even for all colors (numbering perhaps half a million) considered to be commercially different.

The plan of the method is to divide the surface-color solid (see fig. 22) arbitrarily into about 300 compartments, and assign a designation to each in as good conformity as possible to color nomenclature currently used in art, science, and industry. The compartments embracing the black-white axis are given the designations black, dark gray, medium gray, light gray, and white. The compartments adjacent to these are given similar designations formed by adding an adjective indicating the hue, such as yellowish white, dark purplish gray, or greenish black. All other compartments take designations consisting of a hue name (red, orange, yellow, green, blue, purple, pink, brown, olive) preceded by modifiers (light, dark, weak, strong) indicating the lightness and saturation of the perceived color. Table 16 shows the complete list of modifiers used in the ISCC-NBS system; note that *vivid* is an abbreviation for *very strong*; *pale*, an abbreviation for *light, weak*; *deep*, for *dark, strong*, and so on.

The boundaries between the groups of colors known by these designations have been adjusted to accord as closely as possible with common usage and have been expressed in terms of Munsell book notation. Figure 33 shows one of the charts indicating these boundaries, and figure 34 shows the shape of the compartments selected for colors of purple hue.

TABLE 16. Modifiers of hue names in the ISCC-NBS method of designating colors

Very pale (very light, weak) ..	Very light ...	Very brilliant (very light, strong) ...	} Vivid (very strong).
Pale (light, weak)	Light	Brilliant (light, strong) ...	
Weak	Moderate	Strong	
Dusky (dark, weak)	Dark	Deep (dark, strong) ...	
Very dusky (very dark, weak) ..	Very dark....	Very deep (very dark, strong) ...	

The ISCC-NBS designations are not to be considered a substitute for numerical designations of color resulting from application of a suitable colorimetric method, but they do supply a certain precision to ordinary color designations that has previously been lacking. These color designations are used in the current edition of the National Formulary [5] throughout and in some of the monographs of the U. S. Pharmacopoeia [132]. They have been used for describing the colors of building stone [69] and soils [119] and for a considerable variety of research purposes such as, for example, the description of mica colors after heat treatment [48]. They have been used throughout the text of this circular on colorimetry. Nickerson and Newhall [104] have found the Munsell book notations for the central color of each compartment assigned an ISCC-NBS

color designation, and have recommended a system of abbreviations: see table 17.

TABLE 17. Abbreviations for the terms used in the ISCC-NBS color designations

B = blue	lt = light	pk = pinkish
b = bluish	med = medium	R = red
Bk = black	mod = moderate	r = reddish
Br = brown	O = orange	str = strong
br = brownish	o = orange (adj)	v = very
d = dusky	Ol = olive	viv = vivid
dk = dark	ol = olive (adj)	Wh = white
G = green	P = purple	wk = weak
g = greenish	p = purplish	Y = yellow
Gr = gray	Pk = pink	y = yellowish

The ISCC-NBS designations are generally unsuited for use in sales promotion in which it is important to avoid any suggestion of weakness or adulteration. Many products have weak colors, but it is considered to be poor salesmanship to say it right out, weak red or weak green. Instead, names like Rose Ash, or Cedar, or Almond Green, are used. To illustrate this point the Munsell book notations corresponding to the ICI specifications for the glass standard of the ASTM Union colorimeter (see table 13) have been found by interpolation and extrapolation on charts like figure 25, and the corresponding ISCC-NBS color designations have been read [64] and listed in table 18. It will be noted that these designations give a satisfactory description of the colors of petroleum oil, but the term, brown, suggests mud or impurities, and would probably never be applied by an oil dealer to his own product for sales promotion; compare these designations with the corresponding names that have arisen in trade practice and used by the National Petroleum Association (see first col. of table 18). Furthermore, a light colored oil suggests extreme purity; so names emphasizing the paleness of the color tend to become current in trade; note the first eight names in the NPA list. The ISCC-NBS color designations are intended to be as descriptive as possible; they are not intended to supplant color names adopted for sales promotion.

TABLE 18. Munsell book notations and ISCC-NBS Color Designations for the Union colors for petroleum products

NPA names	ASTM Union Color number	Munsell book notation	ISCC-NBS Color Designation
Lily white	1	10Y 9.6/3.8	Pale greenish yellow
Cream white	1½	8Y 8.8/8.0	Light greenish yellow
Extra pale	2	3Y 7.2/12	Strong yellow
Extra lemon pale..	2½	10YR 6.0/12	Deep yellowish orange
Lemon pale	3	7YR 5.7/13.5	Strong orange
Extra orange pale..	3½	4YR 5.0/14	Deep orange
Orange pale	4	2YR 3.2/12	Strong brown
Pale	4½	1YR 2.6/12	Strong reddish brown
Light red	5	1YR 2.2/9.5	Deep reddish brown
Dark red	6	1YR 1.3/8.5	Dark reddish brown
Claret	7	1YR 0.6/4.5	Dusky reddish brown
.....	8	1YR 0.2/1.4	Reddish black

The ISCC-NBS method has been approved for color description of drugs and chemicals by the dele-

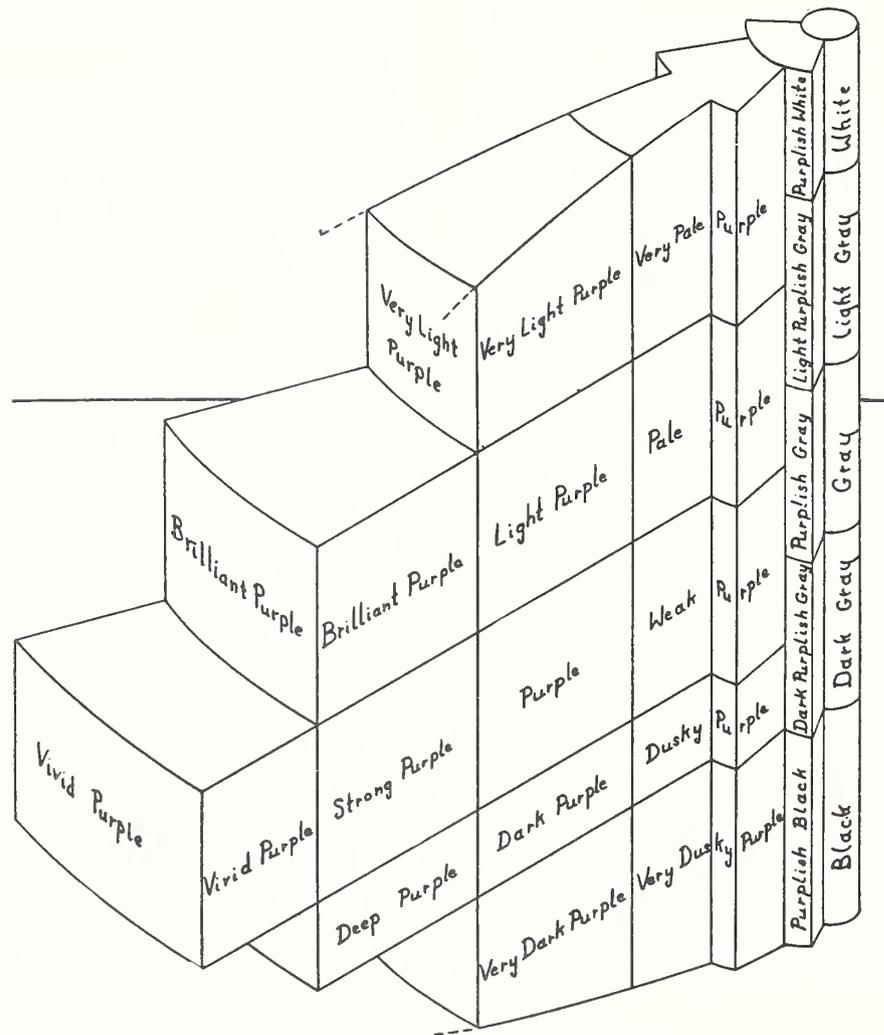


FIGURE 34. Illustration of the tridimensional nature of the definitions of color designations.

The approximate shape of the blocks of the surface-color solid corresponding to the various designations of purple colors in the ISCC-NBS method of designating colors. Figure 33 gives the definitions for only one vertical plane through a system of blocks such as this.

gates of nine national societies to the Inter-Society Color Council; and it has been recommended for general use by the American Standards Association [11] in the American War Standard for the Description and Specification of Color Z44-1942.

5. ASA War Standard Z44-1942 for Specification and Description of Color

In 1942 there was approved and published by the American Standards Association an American War Standard for the Specification and Description of Color under the designation ASA Z44-1942 [11]. This standard is of importance because it points the way toward agreement on basic procedures in colorimetry. Hence the text, with foreword and notes deleted, and with literature references brought up to date, is reproduced here verbatim:

Purpose. To recognize and recommend a basic method for the specification of color, and to facilitate its popular interpretation.

Provisions. (1) The spectrophotometer shall be recognized as the basic instrument in the fundamental characterization of color.

(2) Color specifications computed from spectrophotometric data shall be found by means of the standard observer and coordinate system adopted in 1931 by the International Commission on Illumination [20, 44, 56].

(3) For the popular identification of color, material standards may be used. The only system of material standards that has been calibrated in terms of the basic specification is represented by the 1929 edition of the Munsell Book of Color [36, 68, 91]. The use of this book is recommended wherever applicable to the specification of the color of surfaces. Approximate identifications of Munsell hue, value, and chroma may be secured by direct visual comparison with the samples in the 1929 Munsell Book of Color. When the most accurate visual comparisons are needed, the mask method [64, 65] is recommended. Wherever more exact Munsell notations are desired, they shall be found from the basic specification, Y , x , and y , by interpolation among the smoothed curves [97] for Munsell hue, value, and chroma.

(4) A descriptive name according to the ISCC-NBS

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(4) A descriptive name according to the ISCC-NBS

system of color designation [64, 65] may be derived from the Munsell notation. This name is recommended wherever general comprehensibility is desired and precision is not important. The use of color names for color specification is not recommended.

The National Bureau of Standards cooperated in the drafting of this ASA standard. We find that it summarizes well our principal techniques for the measurement, specification, and description of color.

TABLE 19. *Relative spectral irradiance of ICI illuminants A, B, and C for colorimetry*

Wave-length	E _A	E _B	E _C	Wave-length	E _A	E _B	E _C
<i>mμ</i>				<i>mμ</i>			
380	9.79	22.40	33.00	580	114.44	101.00	97.80
385	10.90	26.85	39.92	585	118.08	100.07	95.43
390	12.09	31.30	47.40	590	121.73	99.20	93.20
395	13.36	36.18	55.17	595	125.39	98.44	91.22
400	14.71	41.30	63.30	600	129.04	98.00	89.70
405	16.15	46.62	71.81	605	132.70	98.08	88.83
410	17.68	52.10	80.60	610	136.34	98.50	88.40
415	19.29	57.70	89.53	615	139.99	99.06	88.19
420	21.00	63.20	98.10	620	143.62	99.70	88.10
425	22.79	68.37	105.80	625	147.23	100.36	88.06
430	24.67	73.10	112.40	630	150.83	101.00	88.00
435	26.64	77.31	117.75	635	154.42	101.56	87.86
440	28.70	80.80	121.50	640	157.98	102.20	87.80
445	30.85	83.44	123.45	645	161.51	103.05	87.99
450	33.09	85.40	124.00	650	165.03	103.90	88.20
455	35.41	86.88	123.60	655	168.51	104.59	88.20
460	37.82	88.30	123.10	660	171.96	105.00	87.90
465	40.30	90.08	123.30	665	175.38	105.08	87.22
470	42.87	92.00	123.80	670	178.77	104.90	86.30
475	45.52	93.75	124.09	675	182.12	104.55	85.30
480	48.25	95.20	123.90	680	185.43	103.90	84.00
485	51.04	96.23	122.92	685	188.70	102.84	82.21
490	53.91	96.50	120.70	690	191.93	101.60	80.20
495	56.85	95.71	116.90	695	195.12	100.38	78.24
500	59.86	94.20	112.10	700	198.26	99.10	76.30
505	62.93	92.37	106.98	705	201.36	97.70	74.36
510	66.06	90.70	102.30	710	204.41	96.20	72.40
515	69.25	89.65	98.81	715	207.41	94.60	70.40
520	72.50	89.50	96.90	720	210.36	92.90	68.30
525	75.79	90.43	96.78	725	213.26	91.10	66.30
530	79.13	92.20	98.00	730	216.12	89.40	64.40
535	82.52	94.46	99.94	735	218.92	88.00	62.80
540	85.95	96.90	102.10	740	221.66	86.90	61.50
545	89.41	99.16	103.95	745	224.36	85.90	60.20
550	92.91	101.00	105.20	750	227.00	85.20	59.20
555	96.44	102.20	105.67	755	229.58	84.80	58.50
560	100.00	102.80	105.30	760	232.11	84.70	58.10
565	103.58	102.92	104.11	765	234.59	84.90	58.00
570	107.18	102.60	102.30	770	237.01	85.40	58.20
575	110.80	101.90	100.15	775	239.37	86.10	58.50
580	114.44	101.00	97.80	780	241.67	87.00	59.10

VII. References

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LYMAN J. BRIGGS, Director

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PHOTOELECTRIC TRISTIMULUS COLORIMETRY WITH THREE FILTERS

By

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PREFACE

The photoelectric cell is finding ever-widening usage in scientific apparatus. The present Circular describes the development and use of an "artificial eye for color measurement", which consists of a barrier-layer photocell and three selected spectral filters. The distinguishing feature of a photoelectric tristimulus colorimeter is the approximate spectral equivalence of the filter-photocell combinations of the apparatus and the tristimulus specifications of the spectrum which characterize the color vision of the average normal observer. Because of this approximate equivalence, the new apparatus will respond to color differences in much the same manner as the normal human eye. Excepting cases in which the present lack of ideal spectral equivalence introduces errors which cannot be tolerated, the photoelectric tristimulus colorimeter described herein can be used to measure both the size and the character of color differences. The potential speed and relative simplicity of the apparatus suggest its use in quantitative studies of color tolerance. Whenever suitable color standards are available, the same advantages recommend the use of the apparatus for tests of materials for compliance with color specifications.

LYMAN J. BRIGGS, *Director.*

PHOTOELECTRIC TRISTIMULUS COLORIMETRY WITH THREE FILTERS¹

By Richard S. Hunter

ABSTRACT

The term "Photoelectric colorimetry" is commonly employed to designate both photoelectric tristimulus colorimetry, used to evaluate the appearance of materials, and abridged spectrophotometry, often used to assist in chemical analyses. This paper is devoted to the first type of measurement.

For a photoelectric tristimulus colorimeter, it is desired to find three or more source-filter-photocell combinations of such spectral character that they duplicate the standard ICI observer for colorimetry. With an instrument having these combinations, tristimulus values would be obtained by direct measurement. Although no one has duplicated the ICI observer perfectly, several investigators have obtained source-filter-photocell combinations suitable for the measurement of color differences between spectrally similar samples.

To measure color differences as small as those which the trained inspectors of paint, textile, plastic, paper, and ceramic products can see, an instrument must have high precision. If the needed precision is available, a photoelectric tristimulus colorimeter may be used to measure: (1) ICI colorimetric values, x , y , and Y , relative to those of a spectrally similar, calibrated standard; (2) relative values of α and β , components of the chromaticity departure from neutral in a new uniform-chromaticness-scale mixture diagram for representing surface colors; (3) amounts of color difference between pairs of spectrally similar samples; (4) amounts of color change accompanying fading; and (5) whiteness of white and near-white surfaces.

In giving examples of the measurement of some of these different properties and in describing the errors of color measurement to which the tristimulus method is subject, reference is made to operations with the author's recently developed multipurpose photoelectric reflectometer.

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¹ A preliminary brief account of this project was presented as part of the ASTM-ISCC joint Symposium on Color held in Washington, March 5, 1941 [19].

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I. INTRODUCTION

The possibility of using photocells to measure color has attracted widespread attention ever since the photoelectric cell became available in practical form. Photoelectric cells have now been used for a number of years in spectrophotometry to measure the spectral reflective and transmissive properties of materials. From the spectrophotometric curves of samples, one may obtain the tristimulus values of color by multiplying the separate spectral values of any sample by the three distribution functions of the standard observer² and totaling the three sets of products throughout the visible spectrum (see eq. 3, below). Although the use of photocells has considerably diminished the time needed to obtain spectrophotometric curves, and although devices such as the one developed by Sears [43]³ are available to facilitate the integration, a considerable expenditure of time and effort is still required [29 p. 259] to obtain the accurate colorimetric specification of a sample by the method of spectrophotometry and integration.

Photoelectric tristimulus colorimetry is direct and rapid, because the result of integration with respect to wavelength is found automatically by the use of specially chosen source-filter-photocell combinations (see eq. 5, below). With this type of photoelectric colorimeter, the tristimulus specification of a sample is found by settings upon it using in succession each of the three or more filters in an instrument. This direct method of color measurement has at present somewhat limited application, because the best available source-filter-photocell combinations fail to be spectrally equivalent to the desired combination of the ICI standard observer and standard illuminant for colorimetry. Nevertheless there are many problems of color measurement to which the shorter procedure may be advantageously applied, and these will doubtless increase in number as research progresses.

Photoelectric tristimulus colorimetry is frequently confused with photoelectric abridged spectrophotometry [11], and both are popularly called photoelectric colorimetry. Therefore, a short discussion

² For descriptions of the ICI standard observer for colorimetry, see references [15, 20, 22, 25, 31, 33 and 45].

³ Numbers in brackets, either with or without specific page numbers, indicate references listed at the end of this Circular.

of the two experimental methods and of the differences between them is appropriate. Instruments for both procedures require a source, several filters, and a photocell. Instead of the prism or grating used in a true spectrophotometer, three to about eight spectral filters are used in an abridged spectrophotometer to isolate spectral bands. These bands are of wider wavelength ranges and are fewer in number than the bands employed in true spectrophotometry, consequently accurate spectrophotometric curves are not obtained [21]. Nevertheless, an abridged spectrophotometer is often a useful instrument. Frequently, it is known that some particular chemical or physical property of a substance may be studied by using a single spectral range or several spectral ranges which may be isolated by filters. An abridged spectrophotometer may serve adequately for the measurements needed.

A tristimulus colorimeter also requires filters, but it does not primarily measure the variation of any property of samples with respect to wavelength. Instead samples are measured with source-filter-photocell combinations which spectrally duplicate, as nearly as possible, the three distribution functions characterizing the standard observer combined with some standard illuminant. With one source and photocell, three or at most four filters are needed. However, the filters for tristimulus colorimetry differ from those used for abridged spectrophotometry in that they transmit bands of wide wavelength range.

Ives described a thermopile tristimulus colorimeter in 1915 [23], which made use of a spectrum selectively projected through a series of carefully cut templates in place of the filters employed in the more recent devices. Twyman and Perry showed in 1928 [46] that an instrument which would yield direct tristimulus measurements could be obtained by finding source-filter-photocell combinations spectrally similar⁴ to the standard-observer, illuminant combination for colorimetry.⁵ However, it was not until 1938 that Perry described an actual instrument for photoelectric tristimulus colorimetry. He called his device the Blancometer [40] because of its special suitability for white and near-white samples. Guild described the experimental model of a tristimulus colorimeter in 1934 [14], and Winch and Palmer [50], Dresler and Frühling [4], and Barnes [2], have described devices for the same purpose since that time. Gibson [11] and Van den Akker [47] have discussed the problem of obtaining the source-filter-photocell combinations giving the closest spectral equivalence to the standard observer.

The present paper describes a three-filter method for approximate photoelectric tristimulus colorimetry which has so far been used chiefly in conjunction with the author's multipurpose reflectometer [18] to measure surface color. Tristimulus measurements with this instrument have proved to be valuable in studies of the colors of paints, ceramic products, textiles, papers, pigments, inks and other materials which reflect light. The major part of the present paper is devoted

⁴ For a discussion of what is meant by spectrally similar, see footnote 7.

⁵ The three ideal source-filter-photocell combinations would each duplicate in spectral response the relative spectral distribution of one of the three functions of the ICI standard observer combined with chosen standard illuminant (as expressed in eq 1, below). A standard illuminant is, in general, relatively nonselective and plays a secondary role in determining the spectral character of the ideal source-filter-photocell combinations. In the remainder of the paper it has sometimes been convenient to speak of spectral duplication of, or spectral similarity to the standard observer when actually spectral duplication of, or spectral similarity to, the standard observer combined with a standard illuminant is meant.

to tristimulus measurements of surface colors, but it should be noted that a number of the methods suggested are equally useful for the study of volume colors by measurements of transmitted energy and for the study of illuminant colors by measurements of emitted energy.

II. TERMS, SYMBOLS, AND DEFINITIONS

It is the purpose of photoelectric tristimulus colorimetry to give by measurement quantities which provide useful information about the appearance of objects and lights. Three different classes of properties must be recognized in the study of the subject: physical, psychological, and psychophysical. Physical properties have no necessary connection with an observer; psychological properties deal directly with the impressions of an observer and are therefore not subject to exact physical measurement; psychophysical properties are an intermediate class which are capable of measurement, but which relate to the impressions of an observer. It is important to distinguish between these three classes of properties and identify the terms which refer to each.

To assist in this differentiation, table 1, which classifies a number of the terms used herein, has been prepared. This table is modeled after figure 1 in the Preliminary Draft of a Report on Nomenclature and Definitions for Colorimetry [24] by L. A. Jones, chairman of the Colorimetry Committee of the Optical Society of America. One term in table 1, chromaticness, has been added to accord with the present draft of the uncompleted report of this committee.

TABLE 1.—*Tabular arrangement of some of the terms used to describe the color of a surface*

Category.....	Physical.....	Psychophysical (Physically measured, psychologically significant).	Psychological.
Instrument.....	Spectrophotometer	Tristimulus colorimeter.....	Visual mechanism of the observer.
Product of the instrument.	Spectral apparent-reflectance curve.	Designation of the surface color by a set of tristimulus values for it; X , Y , and Z ; A , G , and B ; or other.	The perceived surface color.
The separate attributes of the surface color, and the perceived surface color.	}	(1) Luminous apparent reflectance, A_{θ} , θ , (chiefly designated below by Y); the lightness index, L . (2) Dominant wavelength, λ , or hue angle, ϕ . (3) Purity, p , or saturation index, S (2 and 3 combined) Chromaticity, indicated either by λ and p , ϕ and S , or by a pair of tri-linear coordinates, x and y , α and β , etc.	(1) Lightness. (2) Hue. (3) Saturation, or "strength." (2 and 3 combined) Chromaticness.

The definitions, and in some cases symbols, for a number of the terms used in the present paper are separated below according to the three categories.

1. PHYSICAL

A *spectrophotometer* is an instrument which measures transmission or apparent reflectance as a function of wavelength.

A *spectrophotometric curve* is a curve giving transmission, T_{λ} , or apparent reflectance, A_{λ} , of a sample as a function of wavelength.

2. PSYCHOLOGICAL

A *perceived surface color* is color experienced as a property of a surface [30]; the three attributes of a perceived surface color are lightness, hue, and saturation.

The *lightness* of a perceived surface color is that attribute which permits it to be classed as equivalent to some member of the series of grays ranging from black to white [30]. (In the Munsell system, value correlates closely with lightness under usual observing conditions.)

The *hue* of a perceived color is that attribute which permits it to be classed as red, yellow, green, blue, purple, or an intermediate [30].

Chromatic surfaces are those perceived to possess hue (not to be confused with Munsell chroma) [30].

Achromatic surfaces are those such as whites, grays, and blacks which are perceived to possess no hue [30].

Saturation is that attribute of a perceived surface color which determines the degree of its difference from the gray of the same lightness [30]. (Munsell chroma correlates closely with saturation under usual visual conditions.)

The *chromaticness* of a perceived color is determined by its hue and saturation taken together.

3. PSYCHOPHYSICAL

A *tristimulus colorimeter* is a device which measures a color stimulus in terms of three selected stimuli called primaries. The ideal source-filter-photocell combinations for a photoelectric tristimulus colorimeter are those which are spectrally equivalent to the 1931 ICI standard observer for colorimetry in combination with an appropriate illuminant.

The *1931 ICI standard observer for colorimetry* embodies the average colorimetric characteristics of 17 normal observers studied by Guild [13] and Wright [51]. The definition of the standard observer consists of three functions of wavelength, λ , showing the relative amounts (\bar{x}_λ , \bar{y}_λ , \bar{z}_λ) of three primary stimuli required to color-match the various parts of the equal-energy spectrum [15, 22, 25, 31, 33, 45].

A *tristimulus designation* of an unknown stimulus consists of the amounts (such as X , Y , Z)⁶ of the three primary stimuli required to produce a color match for it.

Apparent reflectance is the reflectance which a perfectly diffusing surface would need to have in order to produce, under the same angular conditions of illuminating and viewing, the same instrumental effect as the specimen actually measured. Apparent reflectance given as a function of wavelength is designated *spectral apparent reflectance*, $A_{\lambda(\theta_i, \theta_v)}$, and is most frequently represented by a spectrophotometric curve. Apparent reflectance weighted according to the luminosity function is designated *luminous apparent reflectance*, $A_{(\theta_i, \theta_v)}$, or \bar{Y} . For apparent-reflectance values weighted according to other spectral functions, such as those of the source-filter-photocell combinations described below, symbols which identify the functions are used.

The *Y component* of the tristimulus designation of a color repre-

⁶ It should be noted that there has not been universal agreement on the use of these symbols. The National Bureau of Standards has in the past usually used \bar{x} , \bar{y} , and \bar{z} to represent the tristimulus designation of an unknown stimulus and X , Y , and Z to represent the trichromatic coefficients of the spectrum. In the present paper, however, this former use of symbols has been discarded in favor of the above usage, which is recommended in the present unpublished draft of the OSA Colorimetry Committee report.

sents the luminous component of that color [31, 33]. Thus Y designates the luminous apparent reflectance of a surface color, the luminous transmission of a volume color, and the brightness of an aperture or illuminant color. The methods of measurement described below are in most cases applicable to problems involving volume, aperture, and illuminant colors as well as to the surface colors referred to in the text. For this reason, the symbol Y has been used below instead of A_{θ_i, θ_v} where the text refers to luminous apparent reflectance. It should be noted that the symbol A is used herein to designate settings with the multipurpose reflectometer and amber filter.

The settings, A , G , and B , with the multipurpose reflectometer employing, respectively, the amber, green, and blue filters, are, for a surface color, apparent reflectances for illumination at 45° and viewing at 0° relative to MgO. In practice the instrument is adjusted by means of a working standard calibrated in terms of MgO. Illumination at 45° and viewing at 0° have been internationally adopted as standard conditions for the colorimetry of opaque surfaces [22], because they represent a satisfactory average of the directional conditions under which surfaces are observed in everyday life. Because the combination of source, green filter, and photocell nearly duplicates the standard luminosity (\bar{y}) function times ICI illuminant C , readings obtained with the green-filter combination closely duplicate Y . Because the three source-filter-photocell combinations are nearly equivalent spectrally ⁷ to the ICI standard observer, measurements of A , G , and B of a sample constitute an approximate tristimulus description of its color.

The chromaticity of a color is its characterization by either (1) dominant wave length and purity, (2) hue angle and saturation index, or (3) a pair of trilinear coordinates (x and y , α and β , etc.).

The dominant wavelength, λ [41], and the hue angle, ϕ , of a color are both computed from the tristimulus specification to indicate hue.

The purity, p [41], and the saturation index, S , of a color are obtained from the tristimulus specification to indicate what is variously called saturation or strength.

The trilinear coordinates (x , y , and z , or α , β , and γ) are the amounts of the three primary stimuli expressed as fractions of their total. Because the three trilinear coordinates of a color are fractions of a total and must necessarily sum to unity, two of them (either x and y , or α and β) are sufficient to define the chromaticity of a stimulus.

A chromaticity diagram is a plot according to trilinear coordinates in which position of a point indicates the chromaticity of the stimulus represented.

III. THE SOURCE-FILTER-PHOTOCELL COMBINATIONS

The distinguishing feature of apparatus for photoelectric tristimulus colorimetry is that spectral elements nearly equivalent to the ICI standard observer for colorimetry must be provided. If the device is to be used for the colorimetry of light sources, television screens, or other self-luminous color stimuli, the apparatus will require filters and photocells only. If the samples are to be non-self-luminous

⁷ By "nearly equivalent spectrally to the ICI standard observer" is meant that the spectral distributions of the three source-filter-photocell combinations can be changed by transformation equations which are linear and homogeneous [31, p. 345] to spectral functions which nearly duplicate the ICI standard observer combined with illuminant C (see figure 1, below).

objects, such as opaque surfaces or transparent media, a source for illuminating the objects must be added to the filter-photocell combinations. The resulting sets of source, filter, and photocell must then be nearly equivalent in spectral response to the standard observer combined with the desired standard illuminant.

The combinations of elements described in the present paper are those which have been used for tristimulus colorimetry with the author's multipurpose reflectometer [18]. Although used for the most part in this one instrument, the source-filter-photocell combinations described below are applicable to photoelectric tristimulus colorimetry with other photoelectric devices⁸ of suitable precision.

If the three spectral response functions of the ICI observer are denoted by \bar{x} , \bar{y} , and \bar{z} and the spectral energy of the illuminant for equivalent visual analysis is denoted by E_e , then the ideal source-filter-photocell combinations for photoelectric colorimetry according to the ICI observer will satisfy the following equations:

$$\left. \begin{aligned} E_r T_1 s K_1 &= \bar{x} E_e \\ E_r T_2 s K_2 &= \bar{y} E_e \\ E_r T_3 s K_3 &= \bar{z} E_e \end{aligned} \right\} \quad (1)$$

The source in the instrument is represented by its spectral energy, E_r , the ideal filters by their spectral transmissions, T_1 , T_2 , and T_3 , the photocell by its spectral response, s , and the constants of proportionality between the two sides of the respective equations by K_1 , K_2 , and K_3 .

An instrument was desired which would give numbers corresponding to those furnished by the ICI observer rating objects under ICI illuminant C [22],⁹ a standard illuminant which is similar in spectral quality to overcast sky. Accordingly E_c representing ICI illuminant C , was substituted for E_e in eq 1. Since a source of high efficiency and small area is needed in the apparatus, a Mazda projection-type incandescent lamp was chosen. This is operated at approximately 3,100° K, and hence the relative spectral irradiance corresponding to 3,100° K [44] was used for E_r . The General Electric light-sensitive cell [10] was chosen because¹⁰ of the high current it generates for a given illumination and the relatively high response to light of the blue region of the spectrum, and because data giving the spectral response of the cells available for the author's instrument were at hand.¹¹

It was recognized that ideal filters satisfying eq 1 would not be found. No attempt was made to duplicate with a single source-filter-photocell combination the complete \bar{x} function with its two components. Instead, the two components were separated as suggested by Gibson [11], and the short-wave component was taken to be spectrally similar to the \bar{z} function.¹² A combination was then sought

⁸ These combinations, for example, have been used in the Lumetron Colorimeter, Photovolt Corporation, 95 Madison Avenue, New York, N. Y.

⁹ By using one of the color-temperature altering filters described by Gage [9] or Estey [5] with each of the three source-filter-photocell combinations, it is possible to obtain the effect of raising or of lowering the color temperature of E_e . Thus it is possible to simulate not only visual observations made under ICI illuminant C , but visual observations made under light from a yellow incandescent lamp, or from blue sky.

¹⁰ For a more detailed account of the selection of the photocells used for tristimulus colorimetry in the multipurpose reflectometer, see [18, p. 584, and 18a, p. 538].

¹¹ Kindly supplied by B. T. Barnes, of the General Electric Co., Nela Park, Cleveland, Ohio.

¹² Guild [14], Perry [40], Dresler and Frühling [4], Barnes [2], and Van den Akker [47] have all pointed out that a fourth source-filter-photocell combination is preferable to using the \bar{z} combination in order to simulate suitably the short-wave component of the \bar{x} function. However, the author is chiefly interested in the measurement of small differences in color with the simplest procedures possible. If three filters will suffice, a fourth adds undue complexity to the measurements. Until important improvements are made in the degree to which the three present filters duplicate the ICI observer, the use of a fourth filter is unwarranted.

to duplicate the long-wave component only. This component of the $\bar{x}E_C$ function is approximated by using an amber filter having a spectral transmission, T_A . The $\bar{y}E_C$ and $\bar{z}E_C$ functions are approximated by using green (T_G) and blue (T_B) filters, respectively.

TABLE 2.—Spectral character of the individual components and of the source-filter-photocell combinations used for photoelectric tristimulus colorimetry with the multipurpose reflectometer [18]

Wave-length <i>mμ</i>	Spectral transmissions (Corning filters)			E_I , relative spectral irradiance of source at 3,100° K [44]	s , spectral response of GE cell No. 55 (in micro- amperes per milli- watt)	Spectral specification of the three source-filter-photocell combina- tions. (Each value of k has been chosen so that the sum of the column is 100,000.)		
	T_A , amber filter of No. 330 yellow glass and No. 978 green glass	T_G , green filter of No. 428 green glass and No. 330 yellow glass	T_B , blue filter of No. 554 blue glass and No. 038 yellow glass			$E_I T_A s k_A$ (amber)	$E_I T_G s k_G$ (green)	$E_I T_B s k_B$ (blue)
380	-----	0.010	0.000	0.1188	94.5	-----	31	-----
90	-----	.010	.000	.1425	101.5	-----	39	-----
400	-----	.011	.000	.1688	107.8	-----	56	-----
10	-----	.011	.020	.1979	113.9	-----	70	337
20	-----	.012	.298	.2294	119.5	-----	92	6,109
30	-----	.013	.443	.2634	124.7	-----	120	10,885
40	-----	.017	.478	.2998	129.7	-----	185	13,901
450	-----	.023	.482	.3383	134.2	-----	291	16,368
60	-----	.035	.445	.3790	138.8	-----	514	17,511
70	-----	.050	.338	.4215	143.0	-----	842	15,237
80	-----	.077	.195	.4658	147.2	-----	1,476	10,001
90	0.004	.107	.090	.5114	151.3	96	2,315	5,208
500	.010	.138	.045	.5582	155.5	269	3,350	2,922
10	.020	.173	.015	.6061	159.1	596	4,664	1,082
20	.038	.209	.003	.6548	162.3	1,248	6,210	239
30	.063	.235	.000	.7040	164.9	2,258	7,628	-----
40	.095	.249	-----	.7536	167.0	3,695	8,763	-----
550	.128	.251	-----	.8033	168.4	5,351	9,496	-----
60	.164	.243	-----	.8531	168.7	7,291	9,778	-----
70	.196	.222	-----	.9025	167.4	9,148	9,378	-----
80	.224	.193	-----	.9516	162.9	10,727	8,366	-----
90	.237	.163	-----	1.0000	156.3	11,443	7,125	-----
600	.238	.135	-----	1.0476	147.5	11,363	5,833	-----
10	.227	.109	-----	1.0945	135.4	10,393	4,516	-----
20	.206	.086	-----	1.1403	117.3	8,511	3,216	-----
30	.181	.067	-----	1.1850	97.7	6,472	2,170	-----
40	.153	.053	-----	1.2284	79.1	4,594	1,440	-----
650	.122	.040	-----	1.2704	60.8	2,910	864	-----
60	.099	.032	-----	1.3111	43.7	1,752	512	-----
70	.077	.025	-----	1.3503	28.5	914	268	-----
80	.057	.020	.000	1.3880	18.0	439	140	-----
90	.043	.017	.002	1.4240	11.9	226	81	25
700	.030	.014	.003	1.4584	8.8	117	50	28
10	.022	.012	.004	1.4912	7.2	74	36	32
20	.016	.010	.004	1.5223	6.1	46	25	28
30	.011	.009	.005	1.5517	5.8	31	22	34
40	.008	.008	.004	1.5793	5.7	22	20	27
750	.005	.007	.004	1.6055	5.5	12	17	26
					Σ -----	99,998	99,999	100,000

Spectral specifications for the source-filter-photocell combinations chosen, $E_I T_A s k_A$, $E_I T_G s k_G$, and $E_I T_B s k_B$, are given in table 2. Included in the same table are the spectral transmissions of the three filters,¹³

¹³ The author is indebted to H. J. Keegan, of this Bureau, for numerous spectral-transmission curves obtained during the course of this work.

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the spectral irradiance from a source at 3,100° K., and the spectral response of GE light-sensitive cell No. 55. The values of k were adjusted so that each of the final three columns would total 100,000. Each filter consists of two components, all six components chosen being from stock manufactured by the Corning Glass Works.¹⁴

The success with which the source-filter-photocell combinations duplicate the ICI standard observer combined with illuminant C is shown graphically in figure 1, in which $\bar{x}E_C$, $\bar{y}E_C$, and $\bar{z}E_C$, which

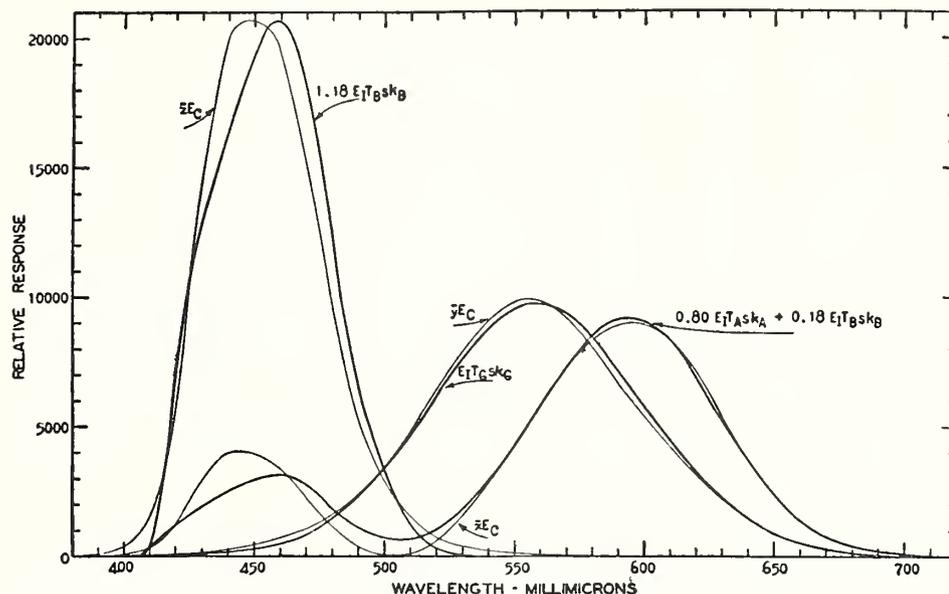


FIGURE 1.—The ICI observer, represented by the curves $\bar{x}E_C$, $\bar{y}E_C$, and $\bar{z}E_C$, compared to its approximate duplication by the source-filter-photocell combinations used in the multipurpose reflectometer.

enclose areas of approximately 98,000, 100,000, and 118,000, respectively, are plotted as functions of wavelength together with

$$0.80E_I T_A s k_A + 0.18E_I T_B s k_B,$$

$$1.00E_I T_G s k_G,$$

and

$$1.18E_I T_B s k_B,$$

which enclose respectively equal areas. The duplication is far from perfect and can be improved by further work. Also, as already noted,

¹⁴ It is possible to obtain suitable tristimulus filters from Corning glass available at the present writing (October 1941), by using the following designations and thicknesses:

Component	Catalog number	Melt designation	Thickness
Yellow component of amber filter.....	326	190	<i>mm</i> 2.42
Green component of amber filter.....	978	* $\beta_{640}=0.338$	1.3
Blue-green component of green filter.....	428	194	3.65
Yellow component of green filter.....	330	17	3.30
Blue component of blue filter.....	554	211	5.00
Yellow component of blue filter.....	038	206	1.5

*This Corning β value bears no relation to the trilinear coordinate β .

the use of a fourth filter would improve the duplication. The following approximate equations designate the relationships shown in figure 1 and serve as the basis for comparison of ICI tristimulus values and photoelectric tristimulus values:

$$\left. \begin{aligned} \bar{x}E_C &\doteq 0.80E_I T_A s k_A + 0.18E_I T_B s k_B \\ \bar{y}E_C &\doteq 1.00E_I T_G s k_G \\ \bar{z}E_C &\doteq 1.18E_I T_B s k_B. \end{aligned} \right\} (2)^{15}$$

IV. REDUCTION OF PHOTOELECTRIC TRISTIMULUS DATA

Values of A , G , and B obtained from a photoelectric tristimulus colorimeter may be converted to useful information about the colors of the samples measured in a number of different ways. Each procedure for reducing data gives numbers which are useful for a particular purpose. In table 3 the quantities which can be computed are identified by the equations for finding them and by their principal uses.

As previously noted, the methods of measurement suggested herein refer chiefly to surface colors. It is believed, however, that several of these suggested methods will prove to be equally applicable to the evaluation of volume colors from transmission measurements and to the evaluation of the colors of self-luminous objects from measurements of radiant flux.

¹⁵ The author is indebted to Miss N. J. Hendley (now Mrs. E. M. Furness) for assistance in finding transformations of the source-filter-photocell combinations which were closely equivalent to the standard observer. After experiment with several transformations giving slightly closer equivalence to the standard observer, it was decided that the simple transformations indicated by eq 2 were more practical for ordinary use.

TABLE 3.—Uses of photoelectric tristimulus measurements

Quantities computed from settings	Equations used	Uses of results
The coordinates, x , y , and Y of the ICI standard system.	<p>From eq 5 and 6:</p> $x \doteq (0.80A + 0.18B)/\Sigma$ $y \doteq G/\Sigma$ $\Sigma \doteq 0.80A + G + 1.36B$ $Y \doteq G$	To find whether sample meets color requirements specified by x , y , and Y . To represent colors in the standard ICI system.
The chromaticity coordinates, α and β , of a uniform-chromaticness-scale system.	<p>Eq 8:</p> $\alpha \doteq (A - G)/(A + 2G + B)$ $\beta \doteq 0.4(G - B)/(A + 2G + B)$	To designate chromaticity in a coordinate system yielding approximately uniform-chromaticness scales for surface color. To indicate changes of chromaticness of specimens with time, exposure, change of composition, change of method of preparation, or other treatment.
The amount of color difference, ΔE , between two surface colors.	<p>Eq 15:</p> $\Delta E = f_{\phi} \{ [7Y^{1/4} \sqrt{\Delta\alpha^2 + \Delta\beta^2} \cdot 10^2]^2 + [k_1 \Delta(Y^{1/2})]^2 \}^{1/2}$	To measure the amount of color difference between two surface colors in NBS units. To find whether a sample differs from a standard by more or less than a specified color tolerance. To measure the amount of color change resulting from exposure, change of composition, change of method of preparation, or other treatment of the sample.
The coordinates, α' , β' , and L' of a uniformly-spaced surface-color solid.	<p>Eq 14:</p> $\alpha' = 700 Y^{1/4} \alpha$ $\beta' = 700 Y^{1/4} \beta$ $L' = k_1 Y^{1/2}$	To designate surface color using coordinate scales which give perceptually nearly uniform measures of stimulus difference.
The hue angle, ϕ , the saturation index, S , and the lightness index, L .	<p>Eq 16:</p> $\phi \doteq \text{angle whose tangent is } \beta/\alpha$ $S \doteq \sqrt{(\alpha^2 + \beta^2)} Y^{1/3}$ $L \doteq Y^{1/2}$	To approximate the hue, the saturation, and the lightness of colors by separate numbers.
The hue, saturation, and lightness components, $\Delta H'$, $\Delta S'$, and $\Delta L'$, of a surface-color difference in NBS units.	<p>Eq 17:</p> $\Delta H' = 12.2 Y^{1/4} \sqrt{\alpha^2 + \beta^2} \Delta\phi$ $\Delta S' = 700 Y^{1/4} \Delta(\sqrt{\alpha^2 + \beta^2})$ $\Delta L' = k_1 \Delta(Y^{1/2})$ <p>($\Delta\phi$ in degrees)</p>	To separate a surface-color difference into the hue, saturation, and lightness components of the difference in NBS units.
The whiteness, W , of a white or near-white surface.	<p>Eq 19:</p> $W = 1 - \left\{ 30 \sqrt{\alpha^2 + \beta^2} \right\}^2 + \left[\frac{1 - Y}{2} \right]^2 \}^{1/2}$	To designate a white or near-white surface with a number on a scale of whiteness which gives an MgO surface a value of 1.00 and a black surface a value of zero.
Yellowness.....	<p>Eq 20:</p> $Y_{\text{ellowness}} = (A - B)/G$	To give a white or near-white surface a number which indicates degree of yellowness if positive, degree of blueness if negative.

1. TO ICI VALUES, x , y , AND Y

The ICI standard system [22] for designating colors has international recognition and is therefore widely used in the specification of colors. The tristimulus specification of a surface color according to the ICI observer is the summation through the spectrum of the spectral apparent reflectance of the sample relative to MgO ($A_{\lambda(\theta_i, \theta_v)}$ being measured by a spectrophotometer), times the spectral energy, E_C , of the illuminant, times the respective spectral distribution functions, \bar{x} , \bar{y} , and \bar{z} of the ICI observer:

$$\left. \begin{aligned} X &\equiv \sum_{\lambda=0}^{\lambda=\alpha} A_{\lambda(\theta_i, \theta_v)} E_C \bar{x} \Delta \lambda \\ Y &\equiv \sum_{\lambda=0}^{\lambda=\alpha} A_{\lambda(\theta_i, \theta_v)} E_C \bar{y} \Delta \lambda \\ Z &\equiv \sum_{\lambda=0}^{\lambda=\alpha} A_{\lambda(\theta_i, \theta_v)} E_C \bar{z} \Delta \lambda. \end{aligned} \right\} \quad (3)$$

If the same sample is compared with MgO in a photoelectric tristimulus colorimeter, the following summations through the spectrum are made automatically:

$$\left. \begin{aligned} \sum_{\lambda=0}^{\lambda=\alpha} A_{\lambda(\theta_i, \theta_v)} E_I T_A s k_A \Delta \lambda &\equiv A \\ \sum_{\lambda=0}^{\lambda=\alpha} A_{\lambda(\theta_i, \theta_v)} E_I T_G s k_G \Delta \lambda &\equiv G \\ \sum_{\lambda=0}^{\lambda=\alpha} A_{\lambda(\theta_i, \theta_v)} E_I T_B s k_B \Delta \lambda &\equiv B \end{aligned} \right\} \quad (4)$$

A , G , and B are, by definition, the settings of the sample relative to those of standard MgO with the amber, green, and blue filters, respectively.

By multiplying both sides of eq 2 by $A_{\lambda(\theta_i, \theta_v)}$ and making summations through the spectrum, we find by way of eq 3 and 4 that:

$$\left. \begin{aligned} X &\doteq 0.80A + 0.18B, \\ Y &\doteq 1.00G, \\ Z &\doteq 1.18B. \end{aligned} \right\} \quad (5)$$

The equations are only approximate, because the source-filter-photo-cell combinations are spectrally somewhat different from the standard ICI observer, as shown in figure 1. However, photoelectric tristimulus measurements according to a substitution method using a calibrated standard spectrally similar to the samples are, as is shown below, suitably accurate for many purposes.

Since the \bar{y} function of the ICI observer is the luminosity function, approximate values of luminous apparent reflectance are given directly by settings with the green filter. To indicate chromaticity in the ICI system, the trilinear coordinates of a color are computed as follows:

$$\left. \begin{aligned} x &\equiv X/(X+Y+Z), \\ y &\equiv Y/(X+Y+Z). \end{aligned} \right\} \quad (6)$$

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Figure 2 shows the ICI (x,y)-diagram and the corresponding photoelectric approximation to the diagram plotted in the same coordinates. In each diagram are the spectrum locus and points representing 12 differently colored porcelain-enamel plaques,¹⁶ each illuminated by

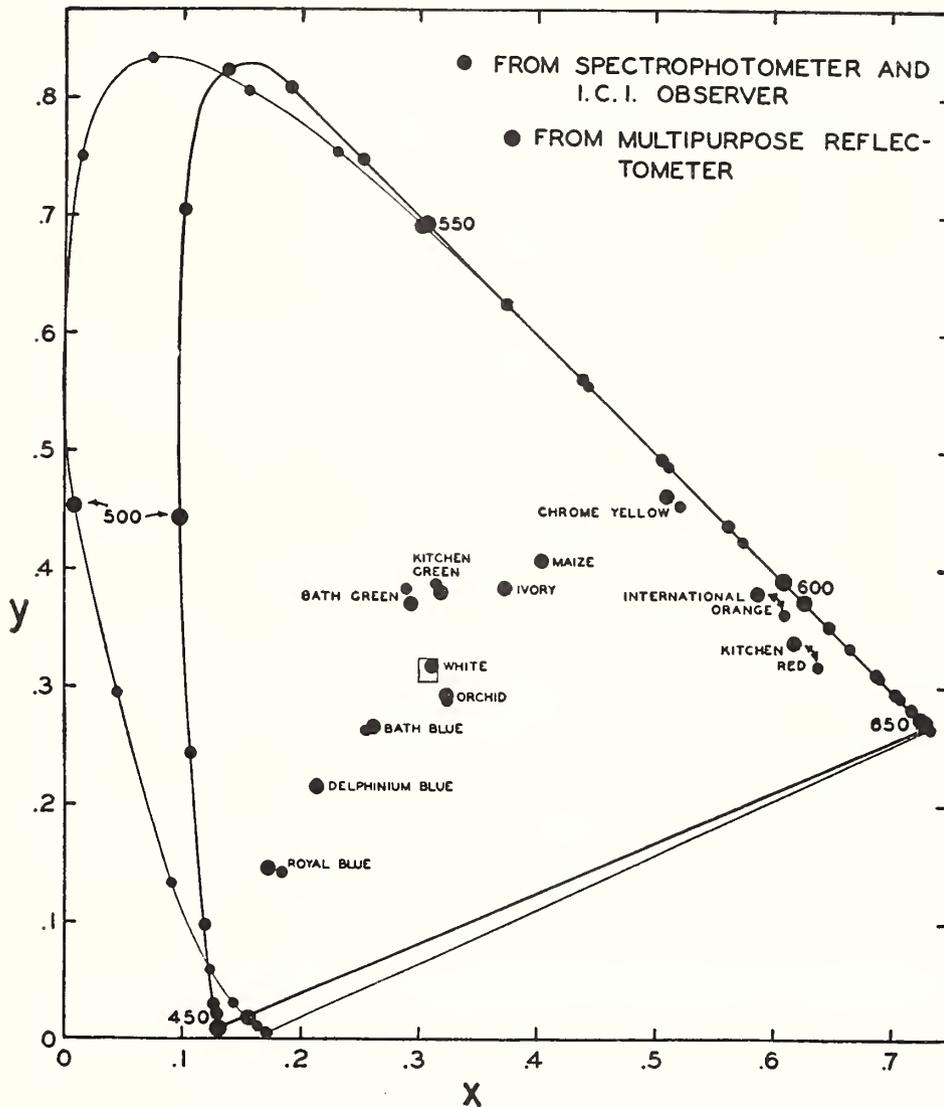


FIGURE 2.—ICI (x,y)-diagram and corresponding photoelectric approximation to the diagram with the point for white (MgO) common to both diagrams.

The pairs of points for the 11 chromatic porcelain-enamelled plaques show the errors in chromaticity measured by the photoelectric tristimulus method. The small rectangle near the center gives boundaries shown in figure 7, below.

ICI illuminant *C*. The point $x=0.3101$ and $y=0.3163$, representing a freshly prepared magnesium-oxide surface similarly illuminated is common to both diagrams. Each of the twelve plaques was measured relative to MgO, first with a spectrophotometer and then with a multipurpose reflectometer; the values of x and y obtained from the two sets of data were computed and plotted in figure 2.

¹⁶ The standards used are 10 kitchen and bathroom colors [36], and plaques representing medium chrome yellow [6] and international orange [7].

Coordinates of the spectrum locus of the ICI mixture diagram are published in many places [15, 22, 25, 31, 33, 45]; coordinates of the boundary of the photoelectric approximate chromaticity diagram were computed by applying eq. 5 and 6 to the values of $E_I T_A s k_A$, $E_I T_G s k_G$ and $E_I T_B s k_B$ given in table 2. At the ends of the visible spectrum (380 to 420 $m\mu$ and 670 to 750 $m\mu$) the source-filter-photo-cell combinations imperfectly duplicate the ICI standard observer as the distributions diminish to zero. Therefore the trilinear coordinates which would result from photoelectric measurement of the spec-

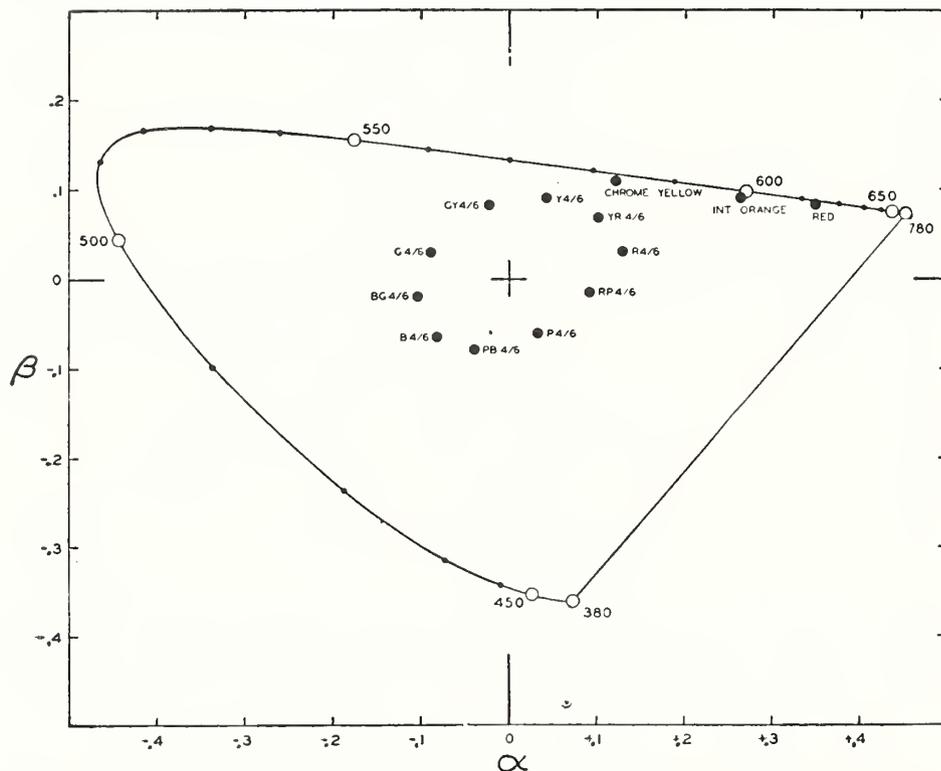


FIGURE 3.—The (α, β) -uniform-chromaticity-scale diagram for surface colors showing the spectrum locus and points representing ten Munsell colors of four-value and six-chroma [38], medium chrome yellow, international orange, and toluidine red.

trum for these wavelengths of low response are highly inaccurate and have been left out of figure 2.

2. TO (α, β) -UNIFORM-CHROMATICITY-SCALE COORDINATES

The (α, β) -chromaticity diagram shown in figure 3 has been planned especially for representing surface colors measured with the multi-purpose reflectometer and tristimulus filters. Cartesian coordinates and an origin located at the point representing MgO were used because of the greater convenience for plotting chromaticity, and because of the simple relation afforded thereby between position of point in the diagram and saturation index and hue angle of the color represented. The convenience of these two features was recognized by Breckenridge and Schaub, who used them in their earlier RUCS triangle [3]. In

addition to these features and the advantage of easy computation from settings with a reflectometer, values of α and β have an important advantage as uniform-chromaticness scale coordinates for representing the colors of paints, papers, textiles, and opaque ceramic products. The equations defining α and β were chosen, as pointed out by Scofield, Judd, and Hunter [42] to give measures of chromaticity in improved accord with visual estimates of the chromaticness of opaque surfaces.

The coordinates of the chromaticity diagram are defined by:

$$\left. \begin{aligned} \alpha &\equiv \frac{2.4266x - 1.3631y - 0.3214}{1.0000x + 2.2633y + 1.1054} \\ \beta &\equiv \frac{0.5710x + 1.2447y - 0.5708}{1.0000x + 2.2633y + 1.1054} \end{aligned} \right\} \quad (7) \quad ^{17}$$

Prior to the selection of these equations defining the (α, β) -diagram, equations were sought which would make the conversion of multipurpose reflectometer settings to values in the new coordinate system a simple matter. After trial with a number of possible sets,¹⁸ the following simple equations were chosen to give approximate values of α and β in the chromaticity diagram:

$$\left. \begin{aligned} \alpha &\doteq \frac{A - G}{A + 2G + B} \\ \beta &\doteq \frac{0.4(G - B)}{A + 2G + B} \end{aligned} \right\} \quad (8)$$

Neither eq 8a nor 8b gives as close approximation to uniform-chromaticness spacing for surface colors as eq 8. Closest accord with the spacing in Judd's UCS diagram [26] is, however, afforded by the use of eq 8b.

Because the settings of A , G , and B for any white or nearly achromatic color are of roughly the same magnitude, suitable values of α and β for these colors may often be found by

$$\left. \begin{aligned} \alpha &\doteq 2.5(A - G)/10G, \\ \beta &\doteq (G - B)/10G. \end{aligned} \right\} \quad (9)$$

Values of α and β computed according to eq 7 for spectrum colors at 10-millimicron intervals are recorded in table 4 and plotted in figure 3 to form the boundary of the (α, β) -diagram. Points repre-

¹⁷ The reverse equations are

$$\left. \begin{aligned} x &= \frac{0.5583\alpha + 0.1631\beta + 0.2466}{0.0100\alpha - 1.4347\beta + 0.7951} \\ y &= \frac{-0.2515\alpha + 0.6285\beta + 0.2515}{0.0100\alpha - 1.4347\beta + 0.7951} \end{aligned} \right\} \quad (7a)$$

¹⁸ Prior to the choice of eq 8, two earlier pairs were used by the author. The first pair, suggested in 1939 [16], was

$$\left. \begin{aligned} \alpha &= \frac{2A - 2G}{2A + 5G + 3B} \\ \beta &= \frac{G - B}{2A + 5G + 3B} \end{aligned} \right\} \quad (8a)$$

and the second pair, suggested in 1940 [17], was

$$\left. \begin{aligned} \alpha &= \frac{2.5(A - G)}{A + 6G + 3B} \\ \beta &= \frac{G - B}{A + 6G + 3B} \end{aligned} \right\} \quad (8b)$$

senting three colors widely used on signs and markers have also been plotted in figure 3. In addition, points have been plotted to show the degree of agreement between the chromaticity scales of the new system and the chromaticness spacing of the Munsell system. Using Nickerson "smoothed" values of x and y for Munsell papers [38], values of α and β were computed by eq 7 for the 10 papers representing the hues and intermediate hues of 4-value and 6-chroma under ICI illuminant C . The points representing these 10 papers would be spaced at equal intervals along the circumference of a circle if the Munsell system and the (α, β) -diagram both gave perfectly uniform chromaticness spacings.

TABLE 4.—Values of α and β , computed according to eq 7, of spectrum colors at 10-millimicron intervals

Wavelength in millimicrons	α	β	Wavelength in millimicrons	α	β
380	+0.0731	-0.3604	550	-0.1793	+0.1557
90	+0.0725	-0.3608	60	-0.0924	+0.1451
400	+0.0718	-0.3612	70	+0.0001	+0.1330
10	+0.0705	-0.3617	80	+0.0952	+0.1204
20	+0.0679	-0.3622	90	+0.1879	+0.1082
30	+0.0613	-0.3611	600	+0.2688	+0.0974
40	+0.0484	-0.3579	10	+0.3320	+0.0891
450	+0.0265	-0.3528	20	+0.3754	+0.0833
60	-0.0095	-0.3430	30	+0.4035	+0.0796
70	-0.0729	-0.3146	40	+0.4229	+0.0769
80	-0.1876	-0.2361	650	+0.4352	+0.0753
90	-0.3373	-0.0977	60	+0.4423	+0.0744
500	-0.4440	+0.0446	70	+0.4458	+0.0740
10	-0.4651	+0.1317	80	+0.4483	+0.0736
20	-0.4166	+0.1661	90	+0.4501	+0.0734
30	-0.3387	+0.1688	700 to } 780 incl}	+0.4506	+0.0733
40	-0.2605	+0.1641	MgO under ICI illuminant C.	.0000	.0000

Since the chromaticness spacing of the Munsell system is known to be fairly good [37], the departure of the actual 10 points plotted in figure 3 from equal spacing on the circumference of a circle can be considered a rough measure of the degree to which the (α, β) -diagram fails to give uniform chromaticness spacing of surface colors. Comparison of the spacing of points in figure 3 with the similar spacings of points representing Munsell papers in the (x, y) -diagram as shown by Newhall [37, p. 640] indicates a considerable advantage in favor of the (α, β) -system.

3. TO THE AMOUNT OF PERCEIVED COLOR DIFFERENCE, ΔE , BETWEEN TWO SURFACE COLORS

Expressions for estimating the amount of difference perceived between two colors and for estimating the whiteness of a perceived color have recently been set up by Judd [28, 29, 32]. Visual estimates of these two properties are widely used in commerce, where it must frequently be decided whether the perceived color of one object is a suitable match for that of another, or whether a material meets a whiteness specification. By making minor changes in Judd's two expressions, methods are available for rapidly converting photoelectric tristimulus settings to values of ΔE and \bar{W} which are in good accord with perceived color difference and whiteness, respectively.

To form his empirical equation for ΔE , the approximate amount of perceived color difference, Judd first assumed that color can be represented by a three-dimensional figure in which distance is a close measure of the perceptibility of the corresponding surface-color difference. In this Euclidian solid, the chromaticity and reflectance components of color difference, ΔE_C and ΔE_Y , are vectors at right angles. By expressing these two components in a unit which gives uniform measure of perceived color difference throughout the color solid, the definition of ΔE may be written

$$\Delta E \equiv \sqrt{\Delta E_C^2 + \Delta E_Y^2} \quad (10)$$

Because the square root of luminous apparent reflectance is known to correlate well with observers' estimates of lightness under usual observing conditions, Judd wrote the reflectance component [28, 29]:

$$\Delta E_Y = k_1 \Delta(Y^{1/2}), \quad (11)$$

where Y is the luminous apparent reflectance.

The chromaticness difference corresponding to a given chromaticity difference varies with the luminous apparent reflectance of the specimens represented. Judd assumed this variation to be proportional to $Y^{1/4}$:

$$\Delta E_C = k_2 Y^{1/4} \Delta S,$$

where the chromaticity difference, ΔS , is expressed in the units of Judd's own uniform-chromaticness-scale diagram [26].

For the present work, use is made of chromaticity differences measured in the (α, β) -coordinate system, represented by $\sqrt{\Delta\alpha^2 + \Delta\beta^2}$. In order to find the factor relating chromaticity differences in Judd's UCS diagram and chromaticity differences in the author's (α, β) -diagram, points representing eight selected stimuli were plotted in each system. The stimuli were chosen so that the points representing them were distributed throughout each diagram and lines were drawn connecting various pairs of these points. It was found by measurement that the lines in Judd's diagram averaged 1.16 (from 1.10 to 1.27) times the lengths of the corresponding lines in the (α, β) -diagram:

$$\Delta E_C = 1.16 k_2 Y^{1/4} \sqrt{\Delta\alpha^2 + \Delta\beta^2}. \quad (12)$$

To establish the "NBS Unit of Color Difference",¹⁹ numbers were assigned to the constants k_1 and k_2 in the expressions representing the lightness and chromaticness components of perceived color difference, respectively. These numbers were intended to make the unit of color difference so small that measured differences of less than one unit would represent perceptually unimportant color differences in most commercial transactions. Measured differences of more than one unit, however, were expected to represent color differences which are commercially important. For k_2 the number 600 was chosen [28, 29]. The quantity k_1 is called the proximity factor; the number assigned is properly chosen on the basis of the visual proximity of the surface areas compared for color difference (see table 5 and accompanying text, below).

By replacing 1.16 times k_2 by its equivalent, 7 times 10^2 , in eq 12, substituting in eq 10, and adding a factor (f_g), explained below, to

¹⁹ This unit has since been called by Balinkin [1] the "judd", after its originator.

account for the masking of the perceived color difference by specularly reflected light from glossy surfaces, the equation for ΔE becomes

$$\Delta E = f_g \left\{ \left[7Y^{1/4} \sqrt{\overline{\Delta\alpha^2 + \Delta\beta^2}} \cdot 10^2 \right]^2 + \left[k_1 \Delta(Y^{1/2}) \right]^2 \right\}^{1/2} \quad (13)$$

As given, eq 13 is not in the most conventional form, because the chromaticity difference, $\sqrt{\overline{\Delta\alpha^2 + \Delta\beta^2}}$, is within a bracket which is itself squared. One would think that the indicated squaring should be carried out. However, the expression is given in the above form because of the convenience of evaluating it graphically. Twice in computing a value of ΔE according to eq 13, it is necessary to find the square root of the sum of two squares. These two operations were purposely provided because they can easily be done graphically with sufficient precision for color-difference measurement by using a pair of dividers and a piece of rectangular cross-section paper to get the length of the hypotenuse of the right triangle whose other two sides are known.

The constant, k_1 , and the gloss factor, f_g , remain to be discussed. With respect to k_1 , experience has shown that the expression $\Delta(Y^{1/2})$ is proportional to lightness difference only so long as the conditions for observation of the samples remain unchanged. To obtain the best agreement between instrumentally obtained data substituted in eq 13 and the average of visual estimates, it has proved necessary to vary values of k_1 to accord with the proximity of the areas visually compared. Thus k_1 has been called the proximity factor. When specimens are compared by holding their colored areas in immediate juxtaposition, differences in $Y^{1/2}$ are found to be readily apparent. The value of k_1 representing this manner of observation must therefore be relatively large and is, in practice, taken as about 100. If, however, specimens must be examined with a border or pattern separating the areas compared, the same differences in $Y^{1/2}$ are found to correspond to less discriminable lightness differences. The value of k_1 representing this manner of observation is therefore smaller, perhaps as low as 40. The values of the proximity factor, k_1 , suggested by Judd [28, p. 425] are listed in table 5.

TABLE 5.—Values of k_1 , the proximity factor, suggested by Judd [28, p. 425]

Conditions of observation giving equivalent visual estimates of color difference	k_1
Samples separated by a very narrow or nonexistent dividing line.....	120
Samples separated by a contrasting, but narrow dividing line.....	90
Samples separated by a broad patterned area different from the areas being compared.....	40
Samples evaluated for whiteness without visual reference to other samples.....	20
However a convenient value for use when samples are separated by a narrow line is.....	100

The factor f_g in eq 13 was added to account for the masking of perceived color difference by specularly reflected light [28, p. 426]. For matte surfaces, f_g is unity; for glossy surfaces it is assumed to be

$$f_g = Y / (Y + K_g),$$

where K_g is a constant referring to the conditions under which the samples are examined visually. In situations yielding considerable

admixture of specularly reflected light, K_g is made large to give correspondingly small values of ΔE . If, on the other hand, care is taken in the corresponding visual situation to illuminate the samples so that only a small portion of the incident light is specularly reflected into the eyes of the observer, K_g should be small. A value of 0.025 was found by Judd to give satisfactory correspondence between visual estimates under the latter conditions and measured values of ΔE for a number of pairs of glossy porcelain-enameled plaques. In figure 4, f_g is plotted against apparent reflectance for $K_g=0.025$.

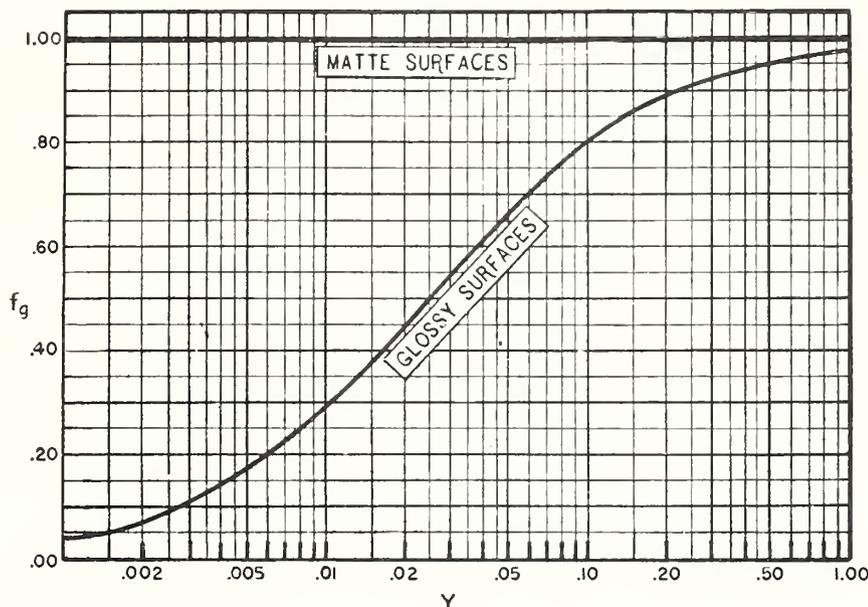


FIGURE 4.—The gloss factor, f_g , plotted as a function of Y for $K_g=0.025$.

4. TO THE COORDINATES, α' , β' , L' , OF A SOLID WITH AN APPROXIMATELY UNIFORM COLOR-PERCEPTION SPACING

From eq 13 rectangular coordinates can be derived for a color solid designed to give spacings which accord with perceptual surface-color differences observed with light-gray backgrounds. If the unit of length along each of the rectangular axes is made the NBS unit of color difference, if the point representing black is made the origin for these axes, and if the components of color parallel to the α , the β , and the $Y^{1/2}$ axes are designated by α' , β' and L' , respectively, then from eq 13 it may be shown that

$$\left. \begin{aligned} \alpha' &= 700 Y^{1/4} \alpha, \\ \beta' &= 700 Y^{1/4} \beta, \\ L' &= k_1 Y^{1/4}. \end{aligned} \right\} \quad (14)$$

A value of 100 is frequently substituted for k_1 .

The (α', β', L') -uniform-surface-color solid, in which

$$\Delta E = \sqrt{\Delta \alpha'^2 + \Delta \beta'^2 + \Delta L'^2}, \quad (15)$$

is illustrated in figure 5. White is at the top of this solid, with a value of L' equal to $k_1=100$. The chromaticity plane at an L' level of approximately 45 has been drawn. Its boundary was determined by finding the points representing colors of maximum saturation index

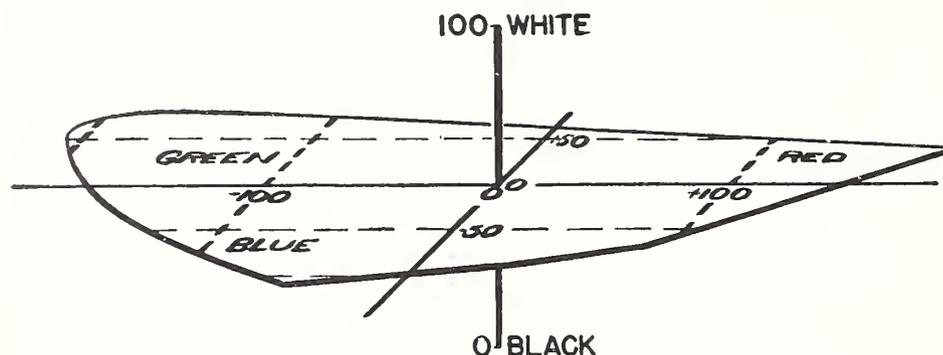


FIGURE 5.—Diagram of the (α', β', L') -surface-color solid showing the chromaticity plane for $Y=0.20$.

[34] for apparent reflectance of 0.20 (corresponding closely to $L'=45$ for $k_1=100$, or a middle gray).

5. TO THE HUE ANGLE, ϕ , THE SATURATION INDEX, S , AND THE LIGHTNESS INDEX, L

In the psychological surface-color solid, which is perhaps best exemplified in the Munsell Color System, the two components of chromaticness, hue and saturation, combine in a polar-coordinate arrangement. To correlate with hue, a number must be a measure of angle; to correlate with saturation, a number must be a measure of radius [31, p. 353 and 33, p. 11]. To obtain numbers from photoelectric tristimulus settings which will correlate well with hue, saturation, and lightness of surface-color perceptions, the following definitions have proved useful:

$$\left. \begin{aligned} \phi &\equiv \text{angle whose tangent is } \beta/\alpha, \\ S &\equiv \sqrt{(\alpha^2 + \beta^2) Y^{1/2}}, \\ L &\equiv Y^{1/2}. \end{aligned} \right\} \quad (16)$$

The above quantities have been designated the hue angle, the saturation index and the lightness index, respectively. It can be seen that the signs of both α and β must be known to place ϕ in the proper quadrant.

6. TO ESTIMATES IN NBS UNITS OF THE HUE, SATURATION, AND LIGHTNESS COMPONENTS, $\Delta H'$, $\Delta S'$, AND $\Delta L'$, OF A PERCEIVED SURFACE-COLOR DIFFERENCE

It is frequently valuable to compute not only an estimate of the size of the difference perceived between two surface colors, but also estimates of sizes of the separate hue, saturation, and lightness components of the difference.

To compute these separate components of color difference, it is necessary to convert the rectangular coordinates of the color solid

defined by eq 14 to cylindrical coordinates. The saturation attribute in this solid is measured by radius from the lightness axis as center:

$$S' \equiv \sqrt{\alpha'^2 + \beta'^2}.$$

The hue-difference component of color difference in this solid, for hue-angle differences not too large, is measured by distance along the circumference which passes through the point midway between the points representing the two colors:

$$\Delta H' \equiv \frac{S_1' + S_2'}{2} \Delta \phi$$

From these relations and eq 14,

$$\left. \begin{aligned} \Delta H' &= 12.2 Y^{1/4} \sqrt{\alpha^2 + \beta^2} \Delta \phi, \\ \Delta S' &= 700 Y^{1/4} \Delta(\sqrt{\alpha^2 + \beta^2}), \\ \Delta L' &= k_1 \Delta(Y^{1/4}), \end{aligned} \right\} \quad (17)$$

where $\Delta \phi$ is measured in degrees;

where the values which do not refer to difference measurements are taken as averages of the corresponding values for the two specimens concerned.

7. TO WHITENESS, W

The whiteness of a sample is assumed, for purposes of its measurement, to be proportional to the perceived degree of approach of its color to that of an ideal white surface. If MgO is assumed to approximate sufficiently well the ideal white, the whiteness of the unknown surface may be quantitatively defined by a relation suggested by Judd [32]:

$$W \equiv 1 - \frac{\Delta E_{\text{MgO to specimen}}}{\Delta E_{\text{MgO to black}}}, \quad (18)$$

which gives a scale of whiteness from 1.00 for MgO to zero for black. Judd found that a proximity factor (k_1) of 20 yielded numerical ratings for the whiteness of surfaces in good accord with visual estimates. By substituting this value in eq 13, the color difference between MgO and black for the assumed conditions of whiteness grading is found to be 20 NBS units. The above equations for whiteness may therefore be written

$$W = 1 - \frac{1}{20} \Delta E_{\text{MgO to specimen}}$$

Since the use of this equation is to be restricted to white and near-white surfaces, eq 13 may be somewhat simplified when incorporated into the relation for whiteness. Within the range of apparent reflectances (0.60 to 1.00) covered by whites and near whites, $(7 \cdot Y^{1/4} \cdot 10^2)/20$ does not vary greatly from 30, and, for the $\Delta(Y^{1/2})$ component, $(1.00 - Y^{1/2})$ does not differ greatly from the more easily computed $(1.00 - Y)/2$. Since, in addition, the values of α and β for MgO are both zero, an approximate expression for whiteness of a white or near-white surface may be written:

$$W = 1 - \left\{ \left[30 \sqrt{\alpha^2 + \beta^2} \right]^2 + \left[\frac{(1.00 - Y)}{2} \right]^2 \right\}^{1/2}. \quad (19)$$

Whiteness evaluated by this expression differs from that by eq 18 by less than 0.025 for W greater than 0.80. This difference is less than the uncertainty of estimates of whiteness equality by direct visual inspection. In correlating visual estimates of whiteness of white papers, finishes, and other materials with colorimetric values of the samples examined, one frequently finds that magnesium oxide is not a suitable standard for whiteness. Judd [27] had 15 experienced paper technologists each rate 29 pieces of paper for whiteness. As a result of the ratings supplied, he found that in the paper industry there are recognized apparently not one, but at least two "ideal whites." One ideal white is "natural paper white," a yellowish color representing the best grade of undyed paper. Another ideal white seems to possess almost the chromaticity of MgO; it is typical of papers "whitened" with blue dye. Some observers judge whiteness by one ideal, others by another.

The fact that some observers use ideal whites differing in color from MgO does not destroy the usefulness of relations for rating whiteness numerically. If the ideal white used by the graders of the material in question can be identified, eq 18 can be used by substituting the colorimetric coordinates of this ideal white for those of MgO.

8. TO YELLOWNESS

White and near-white surfaces are so frequently rated for degree of yellowness that it is convenient to have a scale which provides approximate psychophysical measures of yellowness. The equation

$$\text{Yellowness} = \frac{A-B}{G} \quad (20)$$

is similar to others which have been used for the same purpose [48, 49] and gives a scale of yellowness increasing from zero for MgO or any equally nonselective surface to positive values for yellowish surfaces and negative values for bluish surfaces.

V. STANDARDS FOR PHOTOELECTRIC TRISTIMULUS COLORIMETRY

For almost all photoelectric tristimulus measurements, regardless of the purpose for which they are made, a substitution method should be employed. That is, the specimens being measured should be exposed under the same circumstances as the standard with which they are compared. This standard may be either the actual reference standard, which for surface colors is usually a freshly smoked magnesium-oxide surface, or it may be a secondary or working standard, such as a porcelain-enameled plaque which has been calibrated in terms of the reference standard. If simply the differences in color between the test specimens and a specimen designated as standard are of interest, this arbitrary standard often need not be calibrated.

The present photoelectric tristimulus method is subject to error because of the above-mentioned spectral inaccuracy of the source-filter-photocell combinations. In the following section of this paper, it will be shown that this error tends to increase in amount with the spectral dissimilarity between the specimens compared. Because of this tendency, it is advantageous to use wherever possible in photo-

electric tristimulus colorimetry a working standard which is spectrally similar²⁰ to the samples being measured.

To calibrate a working standard for photoelectric tristimulus colorimetry without reference to a spectrally similar, previously calibrated standard, the spectrophotometric curve of the new standard is first obtained. From this curve, x , y , z , and Y are computed according to one of the customary methods [15, 25, 31, 45]. From these computed values, A_{std} , G_{std} , and B_{std} may be derived using eq 21, which is derived from eq 5 and 6:

$$\left. \begin{aligned} A_{std} &= \frac{Y}{y}(1.25x - 0.1907z) \\ G_{std} &= Y \\ B_{std} &= \frac{Y}{y}0.8475z \end{aligned} \right\} \quad (21)$$

As a specific example of how values of A , G , and B may be assigned a working standard, the pink plaque used for the measurements demonstrated in example 2 below, was given the following ICI colorimetric values by spectrophotometry and computation:

$$\begin{aligned} x &= 0.3585 \\ y &= 0.3345 \\ z &= 0.3070 \\ Y &= 0.606 \end{aligned}$$

Accordingly values of A , G , and B were computed for this working standard according to eq 21:

$$\begin{aligned} Y/y &= 1.81166 \\ \left. \begin{aligned} 1.25x &= 0.448125 \\ 0.1907z &= 0.058545 \end{aligned} \right\} \\ \text{Difference} &= 0.389580 \\ A &= \text{Difference} \cdot (Y/y) = 0.7058 \\ G &= Y = 0.606 \\ 0.8475z &= 0.260182 \\ B &= 0.8475z(Y/y) = 0.4714 \end{aligned}$$

When, instead of ICI values, values of α , β , and Y of the working standard are given, values of A , G , and B are computed by using equations which are essentially eq 8 reversed:

$$\left. \begin{aligned} A &= Y \left(\frac{1.0 + 3.0\alpha + 2.5\beta}{1.0 - \alpha + 2.5\beta} \right) \\ G &= Y \\ B &= Y \left(\frac{1.0 - \alpha - 7.5\beta}{1.0 - \alpha + 2.5\beta} \right) \end{aligned} \right\} \quad (22)$$

To find the colors of a series of spectrally similar specimens, none of which can be used as a working standard because of unstable color,

²⁰ By spectrally similar specimens are meant specimens which are not metamericly different (see text, p. 25).

a stable plaque is assigned values of A , G , and B which relate to the impermanent specimens rather than to its own color. To calibrate such a working standard, one of the unstable specimens must be measured on a spectrophotometer and then compared immediately with the stable plaque on the tristimulus instrument.

VI. ERRORS IN PHOTOELECTRIC TRISTIMULUS MEASUREMENTS

In the paper describing the multipurpose reflectometer [18], 18 potential sources of instrument error are listed. These 18 sources include most of those which will be found in photoelectric tristimulus instruments, and it is believed, therefore, that no further study of the errors of instruments is needed here. With special reference to the errors of photoelectric tristimulus colorimetry, however, there are several matters which merit attention. These matters are considered herein with particular reference to measurements with the author's three-filter multipurpose reflectometer.

It should be pointed out first that high precision is needed for useful photoelectric tristimulus measurements. In order to identify by an instrument, chromaticity differences as small as can be distinguished by an experienced observer, relative values of A , G , and B must be highly precise. To show what this precision must be, changes of 0.001 in the instrument settings on a hypothetical white surface and on a hypothetical yellow surface have been converted to corresponding changes in x , y , α , β , ΔE , and W by using the relations above. These are shown in table 6.

TABLE 6.—Amounts of change in chromaticity and color difference introduced by changes in instrument settings of 0.001

	For a white surface reflecting 80 percent	For a yellow surface reflecting 35 percent
If settings of the instrument should be.....		
{ A	0.800	0.500
{ G800	.350
{ B800	.050
But instead the instrument gives.....		
{ A801	.501
{ G799	.349
{ B801	.051
Changes of the following magnitudes will result.....		
{ in x	+ .00024	+ .00049
{ in y	- .00054	- .00182
{ in α	+ .00062	+ .00160
{ in β	- .00025	- .00064
{ in $\sqrt{\alpha^2 + \beta^2}$00067	.00172
{ in ΔE (maximum, NBS units).....	$\pm .45$	$\pm .94$
{ in W	- .0025

Under favorable observing conditions, a trained inspector can usually distinguish as different the colors of two light-colored surfaces if

$$\Delta E = 0.3,$$

or if

$$\sqrt{\Delta\alpha^2 + \Delta\beta^2} = 0.0005.$$

In general, it can therefore be said that a tristimulus colorimeter giving apparent reflectances imprecise by 0.001 will not serve to iden-

tify color differences as small as those which the trained color inspector can distinguish.

Although the individual settings of a multipurpose reflectometer are usually uncertain by more than 0.001, a technique is used to obtain settings with this instrument which ordinarily reduces the errors of the resulting values of x , y , α , β , ΔE , and W to considerably less than those given in table 6. With this instrument the settings on each specimen with the three filters are made consecutively. By thus taking settings with the three filters almost simultaneously and without moving the specimen, the errors ordinarily introduced by temperature drift and by change of the position of the specimen between exposures are minimized in the computed *differences* between the settings.

It may be seen from eq 8 above that the chromaticity measurement of a sample depends chiefly upon these differences between settings. These differences must be uncertain by less than 0.001 for precise photoelectric colorimetry, since changes in the differences between A , G , and B are relatively more important in determining chromaticity than changes in their magnitudes. The three readings of a sample would have to be simultaneously high, or simultaneously low by four times 0.001 (for a near-white sample) to cause a lightness-component error in ΔE as large as the chromaticity-component error caused by an inaccuracy in $A-G$ of 0.001. Resort to eq 13 above will verify this conclusion.

1. ERRORS IN TRISTIMULUS MEASUREMENTS OF CHROMATICITY DIFFERENCE DUE TO SPECTRAL INACCURACY OF THE SOURCE-FILTER-PHOTOCELL COMBINATIONS

The spectral inaccuracy of the source-filter-photocell representation of the ICI standard observer has been demonstrated above. Although the errors from this source were shown [18] to be not serious in measurements of luminous apparent reflectance, the errors from the same source are serious in measurements of chromaticity.

It was pointed out by Perry [40] that these chromaticity errors are small and can usually be tolerated when the photoelectric tristimulus method is used to measure differences in chromaticity between spectrally similar, or as he said, "spectrophotometrically similar" specimens. Under other circumstances, however, the chromaticity errors are frequently larger than can be tolerated. It can only be recommended, therefore, that the present method be used to measure color differences between spectrally similar specimens.

Before undertaking quantitative evaluations of chromaticity errors, it seems desirable to explain in some detail what Perry meant when he spoke of "spectrophotometrically similar" specimens. Actually a word suggested by Ostwald [39], "nonmetameric", seems to apply more specifically than either spectrally similar or spectrophotometrically similar to the property of the differences which is important. The stimuli produced by two illuminated specimens are metamers when there is a spectral difference between them which is of such a nature that they nevertheless have the same color. As an example, there is the frequently cited case of two specimens which have the same color under one illumination, but different colors under a second illumination. Under the first illumination, the two stimuli are metamers

for the specimens have to be spectrally different to appear different in color under the second illumination.

In the present discussion, reference is made to "degree of metamerism." It is supposed that the spectral difference for two speci-

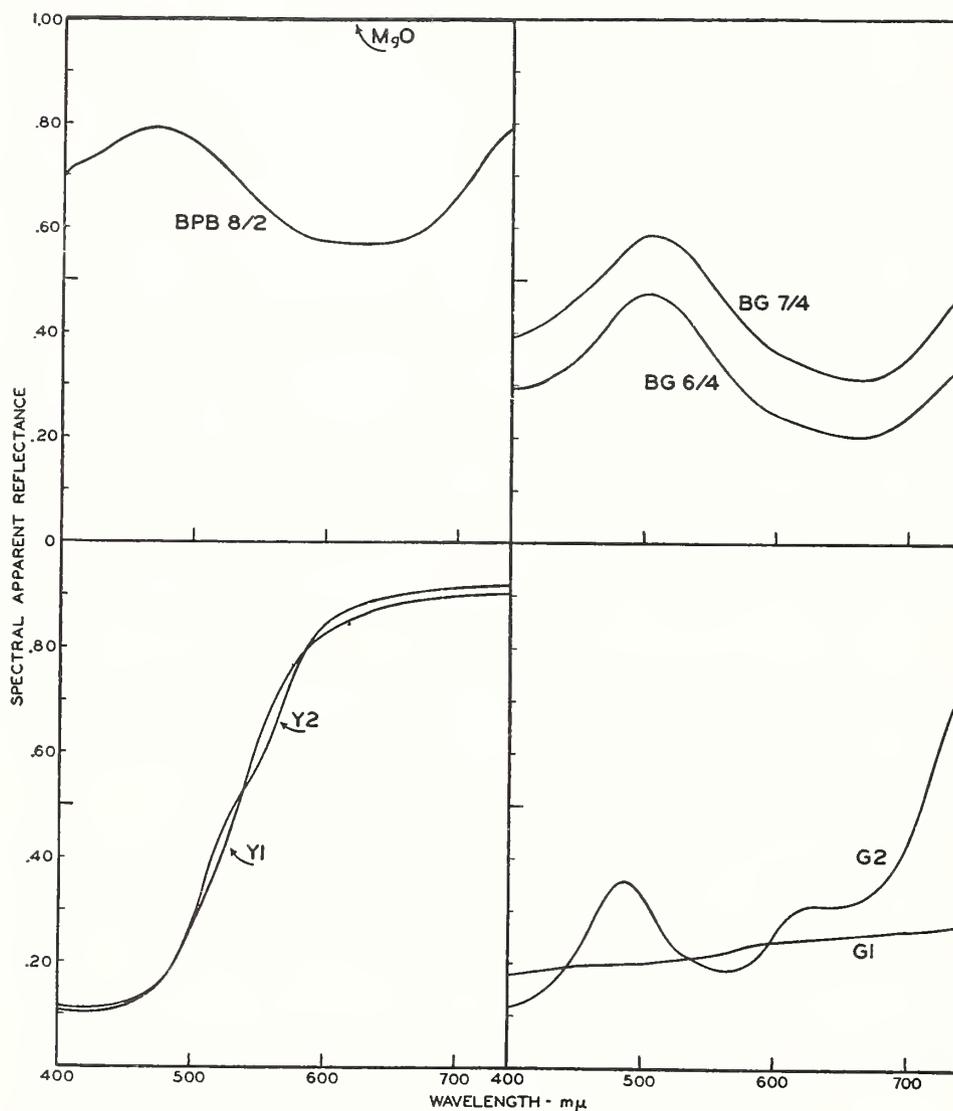


FIGURE 6.—Spectral curves of four pairs of specimens used for the study of amounts of error resulting from failure of the source-filter-photocell combinations to be spectrally equivalent to the ICI standard observer.

Upper two pairs—curves representing essentially nonmetameric differences; lower two pairs—curves representing differences in which there is appreciable metamerism.

mens under a given illumination can be separated into three components: (1) a factor of constant proportionality throughout the spectrum corresponding to the lightness difference, (2) a component which is responsible for the chromaticity difference, and (3) a metameric component yielding no color difference.

In general, the chromaticity error in photoelectric tristimulus measurement increases with the size of the latter two components. For pairs yielding no appreciable metameric component, therefore, the size of the chromaticity error is roughly proportional to the size of the chromaticity difference.

In order to find the approximate factor relating chromaticity error to size of nonmetameric chromaticity difference, and to show also that this factor fails to evaluate the amount of error when there is appreciable metamerism, four chromaticity differences were carefully studied. The errors of these four differences were computed from the spectral curves of the eight specimens involved. With each of the four pairs, one specimen was assumed to represent a standard and the other an unknown sample. True (α , β) and approximate (photoelectric) values of chromaticity were computed, using the spectral specifications of the ICI observer and the source-filter-photocell combinations, respectively. True chromaticity differences were then compared with the corresponding photoelectric-tristimulus chromaticity differences to obtain the errors of the latter values.

The spectral curves of the four pairs of specimens are shown in figure 6. With the top two pairs there is relatively little metamerism; with the bottom two there is significant metamerism. For the first pair, MgO and Munsell paper BPB 8/2 were chosen. The Munsell paper was chosen to represent a specimen which differed in chromaticity from MgO by about as much as any specimen which would be called "near white." Perry pointed out that because white and near-white specimens were relatively nonselective, significantly metameric differences between them were rare. Realizing that a photoelectric tristimulus colorimeter thus possessed special advantages for use on white materials, Perry named the device which he designed the *Blancometer* [40].

The two yellow papers, *Y1* and *Y2*, are very nearly alike in color; in fact the perceived color difference between them will change in character with change from daylight to incandescent illuminant. Thus they represent a case in which the spectral difference is chiefly a metameric difference. The degree of metamerism is even greater for the difference between the two gray samples, *G1* and *G2*.

The true chromaticity differences and the computed errors of the photoelectric tristimulus method are given for the four pairs of specimens in table 7. Since these data are computed from the spectral curves of the specimens, the errors shown are those due solely to the spectral inaccuracy of the source-filter-photocell combinations. From table 7 it can be seen that if sample and standard are not metamERICALLY different, photoelectric tristimulus measurements of chromaticity difference may be in error by roughly 10 percent of the difference measured. If there is appreciable metamerism, however, the error is not related to the size of the chromaticity difference. For this reason parentheses have been placed about the latter two "percent errors" in table 7.

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TABLE 7.—*Errors in measured chromaticity differences which result from substitution of photoelectric tristimulus combinations for the ICI standard observer*

Specimens compared	Size of the chromaticity difference $\sqrt{\Delta\alpha^2 + \Delta\beta^2}$	Error in the photoelectrically measured chromaticity difference	
		Actual	Relative to size of difference
Munsell BPB 8/2 and MgO.....	0.0229	0.0013	%
BG 6/4 and BG 7/4.....	.0150	.0018	6
Yellow Y1 and yellow Y2.....	.00187	.00043	12
Gray G1 and gray G2.....	.0160	.0061	(23)
			(38)

In figure 7 there is experimental confirmation of the conclusion that errors are small in the measurement of the chromaticities of white samples. Points representing twelve white porcelain-enameled plaques which were measured with both the multipurpose photoelectric reflectometer and the visual, subtractive colorimeter [29] are plotted in this figure. The plaques were compared in both instruments with a Vitrolite standard, for which the assigned chromaticity is shown. For none of the plaques is the disparity as great as 0.001 in $\sqrt{\Delta x^2 + \Delta y^2}$, even though it includes the errors of both instruments as well as the error due to spectral inaccuracy of the source-filter-photocell combinations. To show how large a part of the total (x,y) -diagram is covered by figure 7, its boundaries were plotted as a small rectangle near the center of figure 2.

The errors represented in table 7 and figure 7 have involved only small chromaticity differences. In figure 2, above, there is evidence which extends to large chromaticity differences the findings about errors given in table 7 for small differences. If distances between the different pairs of points representing the same colors are measured in figure 2, it will be found that they are, on the average, roughly one-tenth the respective distances from these points to the white point common to both diagrams. Since magnesium oxide, represented by the white point, was the standard against which the several specimens were measured, this average error of one-tenth the chromaticity difference is in good agreement with the errors of roughly 10 percent indicated in table 7. Thus one may expect errors which will average about 10 percent of the measured chromaticity differences for large as well as small nonmetameric differences.

2. CHANGES IN MEASURED CHROMATICITY RESULTING FROM CHANGE OF PHOTOCCELL IN AN INSTRUMENT

Of the three components of each source-filter-photocell combination, the photocell is in practice the most difficult to duplicate spectrally. In the absence of an evaluation of errors possible from all these components, it may be instructive to evaluate the changes in measured chromaticity which can result from change from one photocell in an instrument to another of the same manufacture. Of the several GE cells for which data were available, the cell most different in spectral response from cell No. 55 was cell No. 2. The relative spectral responses for these two cells are compared in figure 8.

The photoelectrically measured chromaticity difference for each pair of samples identified in figure 6 would change with substitution of

cell No. 2 for cell No. 55. The amounts of these changes were computed in the same way as the errors reported in table 7, and are given in table 8. It will be noted that the changes in computed chromaticity difference are somewhat smaller than the errors for the corresponding pairs reported in table 7, but the changes are nevertheless large enough to be important.

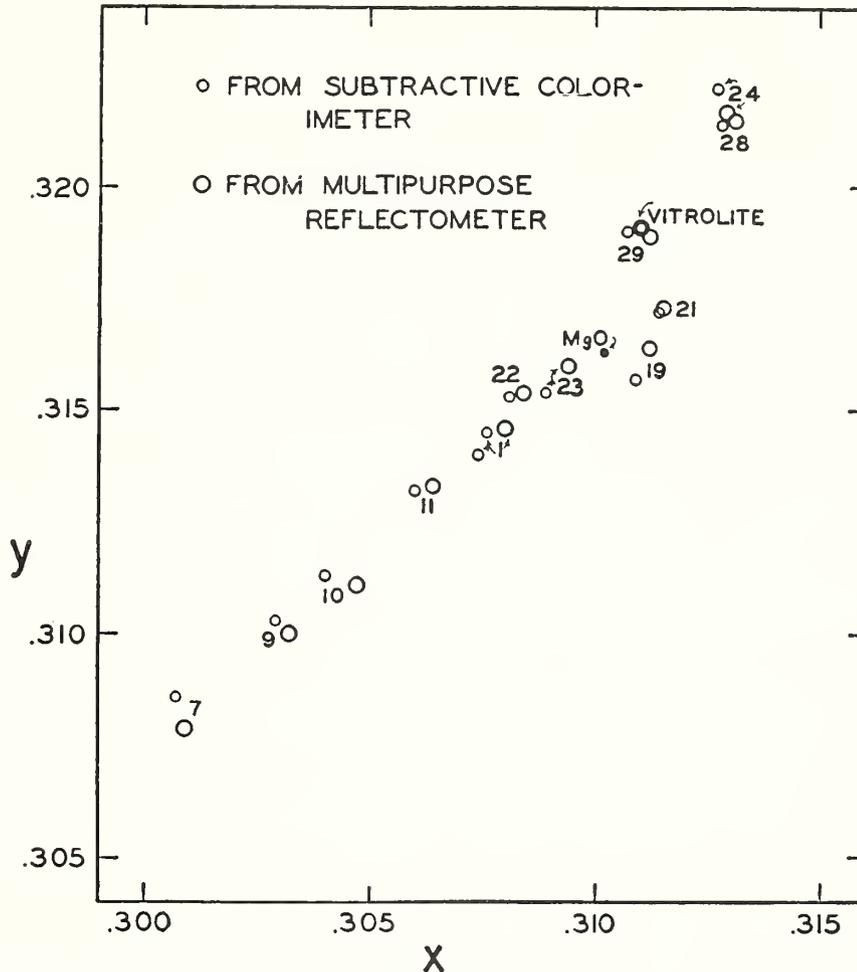


FIGURE 7.—Part of (x,y)-diagram (outlined by rectangle in fig. 2, above) showing x and y of a number of white porcelain-enameled plaques measured both with the visual subtractive colorimeter [29] and with the multipurpose reflectometer [18].

TABLE 8.—Changes in measured chromaticity difference which would result from the substitution of one photocell for another of the same type, but of different spectral response

Specimens compared	Measured size of the chromaticity difference (with cell 55) $\sqrt{\Delta\alpha^2 + \Delta\beta^2}$	Change in the measured chromaticity difference with substitution of cell	
		Actual	Relative to size of difference
Munsell BPB 8/2 and MgO.....	0.0261	0.0016	% 6
BG 6/4 and BG 7/4.....	.0136	.0004	3
Yellow Y1 and yellow Y2.....	.00224	.00035	(16)
Gray G1 and Gray G2.....	.0184	.0024	(13)

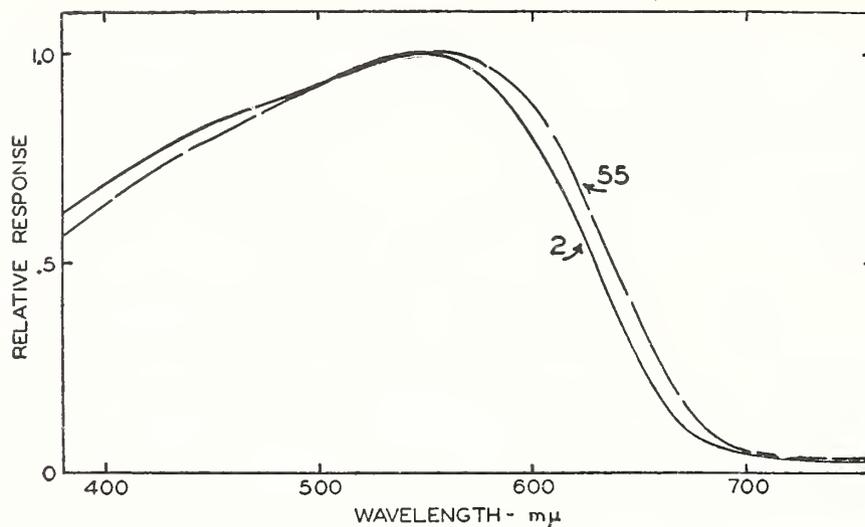


FIGURE 8.—Relative spectral responses of GE cells No. 55 and No. 2 used to demonstrate change in measured chromaticity which may result from change of the photocell in the instrument.

3. FLUORESCENCE ERRORS

The Institute of Paper Chemistry has shown [20, p. TS294] that fluorescence of a sample may cause error in any colorimetric instrument in which the energy striking the sample is spectrally different from that ordinarily used to observe the sample visually. A fluorescent sample has the power to change the spectral character of part of the energy striking it. In any instrument in which the incident energy does not possess the same spectral composition as the ordinary noninstrument illuminant, the energy leaving the sample may be altered in a manner different from that in the noninstrument situation.

If, as in the multipurpose reflectometer, the energy is passed through a filter before incidence on the sample, fluorescence may cause the photocell exposed to the sample to receive energy of wavelengths wholly excluded by the filter and therefore not supposed to be present for the particular measurements.

To illustrate the fluorescence error, the author selected a piece of phenol-tormaldehyde resin found by Gibson and Keegan [12] to give a spectral curve significantly in error because of fluorescence when measured with the GE recording spectrophotometer. These authors showed that this pink plastic gave a strong reddish fluorescence when illuminated by the yellow-green part of the spectrum. On visual inspection, the pink plastic appeared red relative to Munsell paper RYR 5/6. This visual estimate, which was substantiated by measurement with the Judd subtractive colorimeter, is properly represented by the black dots in figure 9.

Measurements with the multipurpose instrument, represented by the open circles in figure 9, erroneously indicate, however, that the resin would be perceived as greener and weaker in color than the Munsell paper. The error, when evaluated by eq 13, amounted to 14 NBS units of color difference and is about half the fluorescence error found by Gibson and Keegan when they measured the same sample with the GE spectrophotometer.

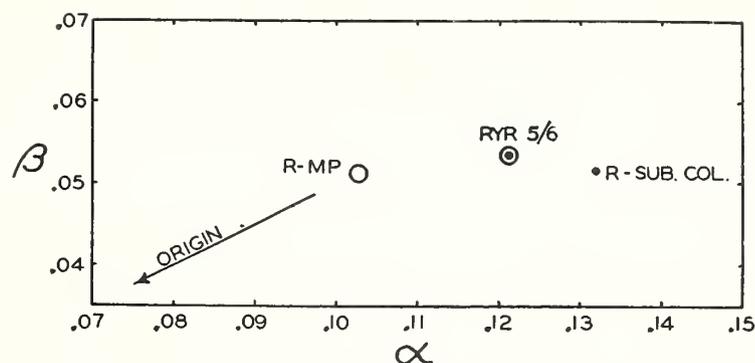


FIGURE 9.—Error in photoelectrically measured chromaticity due to fluorescence of the resin specimen, *R*.

When compared in the subtractive colorimeter [29] with Munsell paper RYR 5/6, the resin is properly assigned the chromaticity represented by the black dot, rather than the erroneous chromaticity assigned by the multipurpose reflectometer [18] and represented by the open circle.

4. SHORT CUTS IN COMPUTATION WHICH PRODUCE ONLY SMALL ERRORS

There are several short cuts which at times may be used to reduce the labor of computing tristimulus quantities from settings with the multipurpose reflectometer. The shortened procedures result in errors which diminish with the computed color difference between the specimens compared. With many small color differences, therefore, the short-cut computational errors are smaller than the errors introduced by lack of precision and are therefore unimportant. Even larger errors from this cause are unimportant if they are appreciably less than the maximum error permitted for a successful solution of the problem.

As an example, short-cut errors are seldom serious when one is following the day-to-day change of the color of a paint or textile material due to fading or other cause. The highest accuracy is not necessary for such measurements so long as the day-to-day reproducibility is fairly good. The short-cut computational errors have negligible effect on this reproducibility.

(a) OMITTING SCALE CORRECTIONS

In practice, the apparent-reflectance scale supplied with a multipurpose reflectometer is not exact. Correction curves, which are found by calibration after the instrument is completed, are necessary to obtain accurate values with the device [18, p. 601; 18a, p. 548]. The corrections do not change value rapidly with the scale reading for most parts of the scales employed. Therefore the omission of scale corrections usually produces only small changes in the computed color differences between samples of similar color. In all but one of the examples of tristimulus color measurement reproduced below, in section VIII, the scale corrections were omitted.

(b) NEGLECT OF THE DIFFERENCE BETWEEN THE KNOWN *A*, *G*, AND *B* VALUES FOR THE STANDARD AND THE APPROXIMATE VALUES READ DIRECTLY FROM THE INSTRUMENT

Because of the usual drift of instrument readings with time, the difficulty of making minute adjustments of photometric balance, and

the frequent necessity of correcting readings for scale errors, the readings of a photoelectric tristimulus instrument seldom give accurate values of A , G , and B directly. Instead, values of A , values of G , and values of B each relative to those of the standard are obtained from instrument settings, after the effect of drift is balanced out in the manner illustrated in examples 1, 3, 5, and 6, below. Thus if one exposes a standard and two samples for which the accurate tristimulus values are

$$\begin{aligned} &A_{\text{std}}, G_{\text{std}}, B_{\text{std}} \\ &A_1, G_1, B_1 \\ &A_2, G_2, B_2, \end{aligned}$$

he actually obtains from the corrected settings

$$\begin{aligned} &k_a A_{\text{std}}, k_g G_{\text{std}}, k_b B_{\text{std}} \\ &k_a A_1, k_g G_1, k_b B_1 \\ &k_a A_2, k_g G_2, k_b B_2. \end{aligned}$$

For many cases in which the highest accuracy is not required, it is not worthwhile to evaluate k_a , k_g , and k_b ; their departures from unity are found to be negligible. In examples 3 and 4, section VIII, use was made of this short-cut B .

One might suppose that a simple additive method could be used to compute accurate tristimulus values from instrument readings. If such a procedure were possible, one could take settings on a standard and sample, and from these settings compute values of x and y . From the computed and known values of x and y for the standard, the supposed additive corrections, Δx and Δy , would be found. That such corrections applied additively are inexact, however, may easily be shown. An "uncorrected" value of x for the standard would be computed from the readings on it by:

$$x_{\text{std}(u)} = \frac{0.80k_a A_{\text{std}} + 0.18k_b B_{\text{std}}}{0.80k_a A_{\text{std}} + k_g G_{\text{std}} + 1.36k_b B_{\text{std}}},$$

whereas the true value of x would be

$$x_{\text{std}} = \frac{0.80A_{\text{std}} + 0.18B_{\text{std}}}{0.80A_{\text{std}} + G_{\text{std}} + 1.36B_{\text{std}}}.$$

It will be seen that there is no simple function of $x_{\text{std}(u)}$ and x_{std} by which the factors k_a , k_g , and k_b can be eliminated.

(c) USING 0.5 AND 0.1 INSTEAD OF THE ACCURATE VALUES FOR THE NEUTRAL-FILTER TRANSMISSION FACTORS

To extend its photometric range, the multipurpose reflectometer makes use of two neutral filters transmitting approximately 0.5 and 0.1, respectively, of the normally incident energy. Actually the transmissions of the two filters used in the author's instrument are

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not 0.5 and 0.1, nor are the filters ideally neutral. Instead the transmissions are as follows:

Nominal transmissions of "neutral" filters	Exact transmissions of "neutral" filters		
	For amber tristimulus filter	For green tristimulus filter (giving luminosity function)	For blue tristimulus filter
0.5	0.480	0.479	0.427
1	.088	.086	.065

When the color differences between the specimens being compared are very small, or when accuracy is not needed, use of 0.5 and 0.1 instead of the proper filter factors is frequently permissible. It should be obvious, however, that these approximate factors should not be used for the computations if the neutral filter employed in conjunction with any one of the tristimulus filters was changed during the settings on the group of specimens. Example 4, section VIII, illustrates this third short cut.

(d) AMOUNTS OF ERROR FROM USING SHORT-CUT METHODS

To give an idea of the sizes of the errors which would be found in actual practice, instrument settings for six typical color differences were converted to amounts of color difference, first by the accurate computational procedure and then by using the short cuts. Two white, two delphinium-blue, and two international-orange pairs of samples were chosen, so that for each color there was one difference of about 1.5 NBS units and one of about 7 units. For each color, pairs of samples were sought which seemed likely to give the most serious errors from the use of the short-cut methods. For these pairs, amounts of color difference were computed five times; first they were computed accurately, then each of the three short cuts was used separately, and finally they were used together.

TABLE 9.—Percentage errors in computed values of color difference from using the suggested short cuts

Short cut	Errors in percentage of total difference—					
	For two pairs of white samples		For two pairs of blue samples		For two pairs of orange samples	
	$\Delta E=1.8$	$\Delta E=6.6$	$\Delta E=1.0$	$\Delta E=7.6$	$\Delta E=1.4$	$\Delta E=6.0$
Scale corrections omitted.....	0.6	1.1	0	4.1	4.2	4.8
Use of instrument values of A, G, and B, having only relatively correct magnitudes.....	0	0	1.0	2.9	26.6	6.3
Use of 0.5 and 0.1 instead of true neutral-filter factors.....			3.9	3.6	7.7	1.0
Combination of above three short cuts.....	0.6	0.3	0	8.7	27.3	4.8

The errors are given in table 9 as percentages of the accurately computed differences. The size of each error depends upon the nature of the color difference involved as well as upon the spectral selectivity

of the samples, and hence some of the differences are surprisingly small. In general, it will be seen that the errors become larger as the samples become more strongly colored. It will also be seen that the errors from the use of more than one short cut are not always additive. By comparison of the percentage errors in table 9 with those in table 7, it will be seen that most of these short-cut errors are smaller than the errors from spectral inaccuracy of the source-filter-photocell combinations. For this reason the short cuts can be recommended for use in many photoelectric tristimulus comparisons of color in which the highest accuracy possible is not needed.

VII. THE RELATION OF PERCEPTUAL IMPRESSIONS AND MEASURED VALUES OF COLOR

Because the ICI standard observer was created from the color matches made by a number of normal observers [13, 51], the numerical description of a color difference in the ICI or a related color system will, if possible interfering circumstances are eliminated, indicate the character of the color difference perceived and described by the normal observer. With some of the methods of measuring color suggested in the present paper, however, numbers are given which, in addition to indicating the character of the difference, are intended to designate quantitatively the perceptual impression of the difference. Unfortunately there is at present little information on the success with which quantitative perceptual impressions are duplicated by these photoelectric tristimulus measurements. One bit of information may be noted. In a study of several methods for measuring color difference, Balinkin [1] compared the photoelectrically measured values of ΔE for 10 pairs of green porcelain tiles with the averages of the visual estimates of the same differences by 60 observers. He found fairly good agreement between measured and estimated values, with a correlation according to ranks of 0.86.

A number of the circumstances which may disturb the correspondence between visual estimates and photoelectric determinations of color and color difference are well known. Some of these circumstances relate to imperfections in the measuring method, others to imperfections in the observing situation. There follows a two-part list in which a number of the circumstances are given. It will be seen that each of those listed under "imperfections in the measuring method" was discussed above.

CIRCUMSTANCES WHICH MAY INTERFERE WITH CORRELATION
BETWEEN VISUALLY ESTIMATED AND PHOTOELECTRICALLY
MEASURED VALUES OF COLOR AND COLOR DIFFERENCE

Imperfections in the Measuring Method:

- Error from spectral inaccuracy of source-filter-photocell combinations.
- Error from fluorescence of sample.
- Error from photometric inaccuracy of measuring instrument.
- Failure of the empirically chosen scales for chromaticity, surface-color difference, and lightness to represent appropriate scales for these quantities.

Imperfections in the Observing Situation:

- Spectral difference between illuminant used for observations and ICI illuminant *C*.
- Angular difference between directions of illuminating and viewing used for the visual observations and those used for the measurements. (This factor frequently affects the correlation if appreciable specularly reflected light is present in one case, but is eliminated in the other.)
- Differences in texture and shape of the specimens being compared.
- Nonuniformity of color from point to point on some of the specimens. (For instrument measurements of pairs of specimens, central areas are usually used; for visual comparisons, the colors of the immediately adjacent areas of the specimens have the greatest influence on estimated color difference.)
- Failure to take proper account in the measured values of the character of the dividing line, separating pattern, or background used during visual observation.
- Abnormality of the observer, either temporary as from fatigue, or permanent as with an observer having abnormal color vision.

VIII. EXAMPLES

To demonstrate uses of the photoelectric tristimulus method, and to illustrate forms for recording the corresponding data, six examples have been prepared. These examples represent multipurpose reflectometer measurements made at the National Bureau of Standards for purposes which have been identified on each of the forms which is shown. All of the steps leading from the initially recorded settings to the desired values for color have been duplicated. The number of settings taken on each sample and the short cuts permitted in the reduction of the data were decided in each case by a consideration of the needs for precision and accuracy of the problem at hand. Whenever two readings were made with each filter for every specimen, the order of readings was: Standard, No. 1, No. 2, No. 3, * * * No. 3, No. 2, No. 1, Standard. This symmetrical order, which was used to eliminate some of the error of drift, was mentioned above. The equations used in the following examples are given in table 3 and in the text above.

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Example 1.—Measurement of the ICI values, x, y, z, and Y of four white painted plaques (see eq 5 and 6, and fig. 10.)

Purpose—To test samples for compliance with color requirements given in Federal Specification TT-P-23a [8], namely,

	<i>x</i>	<i>y</i>	<i>z</i>	<i>Y</i>
Minimum-----			0.355	0.85
Maximum-----	0.324	0.331		

Computational short cut—Scale corrections omitted.

Working standard—vitrolite No. 1 (*A*=0.906, *G*=0.909, *B*=0.890).

Specimen	Standard	No. 1	No. 2	No. 3	No. 4
Blue settings-----	{ 0.8985	0.8480	0.7900	0.7810	0.7080
	{ .8955	.8450	.7880	.7790	.7070
Mean-----	.8970	.8465	.7890	.7800	.7075
<i>B</i> =mean × $\frac{0.890}{0.8970}$ -----		.8398	.7828	.7739	.7020

Amber settings-----	{ 0.9055	0.8785	0.8720	0.8420	0.8530
	{ .9030	.8770	.8680	.8400	.8520
Mean-----	.9042	.8778	.8700	.8410	.8525
<i>A</i> =mean × $\frac{0.906}{0.9042}$ -----		.8795	.8717	.8427	.8542

Green settings-----	{ 0.9100	0.8770	0.8620	0.8340	0.8320
	{ .9070	.8740	.8585	.8320	.8310
Mean-----	.9085	.8755	.8602	.8330	.8315
<i>Y</i> ÷ <i>G</i> =mean × $\frac{0.909}{0.9085}$ -----		.8760	.8607	.8335	.8320

0.18 <i>B</i> -----		0.1512	0.1409	0.1393	0.1264
1.18 <i>B</i> -----		.9910	.9237	.9132	.8284
0.80 <i>A</i> + 0.18 <i>B</i> -----		.8548	.8383	.8135	.8098
Denominator (<i>G</i> + 0.80 <i>A</i> + 1.36 <i>B</i>)-----		2.7218	2.6227	2.5602	2.4702

$x \doteq \frac{0.80 A + 0.18 B}{\text{denominator}}$ -----		0.3141	0.3196	0.3177	0.3278

$y \doteq G/\text{denominator}$ -----		.3218	.3282	.3256	.3368

$z \doteq 1.18B/\text{denominator}$ -----		.3641	.3522	.3567	.3354

Result of test-----		Pass	Fail (<i>z</i>)	Fail (<i>Y</i>)	Fail (all)

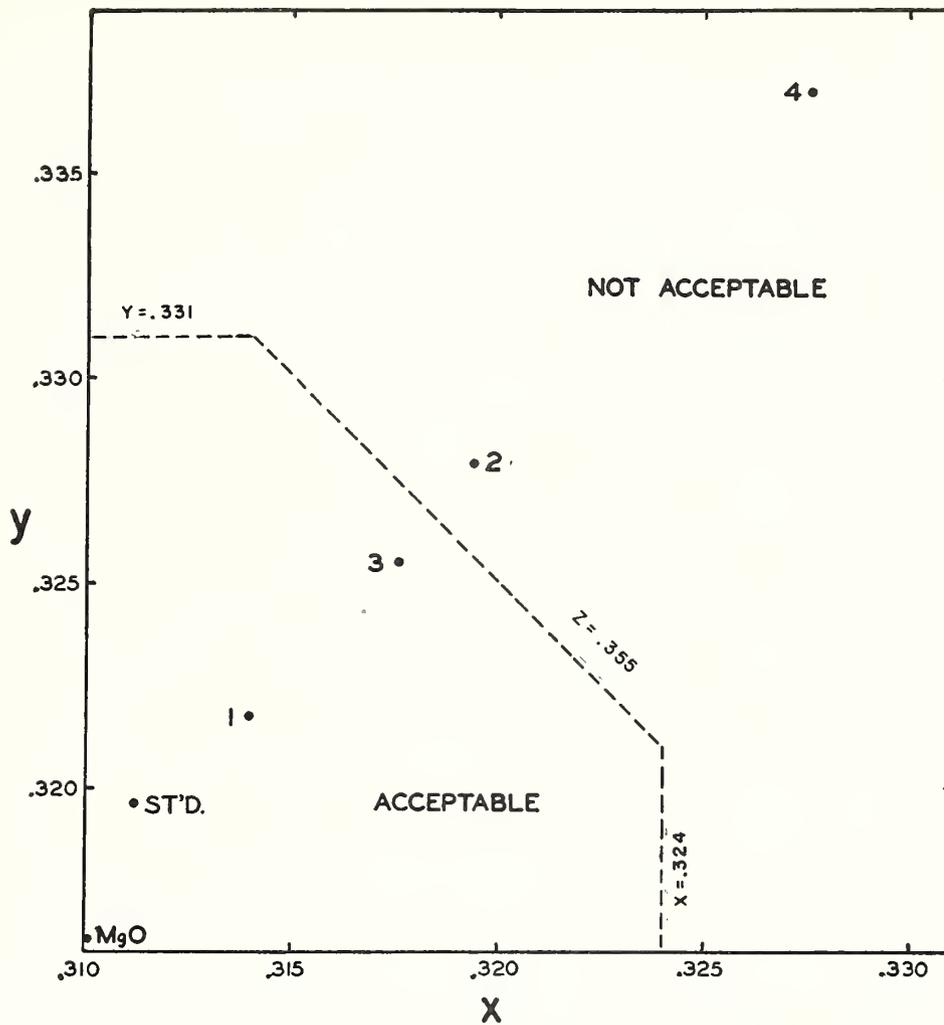


FIGURE 10.—Part of the (x,y) -diagram showing chromaticities acceptable and chromaticities not acceptable according to Federal Specification TT-P-23a for white casein paints.

The plotted points show values obtained with actual samples.

Circulars of the National Bureau of Standards

Example 2.—Measurement of the change of α and β of three painted plaques with exposure (see eq 8 and fig. 11)

Purpose—To follow the chromaticity changes of panels of the same paint exposed out of doors and in two machines which accelerate the changes produced by weathering.

Note—Only one of the many sets of computations used to obtain values of α and β is reproduced below.

Computational short cut—Scale corrections omitted.

Working standard—pink plaque ($A=0.7058$, $G=0.606$, $B=0.4714$).

Specimen	Standard	Roof (4 days)	Machine 1 (43 hours)	Machine 2 (41 hours)
Blue settings.....	{ 0. 4995 . 501 . 5002	0. 300	0. 685	0. 597
Mean.....				
$B = \text{setting} \times \frac{0.4714}{0.5002}$ 2827	. 6453	. 5626
Amber settings.....	{ . 704 . 704 . 704	. 510	. 7075	. 675
Mean.....				
$A = \text{setting} \times \frac{0.7058}{0.704}$ 5113	. 7093	. 6767
Green settings.....	{ . 620 . 619 . 6195	. 424	. 708	. 656
Mean.....				
$G = \text{setting} \times \frac{0.606}{0.6195}$ 4148	. 6926	. 6417
$A - G$		+. 0965	+. 0167	+. 0350
$G - B$		+. 1321	+. 0473	+. 0791
$0.4 (G - B)$		+. 0528	+. 0189	+. 0316
$B + A + 2G = \text{denominator}$		1. 6236	2. 7398	2. 5227
$\alpha \doteq (A - G) / \text{denominator}$		+0. 0594	+0. 0061	+0. 0139
$\beta \doteq 0.4(G - B) / \text{denominator}$		+. 0325	+. 0069	+. 0125

One fact is worthy of comment in connection with the changes of chromaticity accompanying fading of the paint illustrated in figure 11. At the start of exposure, a change in the paint from moderate pink to weak orange took place. This pronounced hue change is unusual in

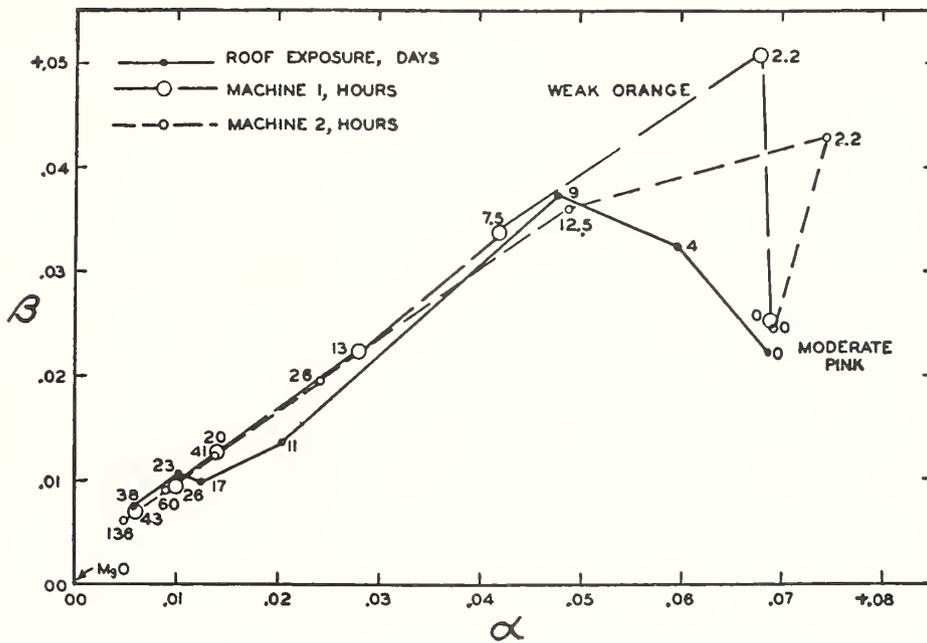


FIGURE 11.—Part of the (α, β) -diagram used to show the changes of chromaticity of a pink-colored paint with exposure outdoors and in two machines designed to accelerate the changes produced in weathering.

fading; paints, textile, and other materials are usually perceived to become merely weaker in color as the result of exposure to light, heat, and humidity. The usual curve of chromaticity change with time of exposure is therefore merely a curve directed toward the origin of the (α, β) -diagram, similar to the curves of figure 11 beyond the points showing the initial hue change.

Circulars of the National Bureau of Standards

Example 3.—Measurement of the amount of color difference, ΔE , between a standard and each of four similarly colored brown automobile pile fabrics (see eq 13).

Purpose—To find which fabrics comply with the requirement that they differ from the standard by no more than one NBS unit of color difference.

Computational short cut—No calibrated standard used.

$k_1=100, f_o=1.00.$

Fabric	Standard	No. 1	No. 2	No. 3	No. 4
Blue settings (with 0.427 filter) {	0. 1700	0. 1744	0. 1620	0. 1690	0. 2024
Mean (M)-----	. 1688	. 1738	. 1618	. 1686	. 2018
Scale correction-----	. 1694	. 1741	. 1619	. 1688	. 2021
Corrected M-----	-. 0139	-. 0138	-. 0138	-. 0136	-. 0121
B=Above times .427-----	. 1555	. 1603	. 1481	. 1552	. 1900
	. 0664	. 0684	. 0632	. 0663	. 0811
Amber settings (with 0.480 filter) {	. 2098	. 2144	. 2022	. 2042	. 2524
Mean (M)-----	. 2082	. 2142	. 2018	. 2036	. 2520
Scale correction-----	. 2090	. 2143	. 2020	. 2039	. 2522
Corrected M-----	-. 0120	-. 0120	-. 0120	-. 0120	-. 0113
A=above times .480-----	. 1970	. 2023	. 1900	. 1919	. 2409
	. 0946	. 0971	. 0912	. 0921	. 1156
Green settings (with 0.479 filter) {	. 1916	. 1946	. 1848	. 1882	. 2300
Mean (M)-----	. 1900	. 1944	. 1844	. 1876	. 2302
Scale correction-----	. 1908	. 1945	. 1846	. 1879	. 2301
Corrected M-----	-. 0127	-. 0126	-. 0130	-. 0129	-. 0122
Y=G=Above times .479-----	. 1781	. 1819	. 1716	. 1750	. 2179
	. 0853	. 0871	. 0822	. 0838	. 1044
A-G-----	+. 0093	+. 0100	+. 0090	+. 0083	+. 0112
G-B-----	+. 0189	+. 0187	+. 0190	+. 0175	+. 0233
0.4 (G-B)-----	+. 0076	+. 0075	+. 0076	+. 0070	+. 0093
Denominator=B+A+2G-----	. 3316	. 3397	. 3188	. 3260	. 4055
$\alpha \doteq (A-G)/\text{denominator}$ -----	+. 0280	+. 0294	+. 0282	+. 0255	+. 0276
$\beta \doteq 0.4(G-B)/\text{denominator}$ -----	+. 0229	+. 0221	+. 0238	+. 0215	+. 0229
$\Delta\alpha = \alpha - \alpha_{std}$ -----		+. 0014	+. 0002	-. 0025	-. 0004
$\Delta\beta = \beta - \beta_{std}$ -----		-. 0008	+. 0009	-. 0014	. 0000
$\sqrt{\Delta\alpha^2 + \Delta\beta^2} \cdot 10^2$ -----		. 16	. 09	. 29	. 04
$Y^{1/2}$ -----	. 292	. 295	. 287	. 289	. 323
$7(Y^{1/4})^*$ -----	3. 78				3. 98
$\Delta C = 7(Y^{1/4})\sqrt{\Delta\alpha^2 + \Delta\beta^2} \cdot 10^2$ -----		. 60	. 34	1. 10	0. 16
$\Delta Y^{1/2}$ -----		+. 003	-. 005	-0. 003	+. 031
$\Delta L = k_1(\Delta Y^{1/2})$ -----		. 3	. 5	. 3	3. 1
$\Delta E/f_o = \sqrt{\Delta C^2 + \Delta L^2}$ -----		. 67	. 60	1. 14	3. 11
ΔE -----		0. 7	0. 6	1. 1	3. 1

*Compute $7(Y^{1/4})$ for standard and for any samples for which the value of Y differs from that of the standard by more than 10 percent. For the latter samples, use the mean of the two values of $7(Y^{1/4})$; for others, use the value for the standard.

Photoelectric Tristimulus Colorimetry

Example 4.—Measurement of the hue-difference estimate, $\Delta H'$, the saturation-difference estimate, $\Delta S'$, and the lightness-difference estimate, $\Delta L'$, between an actual earth sample and two modified earths (see eq 17).

Purpose—To find how the modified earths differ in color from the actual earth.
 Computational short cuts—Scale corrections omitted, no calibrated standard used, and the factor 0.5 used instead of the actual transmissions of the neutral filter.

$k_1 = 100, f_p = 1.00.$

Specimen	Earth mixture	Modification No. 1	Modification No. 2
Blue settings	0. 295	0. 260	0. 370
$B = \text{Above times } 0.5$. 147 ₅	. 130	. 185
Amber settings	. 496	. 446	. 575
$A = \text{Above times } 0.5$. 248	. 223	. 287 ₅
Green settings	. 428	. 386	. 508
$Y \doteq G = \text{Above times } 0.5$. 214	. 193	. 254
$L = Y^{\frac{1}{2}} \doteq G^{\frac{1}{2}}$. 463	. 439	. 504
$\Delta L' = k_1 \Delta(Y^{\frac{1}{2}})$		-2. 4	+4. 1
$A - G$ (note sign)	+ . 034	+ . 030	+ . 0335
$G - B$ (note sign)	+ . 0665	+ . 063	+ . 069
$0.4 (G - B)$	+ . 0266	+ . 0252	+ . 0276
Denominator = $B + A + 2G$. 8235	. 739	. 9805
$a \doteq (A - G)/\text{denominator}$	+ . 0413	+ . 0406	+ . 0342
$\beta \doteq 0.4(G - B)/\text{denominator}$	+ . 0323	+ . 0341	+ . 0281
$\sqrt{a^2 + \beta^2}$. 0525	. 0528	. 0443
$\Delta(\sqrt{a^2 + \beta^2})$		+ . 0003	- . 0082
$700 Y^{\frac{1}{2}} *$		470.	487.
$\Delta S' = 700 Y^{\frac{1}{2}} \Delta(\sqrt{a^2 + \beta^2})$		+0. 14	-3. 99
β/a	+ . 782	+ . 840	+ . 822
$\phi = \text{angle whose tan is } \beta/a$	38. 03°	40. 03°	39. 42°
$\Delta\phi$		+2. 00°	+1. 39°
$12.2 Y^{\frac{1}{2}} *$		8. 19	8. 48'
$12.2 Y^{\frac{1}{2}} \sqrt{a^2 + \beta^2} *$		0. 431	0. 411
$\Delta H' = 12.2 Y^{\frac{1}{2}} \sqrt{a^2 + \beta^2} \cdot \Delta\phi$		+0. 86	+0. 57
Color of modification relative to that of actual earth, according to the measurements.		Darker, greener, slightly stronger.	Lighter, weaker, slightly greener.

*Values which are average for the 2 specimens being compared should be entered in those rows marked by an asterisk.

Circulars of the National Bureau of Standards

Example 5.—Measurement of the whiteness, W, of one new and two laundered sheeting specimens. (See eq 19)

Purpose—To measure the effect of repeated laundering on the whiteness of new sheeting material.

Computational short cut—Scale corrections omitted.

$k_1=20, f_o=1.00$.

Working standard—vitrolite No. 1 ($A=0.906, G=0.909, B=0.890$).

Specimen	Standard	New	1L	20L
Blue settings	{ 0.898	0.837	0.845	0.765
Mean	{ .892	.850	.852	.787
	{ .895	.8435	.8485	.776
$B = \text{mean} \times \frac{0.890}{0.895}$.8388	.8438	.7717

Amber settings	{ .9085	.892	.912	.826
Mean	{ .906	.904	.912	.836
	{ .9072	.898	.912	.831
$A = \text{mean} \times \frac{0.906}{0.9072}$.8968	.9108	.8299

Green settings	{ .9055	.884	.900	.812
Mean	{ .903	.894	.900	.824
	{ .9042	.889	.900	.818
$G = \text{mean} \times \frac{0.909}{0.9042}$.8937	.9048	.8223

$A - G$.0031	.0060	.0076
$2.5(A - G)$.0078	.0150	.0190
$G - B$.0549	.0610	.0506

$\alpha \doteq 2.5(A - G)/10G$.0009	.0017	.0023
$\beta \doteq (G - B)/10G$.0061	.0067	.0062

$\sqrt{\alpha^2 + \beta^2}$.0062	.0069	.0066
$W_c = 30\sqrt{\alpha^2 + \beta^2}$.186	.207	.198

$1.00 - G$.106	.095	.178
$W_L = (1.00 - G)/2$.053	.048	.089

$\sqrt{W_c^2 + W_L^2}$.193	.213	.217
$W = 1 - \sqrt{W_c^2 + W_L^2}$.807	.787	.783

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Example 6.—Measurement of the yellowness of four near-white porcelain-enameled specimens (eq 20):

Purpose—To find which of several enamel specimens are yellowish (+ values) and which are bluish (− values).

Computational short cut—scale corrections omitted.

Working standard—vitrolite No. 1 ($A=0.906$, $G=0.909$, $B=0.890$).

Plaque No.	Standard	1	10	22	24
Blue settings.....	{ 0. 8955	0. 7540	0. 7760	0. 8238	0. 7804
Mean.....	. 8920	. 7490	. 7716	. 8222	. 7780
Mean.....	. 8938	. 7515	. 7738	. 8230	. 7792
$B = \text{mean} \times \frac{0.890}{0.8938}$ 7483	. 7705	. 8195	. 7759

Amber settings.....	{ . 8945	. 7276	. 7236	. 7990	. 7980
Mean.....	. 8936	. 7222	. 7176	. 7962	. 7962
Mean.....	. 8940	. 7249	. 7206	. 7976	. 7971
$A = \text{mean} \times \frac{0.906}{0.8940}$ 7346	. 7303	. 8083	. 8078

Green settings.....	{ . 9037	. 7370	. 7364	. 8094	. 8046
Mean.....	. 9030	. 7316	. 7324	. 8074	. 8030
Mean.....	. 9034	. 7343	. 7344	. 8084	. 8038
$G = \text{mean} \times \frac{0.909}{0.9034}$ 7389	. 7390	. 8134	. 8088

$A - B$		−. 0137	−. 0402	−. 0112	+ . 0319
Yellowness = $(A - B)/G$ (eq 20).....		−. 0185	−. 0544	−. 0138	+ . 0394

IX. SUMMARY

Photoelectric tristimulus measurements are well suited for comparisons of the colors of spectrally similar specimens. In commerce there seem to be many situations in which such measurements may prove to be useful: (1) in the day-to-day control of the colors of materials in production, (2) in obtaining compliance of materials with color-tolerance requirements, (3) in making modifications of color to produce matches, and (4) in the measurements of color changes accompanying fading. For problems of this general character, it is believed that photoelectric tristimulus measurements will prove to be useful.

The advantages and disadvantages of photoelectric tristimulus colorimetry have been summarized as follows:

ADVANTAGES

- Small number of settings required for each sample.
- Small number of computational steps required to obtain x , y , and Y , or α , β , and Y .
- Simplicity and inexpensiveness of apparatus.
- Opportunity to convert settings rapidly to numbers giving size and character of color difference perceived between samples.
- Correspondence between values of α , β , and Y , and positions of points representing colors in the uniformly spaced surface-color solid.

DISADVANTAGES

Errors resulting from the inaccurate spectral response of the source-filter-photocell combinations used.

The necessity for working standards which are spectrally similar to the samples measured.

The necessity for special precautions to attain the high precision required if the tristimulus method is to equal the eye in power to discriminate colors.

Failure of tristimulus measurements to provide the physical description of a color stimulus such as is given by a spectrophotometric curve.

The directness of the method and the small number of computational steps necessary to give numbers indicative of color recommend the method. Its inaccuracy because of failure of the source-filter-photocell combinations to be spectrally equivalent to the ICI standard observer is its most important defect. Nevertheless it can be reasonably expected that the use of photoelectric tristimulus colorimetry will increase as the combinations of elements used are improved in spectral character.

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A PRECISION APPARATUS FOR THE RAPID DETERMINATION OF INDICES OF REFRACTION AND DISPERSION BY IMMERSION

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ABSTRACT

A new immersion method for determining indices of refraction and ν values, employing the double-diaphragm method for securing oblique illumination, is described. The average error in the determination of indices of refraction based upon 144 measurements is 2×10^{-3} ; the maximum error is 5×10^{-5} . Complete measurements of the indices of refraction for the sodium D line and the hydrogen F and C lines may be made in approximately $1\frac{1}{2}$ hours, and from these measurements the ν values may be calculated with an average error of 0.1 and a maximum error of 0.8.

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I. INTRODUCTION

In the production of optical glass, careful control over the optical properties, especially the index of refraction (n_D) and the Abbe value (ν), of the product is essential. Most methods for measuring indices of refraction are sufficiently accurate for the control of the n_D value. The ν value, on the other hand, presents a special problem in index measurement. It is defined as $(n_D - 1)/(n_F - n_C)$, and in order to determine this value within certain specified limits, the index of refraction must be measured very accurately. Some typical tolerances which have to be met and the errors in the index measurements corresponding to these tolerances are shown in table 1.

TABLE 1.—Typical tolerances for n_D and ν values

Glass	Typical values and tolerances		Error corresponding to tolerance in ν
	n_D	ν	
Borosilicate crown.....	1.517 ± 0.001	64.5 ± 0.3	$\pm 2 \times 10^{-5}$
Light barium crown.....	1.572 ± 0.001	57.4 ± 0.3	$\pm 3 \times 10^{-5}$
Dense flint.....	1.649 ± 0.0015	33.8 ± 0.3	$\pm 9 \times 10^{-5}$

It is obvious from table 1 that the required accuracy of index measurements is determined mainly by the ν value tolerances. If the errors in index measurements are as great as those listed in column three, the ν value may be in error by the amount of the permissible tolerances. In order to be certain that the ν values are well within the required limits, the best spectrometer measurements¹ are necessary for final control of crown glasses, and are desirable for flint glasses. However, there are many useful routine checks and measurements that can be made on refractometers.

In normal times at the optical glass plant of the National Bureau of Standards, the problem of control was solved by making two independent sets of measurements. The immersion method of Faick and Finn² was used for immediate production control work. The final and more accurate values of optical constants were later measured on a spectrometer and a precision refractometer.

During wartime, manufacturers are called upon to make new glasses and a greater variety of them. This requires frequent changes in batch compositions and makes necessary more frequent and careful determination of index of refraction and dispersion. Under these conditions the above-mentioned immersion control method was deficient both as to speed and accuracy. The more accurate index measurements as they are generally made, on spectrometers or on carefully calibrated Pulfrich refractometers, require ground and pitch-polished samples. The time-consuming operations of grinding and polishing cause delay in the processing of the glass or in the revision of batch compositions, especially in glass plants that are not equipped with grinding and polishing facilities for the preparation of samples. Most immersion methods permit the use of rough, unprepared samples, but either do not yield sufficiently accurate results or lack the simplicity and speed desired.³ It was, therefore, felt desirable to develop an immersion method for rough samples which would have the required accuracy and permit speed and ease of manipulation.

II. METHOD OF MATCHING

One of the chief requirements of immersion refractometry is a sensitive method for ascertaining the match between the indices of the sample and the immersion liquid. Of the various methods that could be used for this, one embodying the principles of oblique illumination, well known to petrographers, seemed the most promising both for sensitivity and simplicity of operation. A system of oblique illumination, using double diaphragms, has been described by Saylor⁴ and shown to be superior to usual methods, at least when used on a microscope. Another double-diaphragm method, employing a telescope and a long focus lens as parts of the optical system, has been used by Cheshire⁵ for measuring the index of refraction of glass by immersion. This system is too cumbersome for rapid control work.

¹ H. L. Gurewitz and L. W. Tilton, *J. Research NBS* **32**, 39 (1944) RP1572.

² C. A. Faick and A. N. Finn, *BS J. Research* **6**, 993-1002 (1931) RP320.

This method consists essentially in matching the index of refraction of the glass with that of a suitable liquid in which the sample is immersed and then measuring the index of the liquid. The apparatus consists of a telescope and a grid, illuminated by a sodium light, which are placed on opposite sides of a water-cooled tank containing the immersion liquid and sample. The match between the glass and the liquid is obtained by altering the index of the liquid until no distortion of the grid pattern is observed through the telescope. Accuracies within $\pm 3 \times 10^{-4}$ are obtainable with this method.

³ For a discussion of immersion refractometry, see Glazebrook's *Dictionary of Applied Physics* **4**, 130 (The Macmillan Co., New York, N. Y., 1923).

⁴ C. P. Saylor, *J. Research NBS* **15**, 277-294 (1935) RP829.

⁵ R. W. Cheshire, *Phil. Mag.* (6) **32**, 409-420 (1916).

Indices of Refraction by Immersion

Basically, the method finally chosen consists in inserting at the focus of a condensing lens (fig. 1, A) a diaphragm which shades out almost one-half of the beam of light. The emerging beam is then passed through the immersion liquid and again brought to a focus by means of a second converging lens. At this focus (fig. 1, B) a second

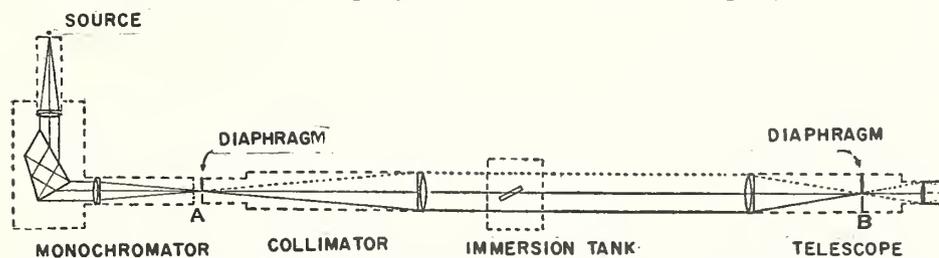


FIGURE 1.—Schematic drawing of the optical system for matching the indices of the glass and immersion liquid.

diaphragm is placed so that most of the remaining bright portion of the beam is shaded out. If a telescope is then inserted in the system, the field, as seen through the eyepiece, will appear dimly but uniformly lighted. Now, when a specimen is immersed in a liquid of the same index of refraction as that of the specimen, the light rays passing through the specimen will not be deviated and, since the second

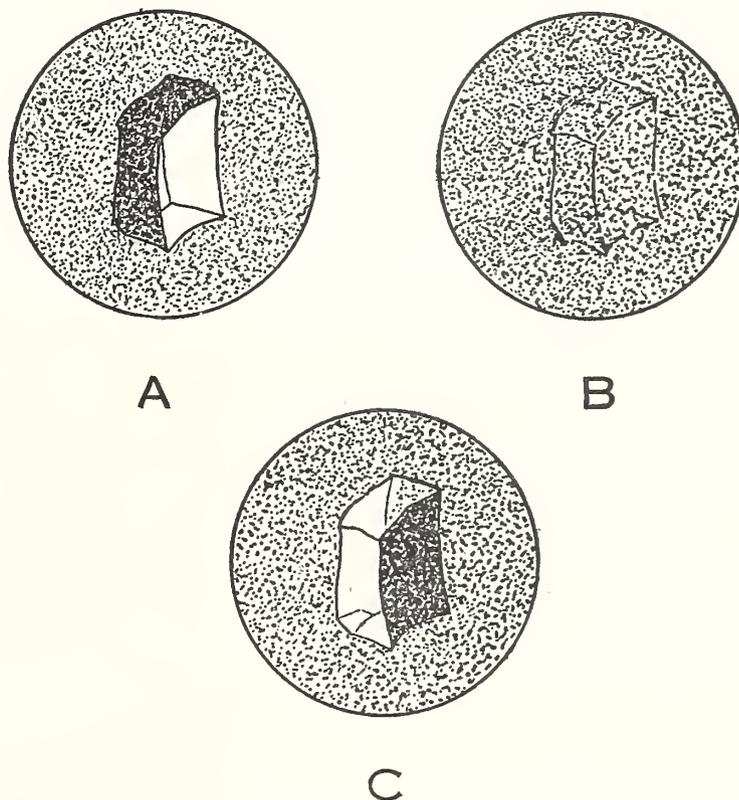


FIGURE 2.—Field through the eyepiece of the telescope—diaphragm on right side of the instrument.

A, When the index of the liquid is greater than the index of the sample; B, When the liquid and sample have the same index; C, When the index of the liquid is lower than that of the sample.

diaphragm takes out the bright portion of the beam, the sample will be practically invisible against the dimly lighted field seen through the eyepiece (fig. 2, B). However, if the match between the sample and liquid is altered, the path of light passing through the sample will be deviated so that the sample no longer blends with the field, but becomes light on one side and dark on the other. When the index of the immersion liquid is greater than that of the glass, the side of the sample (as seen through the telescope) that is on the same side as the diaphragms will be light (fig. 2, A); when the index of the liquid is lower, the opposite side of the sample will be light (fig. 2, C). By altering the index of refraction of the immersion liquid, which is a mixture of two liquids, one of a low and the other of a high refractive index, the match between the specimen and immersion medium can be easily established. After a match has been obtained, the index of refraction of the liquid is measured on a precision refractometer by comparison with a standard of approximately the same index.

III. APPARATUS

The apparatus (figs. 1 and 3) consists essentially of light sources, a monochromator with the exit slit removed, a collimator, an immersion tank, a telescope, a tested precision refractometer, and accessory equipment. Referring to figure 1, the light from the source is first passed through a monochromator,⁶ where a particular spectrum line is isolated and is brought to a focus at the end of the collimator where the first diaphragm is placed. This diaphragm is an adjustable knife-edge that covers part of a circular opening, replacing the usual collimator slit. The light then passes through the collimator, from which it emerges as a parallel beam (or a very slightly converging one) that passes through the tank and then enters the telescope. Near the focal plane, the second diaphragm, which is also adjustable, is located. The objective is selected so that in combination with the eyepiece it forms a telescope that can be focused on the sample in the immersion tank at a distance which is convenient for the operator. As an added convenience, the telescope is fitted with a right-angle prism and mounted in a vertical position.

The light sources used are a sodium-vapor lamp and a hydrogen-discharge tube. They are mounted on a sliding support to facilitate changing from one to the other (fig. 3).

The immersion tank is equipped with plate-glass windows and a water jacket for temperature control. The water jackets of this tank and of the refractometer are connected in parallel, and water from the same source is circulated through both. The accessory equipment includes a water reservoir and circulating pump, a motorized stirrer for mixing the immersion liquids, thermometers⁷ in the immersion tank and refractometer and a vacuum pump and a hydrogen generator for frequent refilling of the discharge tube.

⁶ By use of a monochromator, it was possible to avoid difficulties arising from the other lines in the sodium source, and from the continuous spectrum in the hydrogen tube. Filters like those used on the refractometer were found unsatisfactory on the matching instrument.

⁷ Mercury-in-glass type graduated to a tenth of a degree, permitting the estimation of hundredths of a degree. The thermometers should be carefully checked against each other, since the difference in temperature is the important factor.

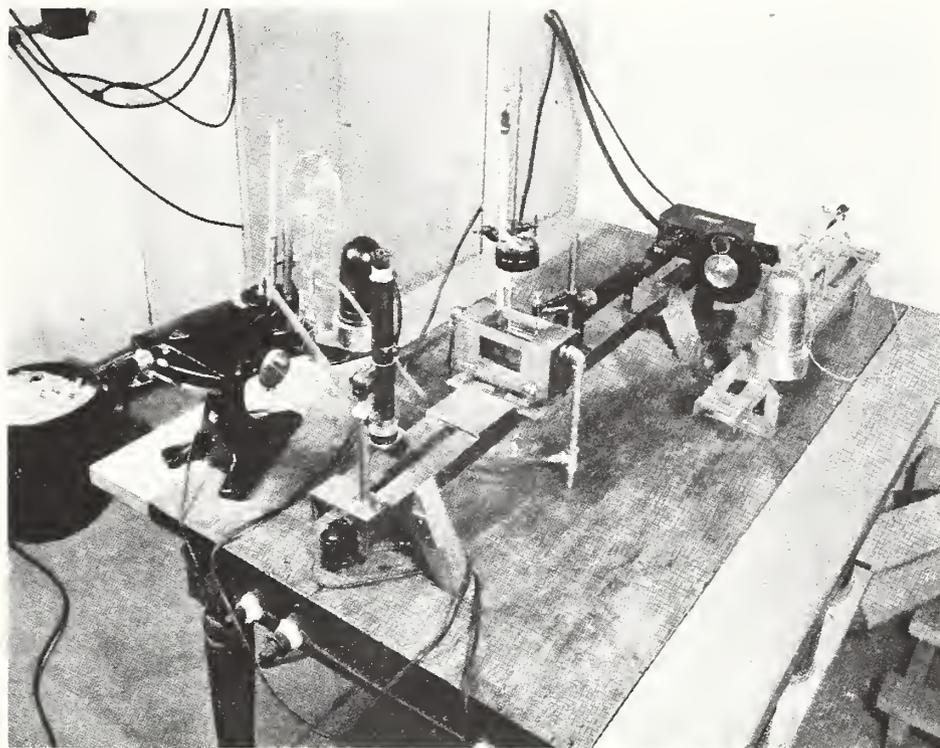


FIGURE 3.—*Complete immersion apparatus.*

IV. PROCEDURE

Six immersion liquids were prepared. The indices of refraction of these liquids were approximately 1.517, 1.572, 1.604, 1.617, 1.620, and 1.649, respectively. The first five liquids were mixtures of liquid petrolatum ($n_D=1.456$) and α -chloronaphthalene ($n_D=1.632$). The sixth liquid was a mixture of α -chloronaphthalene and α -bromonaphthalene ($n_D=1.656$). After isolating a particular spectrum line by means of the monochromator, the sample was immersed in the appropriate liquid, and the two were matched for index by adding small quantities of one of the pure liquids. When a good match had been obtained, the index of the immersion liquid was measured on the refractometer. This process was repeated four times for each sample. In each series of measurements, care was taken to approach the correct value from both a higher index immersion liquid and a lower one. Measurements of the temperature of the immersion liquid and of the refractometer prism block were made with a precision of $\pm 0.02^\circ \text{C}$. Corrections for very small temperature differences were applied by using an average change of -0.00035 in index per degree rise in temperature.

In order to obtain the highest possible accuracy in index measurements, a comparison method recommended by Tilton⁸, was used. By this method, instrumental errors, such as a shift in the zero setting, can be readily compensated. Essentially this method consists in checking the refractometer, before and after each series of immersion measurements, with a standard of known index approximately the same as the rough sample being measured and making the necessary corrections. This check was made for each type of glass and for each wavelength used, by measuring the standard as a Pulfrich slab on the refractometer. In several cases brought to the attention of the authors, considerable difference was found in the readings of refractometers when measuring solid and liquid samples of the same index. Should this be the case, an alternative procedure should be used in checking the refractometer. In the alternative procedure a liquid which in each case is matched with a known sample in index is used as the "standard" instead of the solid sample.

A series of 12 glass samples was tested to obtain the accuracy of the instrument. These samples were cut from prisms that had been previously measured on a spectrometer. The indices of refraction of these test pieces were known to the fifth decimal place. Since these samples were in the shape of rectangular prisms approximately 2 by 2 by 0.2 cm in size and had polished faces, and since the instrument was designed for measuring rough, irregularly shaped specimens, it was necessary to compare the accuracies of measurement obtainable with rough pieces and finished samples. This was done by measuring the indices of refraction of four rough pieces of glass and then grinding these pieces to the same shape as the standards and then remeasuring the indices. The results of this test show that any difference that does exist is within the error of measurement.

⁸ L. W. Tilton, *J. Opt. Soc. Am.* **32**, 371-381 (1942).

TABLE 2.—Comparison of measurements made on rough, irregularly shaped samples and on fine-ground, rectangular specimens

Index of refraction (n_D)			Index of refraction (n_D)		
Rough sample		Ground sample	Rough sample		Ground sample
SAMPLE I			SAMPLE III		
1.....	1.51580	1.51578	1.....	1.60179	1.60178
2.....	1.51582	1.51581	2.....	1.60182	1.60180
3.....	1.51578	1.51583	3.....	1.60180	1.60180
4.....	1.51585	1.51580	4.....	1.60184	1.60181
Average.....	1.51581	1.51581	Average.....	1.60181	1.60180
SAMPLE II			SAMPLE IV		
1.....	1.57187	1.57188	1.....	1.61960	1.61960
2.....	1.57189	1.57187	2.....	1.61961	1.61963
3.....	1.57190	1.57186	3.....	1.61963	1.61963
4.....	1.57188	1.57190	4.....	1.61962	1.61962
Average.....	1.57189	1.57188	Average.....	1.61962	1.61962

V. RESULTS

The values obtained in the test of the rough blanks are given in table 2. A total of 48 measurements were made on the 12 regular samples for each of the wavelengths, corresponding to the sodium *D* line, and hydrogen *F* and *C* lines. The results of these measurements are tabulated in table 3. Table 3 also shows the errors in the measured

TABLE 3.—Comparison of immersion determinations of index of refraction and dispersion with spectrometer values

Method	n_D	Error $\times 10^5$	n_F	Error $\times 10^5$	n_C	Error $\times 10^5$	μ	Error
SAMPLE I								
Spectrometer.....	1.51651	-----	1.52211	-----	1.51411	-----	64.6	-----
Immersion 1.....	1.51653	+2	1.52213	+2	1.51412	+1	64.6	0
Immersion 2.....	1.51653	+2	1.52209	-2	1.51413	+2	64.9	+0.3
Immersion 3.....	1.51650	-1	1.52210	-1	1.51410	-1	64.6	0
Immersion 4.....	1.51651	0	1.52208	-3	1.51412	+1	64.9	+0.3
Average.....	1.51652	1	1.52210	2	1.51412	1	64.8	0.2
SAMPLE II								
Spectrometer.....	1.51748	-----	1.52316	-----	1.51507	-----	64.0	-----
Immersion 1.....	1.51746	-2	1.52318	+2	1.51509	+2	64.0	0
Immersion 2.....	1.51747	-1	1.52316	0	1.51510	+3	64.2	+0.2
Immersion 3.....	1.51750	+2	1.52317	+1	1.51508	+1	64.0	0
Immersion 4.....	1.51746	-2	1.52313	-3	1.51508	+1	64.3	+0.3
Average.....	1.51747	2	1.52316	2	1.51509	2	64.1	0.1
SAMPLE III								
Spectrometer.....	1.57205	-----	1.57906	-----	1.56913	-----	57.6	-----
Immersion 1.....	1.57202	-3	1.57909	+3	1.56914	+1	57.5	-0.1
Immersion 2.....	1.57202	-3	1.57904	-2	1.56909	-4	57.5	-0.1
Immersion 3.....	1.57206	+1	1.57905	-1	1.56911	-2	57.6	0
Immersion 4.....	1.57206	+1	1.57906	0	1.56911	-2	57.5	-0.1
Average.....	1.57204	2	1.57906	2	1.56911	2	57.5	0.1

Indices of Refraction by Immersion

TABLE 3.—Comparison of immersion determinations of index of refraction and dispersion with spectrometer values—Continued

Determination	n_D	Error $\times 10^5$	n_F	Error $\times 10^5$	n_C	Error $\times 10^5$	ν	Error
SAMPLE IV								
Spectrometer.....	1.57151	-----	1.57850	-----	1.56859	-----	57.7	-----
Immersion 1.....	1.57150	-1	1.57849	-1	1.56858	-1	57.7	0
Immersion 2.....	1.57151	0	1.57853	+3	1.56860	+1	57.6	-0.1
Immersion 3.....	1.57154	+3	1.57847	-3	1.56861	+2	58.0	+0.3
Immersion 4.....	1.57151	0	1.57846	-4	1.56858	-1	57.8	+0.1
Average.....	1.57152	1	1.57849	3	1.56859	1	57.8	0.1
SAMPLE V								
Spectrometer.....	1.60428	-----	1.61419	-----	1.60027	-----	43.4	-----
Immersion 1.....	1.60431	+3	1.61416	-3	1.60025	-2	43.4	0
Immersion 2.....	1.60430	+2	1.61416	-3	1.60024	-3	43.4	0
Immersion 3.....	1.60431	+3	1.61420	+1	1.60023	-4	43.3	-0.1
Immersion 4.....	1.60428	0	1.61419	0	1.60028	+1	43.4	0
Average.....	1.60430	2	1.61418	2	1.60025	3	43.4	0
SAMPLE VI								
Spectrometer.....	1.60454	-----	1.61446	-----	1.60053	-----	43.4	-----
Immersion 1.....	1.60454	0	1.61450	+4	1.60056	+3	43.4	0
Immersion 2.....	1.60457	+3	1.61449	+3	1.60054	+1	43.3	-0.1
Immersion 3.....	1.60456	+2	1.61450	+4	1.60053	0	43.3	-0.1
Immersion 4.....	1.60454	0	1.61447	+1	1.60057	+4	43.5	+0.1
Average.....	1.60455	1	1.61449	3	1.60055	2	43.4	0.1
SAMPLE VII								
Spectrometer.....	1.61804	-----	1.63018	-----	1.61320	-----	36.4	-----
Immersion 1.....	1.61804	0	1.63020	+2	1.61322	+2	36.4	0
Immersion 2.....	1.61803	-1	1.63019	+1	1.61323	+3	36.4	0
Immersion 3.....	1.61801	-3	1.63019	+1	1.61324	+4	36.5	+0.1
Immersion 4.....	1.61806	+2	1.63017	-1	1.61325	+5	36.5	+0.1
Average.....	1.61804	2	1.63019	1	1.61324	3	36.5	0.1
SAMPLE VIII								
Spectrometer.....	1.61731	-----	1.62942	-----	1.61249	-----	36.5	-----
Immersion 1.....	1.61734	+3	1.62941	-1	1.61250	+1	36.5	0
Immersion 2.....	1.61729	-2	1.62943	+1	1.61252	+3	36.5	0
Immersion 3.....	1.61728	-3	1.62937	-5	1.61252	+3	36.6	+0.1
Immersion 4.....	1.61729	-2	1.62940	-2	1.61252	+3	36.6	+0.1
Average.....	1.61730	3	1.62940	2	1.61252	3	36.6	.1
SAMPLE IX								
Spectrometer.....	1.62024	-----	1.63248	-----	1.61536	-----	36.2	-----
Immersion 1.....	1.62023	-1	1.63249	+1	1.61536	0	36.2	0
Immersion 2.....	1.62024	0	1.63251	+3	1.61536	0	36.2	0
Immersion 3.....	1.62027	+3	1.63252	+4	1.61540	+4	36.2	0
Immersion 4.....	1.62027	+3	1.63246	-2	1.61539	+3	36.3	+0.1
Average.....	1.62025	2	1.63250	3	1.61538	2	36.2	0
SAMPLE X								
Spectrometer.....	1.62023	-----	1.63247	-----	1.61536	-----	36.2	-----
Immersion 1.....	1.62022	-1	1.63246	-1	1.61539	+3	36.3	+0.1
Immersion 2.....	1.62024	+1	1.63247	0	1.61535	-1	36.2	0
Immersion 3.....	1.62025	+2	1.63242	-5	1.61537	+1	36.4	+0.2
Immersion 4.....	1.62025	+2	1.63248	+1	1.61536	0	36.2	0
Average.....	1.62024	2	1.63246	2	1.61537	2	36.3	+0.1

TABLE 3.—Comparison of immersion determinations of index of refraction and dispersion with spectrometer values—Continued

Method	n_D	Error $\times 10^5$	n_P	Error $\times 10^5$	n_C	Error $\times 10^5$	μ	Error
SAMPLE XI								
Spectrometer.....	1.64918	-----	1.66296	-----	1.64373	-----	33.8	-----
Immersion 1.....	1.64918	0	1.66300	+4	1.64375	+2	33.7	-0.1
Immersion 2.....	1.64917	-1	1.66296	0	1.64370	-3	33.7	-1
Immersion 3.....	1.64915	-3	1.66296	0	1.64371	-2	33.7	-1
Immersion 4.....	1.64917	-1	1.66293	-3	1.64373	0	33.8	0
Average.....	1.64917	1	1.66296	2	1.64372	2	33.7	0.1
SAMPLE XII								
Spectrometer.....	1.64845	-----	1.66220	-----	1.64301	-----	33.8	-----
Immersion 1.....	1.64845	0	1.66215	-5	1.64301	0	33.9	+0.1
Immersion 2.....	1.64847	+2	1.66217	-3	1.64299	-2	33.8	0
Immersion 3.....	1.64842	-3	1.66218	-2	1.64296	-5	33.7	-1
Immersion 4.....	1.64845	0	1.66218	-2	1.64306	+5	33.9	+1
Average.....	1.64845	1	1.66217	3	1.64300	3	33.8	0.1

values as compared with the spectrometer values. An examination of these errors shows that the maximum error, or degree of accuracy, for any single measurement is $\pm 5 \times 10^{-5}$, but the average error for the 144 measurements is only $\pm 1.9 \times 10^{-5}$. The reproducibility, or precision, of these measurements, expressed as the average of the deviations from the mean value of each set of four immersion measurements, is $\pm 1.5 \times 10^{-5}$.

A summary of the average accuracy and precision of all the determinations is given in table 4.

TABLE 4.—Summary of accuracy and precision of determinations listed in table 3

	Average error	Average deviation from mean values
n_D	1.6×10^{-5}	1.4×10^{-5}
n_P	2.1×10^{-5}	1.6×10^{-5}
n_C	2.1×10^{-5}	1.6×10^{-5}
All values.....	1.9×10^{-5}	1.5×10^{-5}

The accuracy attained in these measurements is almost as high as the precision attainable on a refractometer. This indicates that the errors introduced in determining the match between the indices of the glass and liquid are for the most part negligible. According to Tilton,⁹ it would be very difficult to obtain results of this accuracy without the strict use of a comparison or substitution method. The success attained may be largely attributed to the fact that in measuring a known standard, on the refractometer both before and after each group of four immersion measurements, a comparison method was used that approximates a substitution method. Other factors which should be mentioned are (1) temperature differences between the refractometer and immersion tank were rarely greater than 0.1° , and these temperature effects were considered in obtaining the final

⁹ L. W. Tilton, J. Research NBS 30, 311-328 (1943) RP1535.

values, and (2) the recommended tests to eliminate shielding of rays, setting on false edges, etc. (see footnote 9) were frequently made.

Although the errors introduced in matching are, in general, negligible, the experience of the operator plays a part in these determinations. This is due to surface effects that interfere with the determination of a match. The ideal condition is one which produces a uniformly illuminated field against which the sample is invisible. This exists very rarely in actual practice. Usually, under conditions of match, the main body of the sample will blend with the field, but the edges of the sample will be outlined by very thin brilliant lines. In these cases, a match is determined by rotating the sample through 180°. There should be no change in the thickness or brilliance of these lines when this is done. Thus any confusion due to surface effects may be avoided. The experience of the operator will also determine the speed with which measurements are made. An experienced person can measure about six samples in an 8-hour day. Thus it is possible to secure complete information on the index of refraction and ν value for a particular melt within 1½ hours after a pot of glass is opened.

VI. SUMMARY

An improved apparatus for determining indices of refraction and dispersion of glass by immersion is described. The results of 144 measurements indicate that the index of refraction may be determined with an average error of $\pm 1.9 \times 10^{-5}$ and a maximum error of $\pm 5 \times 10^{-5}$. Dispersion as represented by ν values may be calculated from these measurements with an average error of 0.1 and a maximum error of 0.8. This apparatus may, therefore, be used both for routine production control work and for the determination of the optical constants of finished optical components, especially of flint glasses. It may also be applied profitably to research in the determination of the effect of composition on the optical properties of glass, especially where the glasses studied may be unstable. The full possibilities of the matching instrument have not been determined. More accurate refractometers and temperature controls are necessary for this purpose.

WASHINGTON, October 1, 1943.

COLOR-TEMPERATURE CALIBRATION

(To accompany calibration reports on incandescent lamps)

NOTES

1. **DEFINITION.** The color-temperature scale is based on the chromaticity of the light emitted by a tube immersed in an element (platinum, rhodium, or iridium) at its freezing point (2,042, 2,233, or 2,716° K). These chromaticities can be duplicated closely by means of incandescent tungsten-filament lamps by adjustment of the voltage applied, and a lamp emitting light of the same chromaticity as that emitted by a hollow enclosure is said to have a color temperature equal to the true temperature of the walls of the enclosure. Our primary lamp standards were calibrated in this way, (RP677 and RP2053).

2. **SECONDARY STANDARDS.** Secondary lamp standards of color temperature are prepared by visual comparison with the primary lamp standards by means of the 9 by 13° double-trapezoid field of a Lummer-Brodhun contrast photometer head and by use of a substitution method. The color-temperature scale has been extended for these secondary standards to higher and lower color temperatures by means of amber and blue filters.

3. **METHOD.** Duplicates of these secondary standards are prepared by the same substitution method and are issued as working standards. They are tungsten-filament, gas-filled, projection lamps. They are burned base down and are so oriented that the filament supports are away from the photometric test plate. In the course of the comparison each lamp is set to a convenient voltage and the current in amperes flowing through the lamp after about 3 minutes of burning at this voltage is measured. This check current serves to indicate at any later time whether the electrical characteristics have changed.

4. **COLOR-TEMPERATURE PERMANENCE.** Life tests of 400-watt projection lamps indicate that they decline in color temperature about 1 degree per hour of burning at 3,140° K, about 0.1 degree per hour at 2,850° K, and about 0.01 degree per hour at 2,360° K; see RP937.

5. **FORMULA CONNECTING VOLTAGE AND COLOR TEMPERATURE.** It has been found that the relationship between color temperature, T_c , and voltage, V , applied to this type of lamp can be expressed within the uncertainty of the determination by the formula:

$$V = a + [(T_c - b) / d]^2,$$

where a , b , and d are constants to be evaluated for each lamp from the observed voltages for a series of color temperatures.

6. **ESTIMATED UNCERTAINTY.** The uncertainty of a visual setting of color temperature is closely proportional to the square of the color temperature; at the 0.1 of 1 percent confidence level it is about 4° near 2,000° K, and about 9° near 3,000° K. Duplicates of the secondary standards are customarily calibrated by making six settings for color-temperature match, the mean of which is thus uncertain over this color-temperature range by 2 to 4° from this source alone. The estimated uncertainty listed is considerably larger than this because it includes accumulated uncertainties from the calibrations of the primary and the secondary standards and from any extension of the color-temperature scale, in addition to the uncertainty introduced by this final step. Intermediate voltages found by the interpolation formula are subject to a slight additional uncertainty.

7. **USES OF THE STANDARDS.** Working standards of color temperature are chiefly used to set up standard lamps for illumination of specimens for colorimetry. Standard sources A, B, and C recommended in 1931 by the International Commission on Illumination (CIE) all involve a lamp at a color temperature of 2,848° K with constant C_2 of Planck's formula taken as 14,350 (RP2384). In October 1948 the Ninth General Conference on Weights and Measures adopted a revision of the International Temperature Scale in which the value assigned to the constant C_2 was changed to 14,380. By this revision (RP2053) the spectral energy distribution of source A is identified by the color temperature 2,854° K, and this change in the definition of CIE source A was made in 1951 by the International Commission on Illumination. Working standards of color temperature are also used as chromaticity standards in the colorimetry of light sources, as standard lamps for testing the sensitivity of photographic materials and photocells, and as standards of spectral energy distribution between 400 and 700 $m\mu$ for the calibration of spectroradiometers (RP2053).

UNITED STATES DEPARTMENT OF COMMERCE • Sinclair Weeks, *Secretary*
NATIONAL BUREAU OF STANDARDS • A. V. Astin, *Director*

Precision Laboratory Standards of Mass and Laboratory Weights

T. W. Lashof and L. B. Macurdy



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Foreword

National Bureau of Standards Circular 3, Design and Test of Standards of Mass, has been a basic reference on mass standards and weighing since its publication in 1918. The expanding needs of science, industry, and commerce call for the replacement of Circular 3 by a larger and more comprehensive document. Because of the time required to prepare a single large document, it is planned instead to prepare a series of publications covering the subject matter of Circular 3 and issue each as it is completed.

This first publication presents the specifications of the National Bureau of Standards for precision laboratory standards and other laboratory weights. It also presents the regulations governing the submission of these weights to the Bureau for test, and outlines the weight-calibration service of the Bureau. It supersedes chapters VI, VII, and XI and related portions of other chapters of Circular 3.

Other publications will present specifications for commercial standards of mass, methods of weighing, tests for sets of weights, a discussion of fundamental standards and concepts, and the design and test of balances and scales.

The material presented here is based not only on the third (1918) edition of Circular 3, prepared by A. T. Pienkowsky, but also on Mr. Pienkowsky's 1941 manuscript for a fourth edition. It also is based on suggestions and assistance received from manufacturers and suppliers of weights for scientific use at the 1952 Annual Meeting of the Scientific Apparatus Makers Association.

A. V. ASTIN, *Director.*

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Precision Laboratory Standards of Mass and Laboratory Weights

T. W. Lashof and L. B. Macurdy

This is the first part of the revision of National Bureau of Standards Circular 3, Design and Test of Standards of Mass, last revised in 1918. Laboratory standards of mass in order of decreasing precision are classes J, M, S, and S-1. The newly introduced class J weights, with a tolerance of 0.003 milligram for each weight, may be used for the calibration of equipment for ultramicroanalysis. Class M weights are reference standards for high-precision work and work demanding high constancy. Class S weights are laboratory working standards. Maintenance tolerances start at 0.0054 milligram for class M fractional weights and 0.014 milligram for the smallest class S weights, and decrease to 5 parts per million for the larger weights of both classes. Class S-1 are for use in routine analytical work with quick-weighing balances and are the most precise weights available in nonmetric units. Tolerances vary from 0.025 milligram for the smallest weights to 10 parts per million for the larger weights.

Classified for the first time are class P laboratory weights for routine analytical work, class Q for technical and student use, and class T for rough weighing operations. Maintenance tolerances for these weights start at 0.1 milligram for classes P and Q and 0.8 milligram for class T and decrease to 40, 100, and 300 parts per million, respectively, for the larger weights of these classes.

In addition to the tolerances, the denominations, composition, construction, marking, packing, and performance of weights of each class are fully described. Also described are the nature and precision of the tests available and other features of the Bureau's weight-calibration service. In general, the calibration service is intended primarily to provide standard weights for the calibration of other weights, for legal use, and for use in scientific work where the highest precision is required.

1. Introduction

The National Bureau of Standards throughout its history has been the center in the United States for the calibration of precision standards of mass. Because of its position, which stems from its custody of the national standards of mass, the Bureau has been the logical source of a system of classification for both commercial and scientific weights. Although the National Bureau of Standards is not invested with the authority to impose mandatory classifications and tolerances for weights, the classifications and tolerances that have been recommended by the Bureau have met with almost universal acceptance in this country.

The first classification issued by the National Bureau of Standards was given in Circular 3, in 1903. This was revised in 1907 and again in 1918. The 1918 revision of the Circular (although out of print for 25 years or more) has continued as a basic reference on mass standards up to the present time, even though additional classes of weights have been added to supplement the original ones.

During the 35 years that have passed since the publication of the third edition of Circular 3, a great expansion of scientific, technical, and commercial activity has taken place, bringing with it the need for new classes of weights and demands for improved constancy and accuracy in certain ranges of the established classes of weights. The

major changes that have been made to meet these needs are:

1. The introduction of a new class J for very accurately adjusted microweights.

2. The use of tighter tolerances for classes M and S, especially for the smaller weights, in order that in semimicro and analytical work the corrections for these weights normally may be neglected.

3. The introduction of "group" tolerances in classes M and S, for the above purpose, and in order to prevent accumulation of errors when many small weights are used.

4. The separation of class S into an improved class S and a new class S-1, the latter being almost the equivalent of the class S of Circular 3, third edition, and these two classes conforming to the trade practice of double- and single-checked weights.

5. The provision of nonmetric denominations under class S-1, to meet the need for laboratory standards in these denominations.

6. The introduction of three classes, P, Q, and T, of rough laboratory weights, essentially equivalent to the classes of weights now available from the laboratory supply houses, in order to provide the standardization desired by the industry.

7. The use of a double tolerance scheme (acceptance tolerances for new or newly adjusted

weights and maintenance tolerances for weights that have been in use), which recognizes that weights of certain denominations and classes can and should be adjusted very closely so that they will remain within useful tolerances for a reasonable length of time.

S. A more detailed statement of the requirements, particularly with regard to material, design, and surface finish, and other factors that affect the constancy of weights, with a tightening of these requirements for classes M and S.

Several manufacturers are now making weights that meet the closer tolerances and other requirements for classes M and S mentioned above. As a matter of fact, the Bureau in 1951 increased the accuracy and precision with which it certified these weights. Modern research and the new quick-weighing devices demand the improved constancy and accuracy.

The Bureau will accept weights for certification under these new requirements, beginning 3 months after the date of this publication. For a period of a year after the date of this publication, the

Bureau will continue to certify weights under the previous requirements of Circular 3. After the end of the 1-year period, noncommercial weights will be tested on the basis of these new requirements, and will be certified under one of the classes J, M, S, S-1, P, Q, and T, only when they meet all the requirements contained herein for the class for which they are submitted.

Although every effort has been made to meet present needs and to anticipate future needs for scientific mass standards and other laboratory weights, situations undoubtedly will arise that will indicate the need for amendments and additions to the material contained in this publication. The staff of the National Bureau of Standards will consider suggestions for modifications of these specifications. Recommendations for changes should be submitted to the National Bureau of Standards in writing, with sufficient detail and with the information, research documents, etc., necessary to establish the need for the recommended changes.

2. Classification of Weights

Weights may be divided into four groups according to their use, namely, precision laboratory standards, laboratory weights, commercial standards, and trade weights. Precision laboratory standards include weights used in scientific and technical laboratories as standards of mass for the calibration of other weights and weighing equipment or as precision laboratory weights for analytical work. Laboratory weights include weights used for rough analytical work and for general laboratory and technical work. Commercial standards include the State, office, and working standards used in law enforcement. Trade weights include weights used in the purchase and sale of goods both by the Government and in ordinary trade.

Table 1 lists the classes of weights under each of these groups except the trade group. The classification, specifications, and regulations for trade weights (as adopted by the National Conference of Weights and Measures and recommended by the National Bureau of Standards for promulgation by the several States) are covered in NBS Handbook 44,¹ revised to date, or its successor.

The intended use or purpose of weights of each of the laboratory classes is given at the beginning of chapters 3 and 4; that for each of the commercial classes (A, B, and C) is given in Circular 3.²

TABLE 1. *National Bureau of Standards classification of weights*

Class	Application
PRECISION LABORATORY STANDARDS	
J	Microweight standards (microbalance work).
M	High-precision scientific standards (reference, high-precision, and high-constancy work).
S	Scientific standards (reference, calibration, and precision analytical work).
S-1	Laboratory standards (routine analytical and precision nonmetric work).
LABORATORY WEIGHTS	
P ¹	Analytical and precise technical weights.
Q	General laboratory, technical, and student weights.
T	Utility weights.
COMMERCIAL STANDARDS (reference, working, and field standards used in law enforcement)	
A ²	State primary standards.
B ²	State working standards.
C ²	Test weights.
TRADE WEIGHTS (weights used in the sale of commodities and services)	
See classes listed in NBS Handbook 44.	

¹ NBS Handbook 44, Specifications, tolerances, and regulations for commercial weights and measures, and weighing and measuring devices, for sale by the Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C., \$1.25.

² NBS Circular 3, 3d ed., Design and test of standards of mass (1918.) Out of print, but available in many libraries.

¹ Formerly class S-2.

² This classification is being revised.

3. Requirements for Precision Laboratory Standards of Mass— Classes J, M, S, and S-1

Class J. These are metric weights designed primarily as standards for the calibration of weighing equipment used in the precise determination of very small masses in ultramicroanalysis. The effect of changes in relative humidity will be small for properly constructed class J weights.

Class M. These are metric weights designed for use as reference standards, for work of the highest precision, and for investigations demanding a high degree of constancy over a period of time. Tolerances for class M weights up to 5 g have been so selected that for most semimicro and microchemical work the corrections for the individual weights may be neglected.

When used with care, properly constructed class M weights will be constant within considerably less than the prescribed tolerances. As a group, these weights should be constant for several years within the indicated accuracy of the high-precision class M calibration (see 5.1, class M (a)).

Class S. These are metric weights designed as working standards for the calibration of other weights or as high-precision analytical weights for the more precise weighings in physical and chemical laboratories and in assay work. The tolerances for class S weights are such that the weights may be used without corrections, other than allowance for air buoyancy, for most analytical work and as keyboard or dial-controlled weights in quick-weighing analytical balances having a 200-g capacity.

Class S weights may be used also as reference standards. However, they may not be expected to remain as constant as class M weights.

Class S-1. These are designed for use as precision laboratory weights in routine analytical work with balances using quick-weighing devices, such as a chain. Class S-1 weights bridge the area between the best laboratory weights (class P) and the precision laboratory standards. Class S-1 includes weights in avoirdupois, apothecary, troy, and certain other units (see table 4), as well as those in metric units. Weights that meet class S-1 specifications are the most precise laboratory working standards currently available in non-metric units.

3.1. Material

3.1.1. HARDNESS

Class J. Weights shall be constructed of hard, nonporous material, resistant to abrasion, and not likely to chip or spall in use. The Knoop hardness number of this material when determined at a test load of 100 g shall be 165 or greater (e. g., platinum-iridium or tantalum).

Classes M, S, and S-1. The hardness requirement shall apply to the material of which the body (including knob) of the weight is constructed

and also to the plating of a plated weight, except as noted below under 3.1.1.1. The Knoop hardness number shall be as follows: (a) 125 or greater at a test load of 200 g for weights above 30 mg or equivalent (brass and harder materials, such as stainless steel); (b) 55 or greater at a test load of 100 g for weights 30 mg and below (aluminum and harder materials).

3.1.1.1. OPTION

Classes M and S. At the option of the purchaser, if class M or S weights are to be used as reference standards, the surface of weights 1 g and above may be protected with gold.

3.1.2. CORROSION RESISTANCE

Classes J, M, S, and S-1. Weights shall be constructed of material that is resistant to oxidation or corrosion, except as follows:

Classes M, S, and S-1. Brass and materials similarly susceptible to oxidation and corrosion may be used for weights 1 g and above, provided that the surface is suitably protected in accordance with 3.3.3. Aluminum, aluminum alloys, and other metals that have resistance to oxidation and corrosion, depending largely on the surface condition of the metal, may be used only when the surface will take and hold a high polish.

3.1.2.1. ADJUSTING MATERIAL

Classes S and S-1. Tantalum, copper, aluminum, tin, or material equally resistant to oxidation and corrosion shall be used if adjusting material is required. Materials that readily oxidize and corrode (lead, dirt, powdered materials, organic materials, such as paper and oil, etc.) shall not be used in adjusting cavities.

3.1.2.2. CONTAMINATION

Classes J, M, S, and S-1. All surfaces, exterior and interior (including the adjusting cavity and threads of the knob) shall be free of deposits, residues, and other contaminating substances, such as residues from electroplating and cleaning solutions.

3.1.3. MAGNETIC PROPERTIES

Classes J, M, S, and S-1. Weights shall be constructed of nonmagnetic material. Neither ferromagnetic nor strongly paramagnetic materials may be used.

Classes S and S-1. Nonmagnetic stainless-steel alloys (including type 18-8) may be used, provided the nickel content is at least 50 percent by weight of the chromium content.

3.1.4. DENSITY

Classes J and M. No requirements.

Classes S and S-1. The total volume, including any air-tight cavity, shall be such that the average density is within the ranges shown in table 2.

TABLE 2. Density for class S and S-1 weights

Weights	Allowed density
	<i>g/cm³</i>
Greater than 1 g (or equivalent)-----	7.8 to 9.1
1 g to 50 mg, inclusive (or equivalent)-----	4.5 and up
Less than 50 mg-----	2.6 and up

3.2. Design

3.2.1. GENERAL SHAPE

Classes J, M, S, and S-1. A weight may have any shape that does not introduce features that reduce the reliability of the weight. All weights shall be free of ragged or sharp edges or ends. Wire weights shall not be excessively long and shall not be tightly coiled or otherwise shaped so as to have a tendency to catch and hold lint or dust. Both sheet-metal and wire weights shall be free from cracks such as may be formed in bending.

3.2.2. THICKNESS

Classes J, M, S, and S-1. Sheet-metal weights shall not be unnecessarily thin. In particular, the weights shall not be so thin that the surface tension of water will bend them out of shape.

Class J. The nominal thicknesses of the material from which sheet-metal weights are formed shall be equal to or greater than the values in table 3. The actual thickness shall be not less than 90 percent of the nominal thickness.

3.2.3. NUMBER OF PIECES

Classes J, M, S, and S-1. The entire weight shall be a single piece and homogeneous, except as follows:

Class M. Surface protection in accordance with 3.3.3; gold adjusting material fused on the upper surface of sheet-metal weights.

Classes S and S-1. Surface protection (3.3.3); means of adjustment (such as a screw knob covering an adjusting cavity); adjusting material in adjusting cavity; gold adjusting material fused on the upper surface of sheet-metal weights.

TABLE 3. Nominal thickness for class J sheet-metal weights

(Minimum values)

Weight	Thickness	Weight	Thickness
<i>mg</i>	<i>in.</i>	<i>mg</i>	<i>in.</i>
50	0.0059	1	0.0009
30	.0047	0.5	.0006
20	.0042	.3	.00045
10	.0031	.2	.00037
5	.0023	.1	.00026
3	.0015	.05	.00019
2	.0013		

3.3. Surface

3.3.1. IRREGULARITIES

Classes J and M. The entire surface of the weight shall be smooth, except for such markings as are allowed under 3.5, and shall be highly polished.

Classes S and S-1. The entire surface of the weights shall be smooth, except for such markings as are required under 3.5, and shall be carefully polished or have equivalent finish.

3.3.2. POROSITY

Classes J, M, S, and S-1. When subjected to unaided visual inspection, the surface shall appear free from pits and pores and shall show no effects of porosity.

3.3.3. PROTECTION

(a) LARGER WEIGHTS (i. e., weights other than sheet-metal or wire weights)

Class M. Weights of brass, bronze, or other metals that tarnish on exposure to the atmosphere shall be plated with platinum, rhodium, or other suitable metal. Gold plating may be used at option of purchaser (3.1.1.1). There shall be no darkening of the surface and no formation of spots of any kind when the weights are boiled for 3 half-hour periods in distilled water and cooled in distilled water between boilings, or when the weights are subsequently dried at a temperature of 110° C for 1 hour.

Classes S and S-1. Unless the surface material is at least as resistant to atmospheric corrosion as aluminum, it shall be plated with metals such as platinum or rhodium, or shall be lacquered. Lacquer, if used, shall be hard, of only moderate thickness, transparent, and not easily chipped. Lacquer shall not be used, however, on weights that are made of, or are plated with, a material that normally need not be protected against oxidation or corrosion. Gold plating shall not be used as the final coating, except that class S weights that are to be used as reference standards may be gold plated at option of purchaser (3.1.1.1).

(b) SHEET-METAL AND WIRE WEIGHTS

Classes J, M, S, and S-1. Sheet-metal and wire weights shall be made of a material that requires no surface protection. If these weights are adjusted by fusing or plating, the adjusting material shall require no surface protection. Lacquer or other coatings, other than electroplating, shall not be used.

3.4. Denominations

3.4.1. SYSTEM IN WHICH DENOMINATIONS ARE EXPRESSED.

Classes J, M, and S. The system shall be metric.

TABLE 5. Maximum and minimum denominations of commonly used sets of precision laboratory standards of mass

Class and purpose	From maximum	To minimum
<i>Class J:</i> Microbalance.....	50 mg	0.05 mg
<i>Classes M and S:</i> GENERAL PURPOSE		
Analytical balance.....	100 g	1 mg
Semimicro-balance.....	50 g	1 mg
Microanalytical balance.....	20 g	1 mg
Assay balance.....	1 g	1 mg
BALANCE WITH BUILT-IN WEIGHTS (100 mg to 1 mg)		
Analytical balance.....	100 g	100 mg
Semimicro balance.....	50 g	100 mg
Microanalytical balance.....	20 g	100 mg
<i>Class S-1:</i> GENERAL PURPOSE (metric)		
Large-capacity balance.....	20, 5, 2, or 1 kg	1 g or 5 mg
Large-capacity analytical balance.....	500 or 200 g	5 mg
Analytical balance.....	100 g	5 mg
ANALYTICAL BALANCE		
With chain.....	100 g	100 mg
With chain and rider or with chain and built-in weights.....	100 g	1 g
GENERAL PURPOSE (customary)		
Avoirdupois.....	50, 10, 5, 2, or 1 lb or 8 oz	$\frac{1}{32}$ oz
Do.....	50, 10, 5, 2, or 1 lb	0.001 lb
Do.....	10 oz	0.001 oz
Apothecary.....	12 oz ap or 4 dr ap	0.1 grain
Troy.....	500 oz t	0.1 grain
Do.....	500 oz t	0.001 oz t
Grain.....	1,000 grain	0.1 grain
Carat.....	(with two 200-c weights) 200 c	0.01 c
Assay ton.....	4 or 1 A.T.	$\frac{1}{20}$ A.T.

3.4.3.2. OPTIONAL MAXIMUM AND MINIMUM DENOMINATIONS.

Classes J, M, S, and S-1. Maximum and minimum denominations of commonly used sets of laboratory standards of mass are given in table 5.

3.5. Marking of Weights

3.5.1. DENOMINATION

Classes J, M, S, and S-1. Weights shall be marked with the number and unit, as specified in 3.5.1.1 and 3.5.1.2

3.5.1.1. NUMBER

By "number" is meant the number representing the nominal mass or "value" of each weight as expressed in terms of some acceptable unit.

Class J. If the shape or size of a weight does not distinguish it from other weights of the set, it may be marked with small shallow dots or other distinguishing marks. The numbers may be marked on the larger weights, and, if marked, shall be expressed in milligrams

Class M. The numbers may be marked on the weights, and if marked, shall be expressed in kilograms, grams, or milligrams.

Class S. The number shall be plainly marked upon each weight, except in the case of riders and other wire weights, and shall be expressed in kilograms, grams, or milligrams.

Class S-1. The number shall be plainly marked upon each weight, except in the case of riders and other wire weights, and shall be expressed in terms of one of the units listed in appendix 2.

3.5.1.2. UNIT

Classes J, M, S, and S-1. The unit (in terms of which the nominal value of each weight is expressed) shall be as specified in 3.5.1.1 for each class. The name of the unit, if abbreviated, shall be abbreviated in accordance with appendix 2. The abbreviation shall not include a period.

Classes J and M. The unit shall not be marked on weights less than 100 g.

Classes S and S-1 (metric). The unit may be marked on the larger sheet-metal and cylindrical weights.

Class S-1 (nonmetric). The markings shall include the name of the unit or its accepted abbreviation in accordance with appendix 2, except that on cylindrical weights below ½ oz or equivalent and on sheet-metal weights below 1 grain or equivalent, the name of the unit may be omitted when space does not permit its inclusion in legible form.

3.5.2. DUPLICATE WEIGHTS

Classes J, M, S, and S-1. Duplicate weights shall each be marked with one or more distinguishing marks.

3.5.3. UNNECESSARY MARKINGS

Classes J, M, S, and S-1. Markings other than those required or allowed in 3.5.1.1, 3.5.1.2, and 3.5.2 shall not be used.

3.5.4. DEPTH OF MARKINGS

Classes J, M, S, and S-1. Markings shall be shallow, relatively broad, and free from burrs and sharp angles. The markings shall not perforate or crack sheet-metal weights.

3.6. Lifters

Classes J, M, S, and S-1. Special lifters or forceps shall be provided for sets of weights. Lifters or forceps shall be provided for individual weights when specified by the purchaser.

3.6.1. DESIGN OF LIFTERS

Classes J, M, S, and S-1. The lifters or forceps shall hold securely the weights for which they are designed. Additional pressure shall not cause the dropping of small weights or the forceful ejection of large weights.

(a) For weights 500 g and larger the parts of the lifters that may come into contact with the weights shall be covered with some material softer than the surface of the weight, such as plastic, velvet, or chamois skin from which the grease has been removed.

(b) For smaller weights the lifters may be of the same design, where practicable, or may be of a material softer than the weights, such as ivory, close-grained wood, or plastics that are not affected by alcohol. When the parts of the lifters or forceps that come in contact with the weights are not covered with a soft material, they shall be smooth and polished and the edges on which weights may be partially or wholly lifted shall be well rounded.

(c) If forceps are used solely for lifting sheet-metal weights, steel forceps with gold-plated tips may be used.

3.6.2. WARPING OF LIFTERS

Classes J, M, S, and S-1. Forceps shall be so constructed that with use or with storage in a closed position they will not warp enough to interfere with their performance.

3.7. Case

Classes J, M, S, and S-1. One or more suitable cases shall be provided with each set of weights. The case shall be so designed that, as long as the lid remains closed, the weights cannot get out of their pockets (3.7.1). The hinges and locks shall be adequate to hold the lid closed with any reasonable handling. There shall be no discoloration of the weights due to the lining or the case, such as might result from long storage in a warm or damp location.

Class J. The case shall be dustproof.

3.7.1. POCKETS

Classes J, S, and S-1. A separate pocket shall be provided for lifters or forceps and for each weight, and all pockets shall be large enough so that no appreciable frictional force will be encountered in inserting or removing weights. If the cover is not lined, the individual holes in the cover for the knobs of the weights shall be smooth or lined. Pockets for weights 1 g or equivalent and larger shall be lined with some soft material, such as velvet.

Class M. Either a lined pocket shall be provided, as specified for classes J, S, and S-1, or the position of the weight may be determined by a shallow ring or plate. If a guard ring or plate is used, the diameter of the unlined pocket or opening shall be at least 10 percent larger (and in all instances at least 2 mm larger) than the diameter of the weight, and all parts of the case shall be designed so that the weights can be inserted or removed without danger of rubbing on any part of the case.

3.7.2. MARKING OF CASE

Classes J, M, S, and S-1. The class and the unit or system of units shall be marked conspicuously on the case. In addition, denominations shall be marked as follows:

Class J. Each pocket of the case shall be marked with the denomination of the weight to be kept in the pocket.

Class M. If the denomination is not marked on the weight (3.5.1), it shall be marked beside the pocket in which the weight is kept.

Classes S and S-1. The denominations may be marked beside the pockets in which the weights are kept.

3.8. Tolerances

The tolerance of a weight is the maximum allowable departure of the weight from its nominal value.

3.8.1. BASIS FOR ADJUSTMENT

Classes J, M, S, and S-1. Ordinarily, weights shall be adjusted according to their "apparent mass versus brass" values. However, in those cases in which the larger weights of a set are of

TABLE 6. Tolerances for class S-1 weights

Not more than one-third of the weights of a set of new or newly adjusted weights may be in error by more than one-half of these tolerances, and all weights shall be correct within these tolerances.

Avoirdupois			Troy			Apothecary							
Denomination	Tolerance	Denomina- tion	Tolerance	Denomina- tion	Tolerance	Denomina- tion	Tolerance						
lb	100	450	mg	0.010	oz	10	0.043	Grain	10,000	oz ap	12	0.059	Grain
	50	230	mg	0.035	oz	8	0.022	Grain	5,000	dr ap	6	0.048	Grain
	25	110	mg	0.022	oz	5	0.017	Grain	3,000	oz ap	10	0.039	Grain
	20	91	mg	0.017	oz	4	0.010	Grain	2,000	oz ap	8	0.029	Grain
	10	45	mg	0.010	oz	2	0.0066	Grain	1,000	oz ap	6	0.025	Grain
	8	36	mg	0.0066	oz	1	0.0046	Grain	500	oz ap	4	0.019	Grain
	5	23	mg	0.0046	oz	1/2	0.0032	Grain	300	oz ap	3	0.014	Grain
	4	18	mg	0.0032	oz	1/4	0.0025	Grain	200	oz ap	2	0.011	Grain
	3	14	mg	0.0025	oz	1/8	0.0018	Grain	100	oz ap	1	0.0069	Grain
	2	9.1	mg	0.0018	oz	1/16	0.0015	Grain	50	oz ap	3	0.0059	Grain
1	4.5	mg	0.0015	oz	1/32	0.0012	Grain	30	oz ap	2	0.0053	Grain	
lb	0.5	2.3	mg	0.0012	oz	1/64	0.0009	Grain	20	oz ap	1	0.0048	Grain
	0.2	0.91	mg	0.0009	oz	1/128	0.0007	Grain	10	oz ap	1	0.0048	Grain
	0.1	0.45	mg	0.0007	oz	1/256	0.0005	Grain	5	oz ap	1	0.0048	Grain
	0.05	0.23	mg	0.0005	oz	1/512	0.0004	Grain	3	oz ap	1	0.0048	Grain
	0.02	0.11	mg	0.0004	oz	1/1024	0.0003	Grain	2	oz ap	1	0.0048	Grain
	0.01	0.058	mg	0.0003	oz	1/2048	0.0002	Grain	1	oz ap	1	0.0048	Grain
	0.005	0.029	mg	0.0002	oz	1/4096	0.0001	Grain	0.5	oz ap	1	0.0048	Grain
	0.002	0.014	mg	0.0001	oz	1/8192	0.00005	Grain	0.3	oz ap	1	0.0048	Grain
	0.001	0.007	mg	0.00005	oz	1/16384	0.00002	Grain	0.2	oz ap	1	0.0048	Grain
	0.0005	0.0035	mg	0.00002	oz	1/32768	0.00001	Grain	0.1	oz ap	1	0.0048	Grain
0.0002	0.0017	mg	0.00001	oz	1/65536	0.000005	Grain	0.05	oz ap	1	0.0048	Grain	

some material having a density markedly different from brass, all the weights of the set may be adjusted (at the option of the purchaser) according to the apparent mass vs. platinum, tantalum, gold, or whatever is the material of the larger weights.

"Apparent mass vs. brass" values are those that the weights would be assigned on the basis of a comparison at 20° C in normal air against normal brass standards. "Apparent mass vs. other material" values are those that the weights would be assigned on the basis of a comparison at 20° C in normal air against the true mass of standards of this other material. In both cases, no corrections would be applied for the buoyant effect of the "normal air" (defined as air having a density of 1.2 mg/cm³), and values would be derived from the true mass of the standards (normal brass standards being defined as standards composed of brass having a density of 8.4 g/cm³ at 0° C and a coefficient of cubical expansion of 0.000054 per deg Celsius (centigrade)).

3.8.2. ACCEPTANCE TOLERANCES

Classes J, M, S, and S-1. New and newly adjusted weights shall meet the tolerance requirements specified in 3.8.2.1 and 3.8.2.2.

3.8.2.1. TOLERANCES FOR INDIVIDUAL WEIGHTS

Class J. For weights that can be intercompared readily in a series (i. e., weights of the preferred denominations listed in table 4), the tolerance for each weight shall be 0.003 mg. For weights the denominations of which are of irregular amounts not readily intercomparable in a series, the tolerance for each weight shall be specified by the purchaser, but normally shall be not less than the tolerances for the class M weights nearest it in value.

Class M. Individual weights shall be correct within the tolerances prescribed in table 7 in the column marked "Individual."

Class S. Individual weights shall be correct within the tolerances prescribed in table 8 in the column marked "Individual."

Class S-1. Weights shall be correct within the tolerances prescribed in table 6, and, in addition, not more than one-third of the weights of a set of new or newly adjusted weights may be in error by more than one-half of these prescribed tolerances.

Classes M, S, and S-1. For an individual weight of a denomination not listed in the tables, the tolerance shall be determined by proportional interpolation between tolerances given for weights of the next larger and next smaller amounts.

3.8.2.2. GROUP TOLERANCES

Classes M and S. The corrections of the individual weights shall be such that no combination of weights that is intended to be used in a weighing shall differ from the sum of the nominal values by more than the amount listed under the group tolerance. The group tolerances and the weights to which they apply are as follows:

Class M. Weights of a set that include weights less than 10 g shall conform to the group tolerances prescribed in table 7 in the column marked "Group."

Class S. Weights of a set that include weights smaller than 50 g shall conform to the group tolerances prescribed in table 8 in the column marked "Group."

3.8.3. MAINTENANCE TOLERANCES

Classes J, M, S, and S-1. Weights that have been in use shall meet the tolerance requirements specified in 3.8.2.1. and 3.8.2.2., except as follows:

Class M. Maintenance tolerances for weights from 500 mg to 100 mg shall be 0.0105 mg for individual weights and 0.020 mg for the group.

Class S. Maintenance tolerances for weights 100 mg and larger shall be twice the acceptance tolerances for these weights, as shown in table 8 both for individual weights and for decade groups.

TABLE 7. Acceptance tolerances¹ for class M weights

Denomination	Individual	Group
<i>kg</i>	<i>mg</i>	<i>mg</i>
25	125	
20	100	
10	50	
5	25	
3	15	
2	10	
1	5.0	
<i>g</i>		
500	2.5	
300	1.5	
200	1.0	
100	0.50	
50	.25	
30	.15	
20	.10	
10	.050	
5	.034	} 0.065
3	.034	
2	.034	
1	.034	
<i>mg</i>		
500	.0054	} 0.0105
300	.0054	
200	.0054	
100	.0054	
50	.0054	} 0.0105
30	.0054	
20	.0054	
10	.0054	
5	.0054	} 0.0105
3	.0054	
2	.0054	
1	.0054	
0.5	.0054	} 0.0105
.3	.0054	
.2	.0054	
.1	.0054	
.05	.0054	0.0105

¹ Maintenance tolerances are the same as acceptance tolerances, except that the maintenance tolerances for weights from 500 mg to 100 mg are 0.0105 mg for individual weights and 0.020 mg for the group.

TABLE 8. Acceptance tolerances ¹ for class S weights

Denomination	Individual	Group
<i>kg</i>	<i>mg</i>	<i>mg</i>
25	62	
20	50	
10	25	
5	12	
3	7.5	
2	5.0	
1	2.5	
<i>g</i>		
500	1.2	
300	0.75	
200	.50	
100	.25	
50	.12	
30	.074	} 0.154
20	.074	
10	.074	
5	.054	
3	.054	} 0.105
2	.054	
1	.054	
<i>mg</i>		
500	.025	} 0.055
300	.025	
200	.025	
100	.025	
50	.014	} 0.034
30	.014	
20	.014	
10	.014	
5	.014	} 0.034
3	.014	
2	.014	
1	.014	
0.5	.014	} 0.034
.3	.014	
.2	.014	
.1	.014	
.05	.014	0.034

¹ Maintenance tolerances for weights below 100 mg are the same as the acceptance tolerances. For weights 100 mg and above the maintenance tolerances are twice the acceptance tolerances.

3.9. Constancy Under Variations in Humidity

Classes S and S-1. When the relative humidity of the surrounding atmosphere is kept at 30 percent for 3 days, raised to 70 percent and kept there for 4 days, and then brought back to 30 percent and kept there for 2 days, neither the mean variation nor the net gain shall be more than the amounts specified or calculated in accordance with table 9.

TABLE 9. Tolerances on variability and net gain caused by variations in atmospheric humidity from 30 percent to 70 percent for weights of classes S and S-1

Denomination	Maximum allowable variation and net gain for—	
	Class S	Class S-1
<i>g</i>	<i>mg</i>	<i>mg</i>
Σ221	0.22	0.65
Σ200	.20	.60
Σ121	.15	.44
Σ100	.13	.38
Σ50	.085	.25
100	.070	.21
50	.050	.14
30	.030	.090
20	.025	.075
10	.016	.048
5	.011	.032
3	.009	.028
2	.007	.020
1	.005	.015

The "mean variation" is defined as the average of the quantities B-A and C-D, and the "net gain" is defined as the quantity D-A, where A, B, C, and D are the measured masses (corrected for the buoyant effect of the air) of the weights under the following conditions: (A) After the first 3 days at 30 percent relative humidity, (B) after 1 day at 70 percent, (C) after 4 days at 70 percent, and (D) after again being at 30 percent for 2 days.

For individual weights of denominations listed in table 9, the allowable variation and net gain shall be as listed in the table. For individual weights of denominations not listed in the table, the allowable variations and net gain shall be determined by proportional interpolation between values given for weights of the next larger and next smaller amounts.

For all ordinary sets and for groups the sum of which does not exceed 250 g or equivalent, the allowable variation and net gain shall be applied to the sum of the weights. Larger groups shall be divided in any appropriate manner into groups of 250 g or smaller. For sets or groups of weights listed in table 9, the allowable variation and net gain shall be as listed. For sets or groups of weights not listed in the table, the allowable variation and net gain shall be equal to the sum of the amounts allowable on weights of the denominations actually in the group.

3.10. Packaging

This section applies to new or newly adjusted weights.

3.10.1. SEPARATE PACKAGE

Classes J, M, S, and S-1. Each set of weights

and each individual weight not part of a set shall be packaged separately.

3.10.2. SEAL

Classes J, M, S, and S-1. The package shall be sealed by the manufacturer or adjuster so that the weights cannot be removed from the package without destroying the seal. The weights shall be packed in accordance with 6.4 before sealing.

3.10.3. MARKING

Classes J, M, S, and S-1. The sealed package shall be marked with the class of weight, the maximum and minimum denominations contained therein, the unit or system of units, the name of the manufacturer, the manufacturer's type num-

ber, a caution against breaking the seal, and any other appropriate information.

3.10.4. MANUFACTURER'S INFORMATION SHEET

Classes J, M, S, and S-1. A manufacturer's information sheet shall be packaged with new weights. It shall list the density to two or three significant figures and also the composition of the alloy, commercial grade, or accepted trade name of the material of which the weights are composed. (See 3.1 for specifications as to density and composition.) The nature of the surface protection (3.3.3) and the construction (one-piece, screw-knobs, screw-knob pinned, driven knob, etc.) shall also be described on the information sheet.

4. Requirements for Laboratory Weights—Classes P, Q, and T

Class P (formerly class S-2). These weights are designed for routine analytical work in the scientific or technical laboratory.

Class Q. These weights are designed for use with precision pressure gages and for the technical work of commercial and student laboratories. Weights of this class are suitable for the laboratory dispensing of chemicals and pharmaceuticals, rough determination of mass in the elementary physics laboratory, etc.

Class T. These weights are designed for rough weighing operations in the physical and chemical laboratories, such as with force-measuring apparatus.

4.1. Material

4.1.1. HARDNESS

Classes P, Q, and T. The Knoop hardness number shall be as follows: (a) 125 or greater at a test load of 200 g for weights above 5 g or ¼ oz or equivalent (brass and harder materials); (b) 55 or greater at a test load of 100 g for weights of 5 g or equivalent and below (aluminum and harder materials).

4.1.1.1. OPTION

Classes P, Q, and T. If the weights are to receive rough use, the hardness requirement may be changed at the option of the purchaser to read: The Knoop hardness number shall be 225 or greater at a test load of 500 g for all weights (cast iron and stainless or dense steel).

4.1.2. CORROSION RESISTANCE

Classes P, Q, and T. Weights shall be constructed of material that is resistant to oxidation

or corrosion. Lead shall not be used. Cast iron and other metals with similar rates of oxidation may be used for weights of class T but shall not be used for weights of the other classes. Iron and steel shall not be used for weights of 5 g or ¼ oz or equivalent and below, except that stainless steel may be used for weights of all denominations.

4.1.2.1. ADJUSTING MATERIAL

Classes P, Q, and T. Brass, copper, aluminum, tin, or material equally resistant to oxidation and corrosion shall be used if adjusting material is required.

Classes P and Q. Lead shall not be used.

4.1.2.2. CONTAMINATION

Classes P, Q, and T. All surfaces, exterior and interior (including the adjusting cavity and threads of the knob) shall be free of deposits, residues, and other contaminating substances, such as cutting oil and residues from electroplating and cleaning solutions.

4.1.3. MAGNETIC PROPERTIES

Class P. Weights shall be constructed of nonmagnetic material, with the following exceptions or provisions:

- (a) Nickel may be used for surface protection.
- (b) Nonmagnetic stainless steel alloys may be used, provided the nickel content is at least 50 percent by weight of the chromium content.
- (c) Dense steel, rolled or otherwise worked, may be used for weights of 10 kg or 20 lb or equivalent and above.

Class Q. Class P specifications apply except that dense steel, rolled or otherwise worked, may be used for weights above 5 g or ¼ oz or equivalent.

Class T. Class Q specifications apply except that cast iron may be used for weights of 100 g or 4 oz or equivalent and above.

4.1.4 DENSITY

Classes P, Q, and T. Weights shall be constructed of materials having densities within the ranges shown in table 10.

TABLE 10. *Density for laboratory weights*

Class	Weights	Allowed density
		<i>g/cm³</i>
P, Q	2 g or 1/16 oz or 30 grains and greater	7.2 to 10.0
	Less than 2 g or 1/16 oz	2.6 and up
T	100 g or 4 oz and greater	7.0 to 10.0
	Less than 100 g or 4 oz and greater than 30 g or 1 oz	2.6 and up
	30 g or 1 oz and smaller	1.0 and up

4.2. Design

4.2.1. EDGES AND CORNERS

Classes P, Q, and T. A weight shall have no sharp edges or corners or other features, such as perforations or cracks, that introduce danger of excessive change with ordinary use. Sheet-metal weights shall not be unnecessarily thin.

4.2.2. RINGS

Classes P, Q, and T. A weight shall have no movable parts, except as follows:

Class T. A weight of this class may have a ring provided that the ring is not split or removable.

4.2.3. KNOBS

Classes P, Q, and T. A knob shall be held in place tightly enough to prevent its working loose with ordinary handling.

4.2.4. SHAPE

Classes P, Q, and T. A weight shall be shaped so as not to be susceptible to undue variability, in particular:

Classes P and Q. A weight of these classes shall not have a coin shape, unless the weight meets the hardness requirement for rough use as specified in 4.1.1.1.

4.2.5. ADJUSTING MATERIAL

Classes P, Q, and T. Adjusting material shall be securely contained and shall not project beyond the surface of the weight.

4.3. Surface

4.3.1. IRREGULARITIES

Classes P, Q, and T. Any irregularities of the surface shall be slight and not of sharp contour. The surface shall be free from pits or pores visible to the unaided eye.

4.3.2. PROTECTION

Classes P, Q, and T. A weight shall be resistant to corrosion or tarnishing by the air or by handling with the bare hands. When the base metal is of such material that protection is required, a plating such as nickel or a thin coating of transparent paint or lacquer shall be used. If transparent paint or lacquer is used, it shall be hard and not likely to chip. No lacquer or paint of any kind shall be used on sheet-metal weights or on weights composed of a material needing no surface protection. If sheet-metal weights are adjusted by fusing or plating, the adjusting material shall require no surface protection. Opaque paint may be used as follows:

Classes P and Q. On weights above 50 kg or 100 lb.

Class T. On all weights, except as noted above.

4.4. Denominations

4.4.1. INDIVIDUAL WEIGHTS

Classes P, Q, and T. Except when weights are intended for a special application, the denominations of the weights shall be selected from table 11.

4.4.2. SETS OF WEIGHTS

Classes P, Q, and T. A set of weights shall include the duplicates necessary to permit weighing of any load within the range of the set.

4.4.2.1. OPTIONAL MAXIMUM AND MINIMUM DENOMINATIONS

Classes P, Q, and T. Maximum and minimum denominations of commonly used sets of laboratory weights are given in table 12.

4.5. Marking of Weights

4.5.1. DESIGNATION OF VALUE

Classes P, Q, and T. The nominal mass or "value" of each weight shall be plainly marked upon it, except in the case of riders and other wire weights. Wire weights shall be bent in such forms as to suggest the denomination, unless they are to be used as riders or for other purposes that demand special forms. Nominal values shall be expressed in terms of one of the units listed in appendix 2.

4.5.2. DESIGNATION OF UNIT

Classes P, Q, and T. The marking shall include the name of the unit or its accepted abbreviation in accordance with appendix 1, except that on knob weights below 10 g or 1/2 oz or equivalent and on sheet-metal weights below 100 mg or 1 grain or equivalent the name of the unit may be omitted when space does not permit its inclusion in legible form. The abbreviation shall not include a period. In the case of weights of the troy or apothecary systems for which the nominal values

TABLE 11. Preferred denominations for laboratory weights

Classes P, Q, and T			Classes P and Q			Class P	
Metric	Avoirdupois		Apothecary	Troy		Grain	Carat
<i>kg</i>	<i>lb</i>	<i>oz</i>	<i>oz ap</i>	<i>oz t</i>	<i>dwt</i>	<i>Grain</i>	<i>c</i>
1,000	2,500	10	12	1,000	10,000	10,000	2,500
500	2,000	8	10	500	5,000	5,000	2,000
200	1,000	5	8	300	3,000	3,000	1,000
100	500	4	6	200	2,000	2,000	500
50	200	3	5	100	1,000	1,000	300
25	100	2	4	50	500	500	200
20	50	1	3	30	300	300	100
10	25	$\frac{1}{2}$	2	20	200	200	50
5	20	$\frac{1}{4}$	1	10	100	100	30
3	10	$\frac{1}{8}$		5	50	50	20
2	8	$\frac{1}{16}$	<i>dr ap</i>	3	30	30	10
1	5	$\frac{1}{32}$	6	2	20	20	5
	4	$\frac{1}{64}$	5	1	10	10	3
	3		4	0.5	5	5	2
<i>g</i>	2	0.5	3	.3	3	3	1
500	1	.3	2	.2	2	2	0.5
300		.2	1	.1	1	1	.3
200		.1	$\frac{1}{2}$.05		0.5	.2
100	0.5	.05		.03		.3	.1
50	.3	.03	<i>s ap</i>	.02		.2	.05
30	.2	.02	2	.01		.1	.03
20	.1	.01	1	.005		.05	.02
10	.05	.005		.003		.03	.01
5	.03	.003		.002		.02	
3	.02	.002		.001		.01	
2	.01	.001		.0005		.005	
1	.005	.0005		.0003			
	.003	.0003		.0002			
<i>mg</i>	.002	.0002		.0001			
500	.001	.0001					
300	.0005	.00005					
200	.0003	.00003					
100	.0002	.00002					
50	.0001	.00001					
30	.00005						
20	.00003						
10	.00002						
5	.00001						
3							
2							
1							
0.5							
.3							
.2							
.1							
.05							

are expressed in pounds, ounces, drams, or scruples, the identification letter "t" or "ap" shall be used in addition to the name or abbreviation of the unit.

4.5.3. UNNECESSARY MARKINGS

Classes P, Q, and T. Markings other than those required or allowed in 4.5.1 and 4.5.2 shall not be used, except as follows: (a) On weights of 25 kg or 50 lb or equivalent and above, unnecessary markings such as the name or the trade mark of maker or dealer shall be limited to no more than the shortest name by which the firm commonly is known, and the numbers or letters composing the marking shall be no larger than those of the denomination. (b) On weights less than 25 kg or 50 lb or equivalent, unnecessary markings shall be

relatively inconspicuous as compared with the denomination.

4.5.4. DEPTH OF MARKING

Classes P, Q, and T. Raised or depressed letters or figures shall not be deeper than 0.04 inch (1 mm).

4.6. Lifters

Class P. Special lifters or forceps shall be provided for weights of a set. All parts of the lifters that come in contact with the weights shall be smooth, and shall have no sharp or rough edges. Forceps shall be so constructed that with use or with storage in a closed position they will not warp so as to interfere with their performance.

TABLE 12. Maximum and minimum denominations of commonly used sets of laboratory weights

Class and system	From maximum	To minimum
<i>Class P (formerly S2):</i>		
Metric.....	100 or 50 g	1 g or 5 mg
Avoirdupois.....	500 mg	5 mg
Apothecary.....	50, 10, 5, or 2 lb	½ oz
Grain.....	12 oz ap or 4 dr ap	0.1 grain
Troy.....	10 grain	0.1 grain
Carat.....	5 oz t	0.1 grain
	(with two 200-c weights) 200 c	0.01 c
<i>Class Q:</i>		
Metric.....	5, 2, or 1 kg	1 g
Avoirdupois.....	500, 200, or 100 g	1 g
Apothecary.....	2 or 1 kg, 500, 200, or 100 g	10 mg
Grain.....	50, 10, or 1 g, or 500 mg	10 mg
Troy.....	8, 4, 2, or 1 lb, or 8 oz	¼ oz
	8, 4, or 2 dr ap	½ grain
	20 grain	1 grain
	50, 20, or 5 oz t	½ grain
<i>Class T:</i>		
Metric.....	20, 5, 2, or 1 kg	10 g
Avoirdupois.....	50, 8, 4, 2, or 1 lb	½ oz

4.7. Case

Classes P, Q, and T. When a closed case is required or provided, it shall be so designed that as long as the lid remains closed the weights cannot get out of their pockets (4.7.1). The hinges and locks shall be adequate to hold the lid closed with any reasonable handling.

Class P. A closed case or box shall be provided with each set of weights.

Classes Q and T. A closed or an open case may be provided for weights of these classes.

4.7.1. POCKETS

Classes P, Q, and T. When a case is provided, it shall contain a separate pocket for lifters or forceps and for each weight, and all pockets shall be large enough so that no appreciable frictional force will be encountered in inserting or removing the weights. Deep pockets either shall be lined with some soft material, such as velvet, or shall be smooth, free from grit or hard specks or streaks, and of fine-grained wood or other material not likely to scratch the weights.

4.7.2. MARKING OF CASE

Classes P, Q, and T. The name of the class and the name of the unit or system of units shall be conspicuously marked on the case.

4.8. Tolerances

4.8.1. BASIS FOR ADJUSTMENT

Classes P, Q, and T. The weights shall be adjusted on the basis of their apparent mass as determined by comparison with brass standards in air.

4.8.2. ACCEPTANCE TOLERANCES

Classes P, Q, and T. New and newly adjusted weights shall be correct within the tolerances prescribed in table 13. The tolerance for a weight less than those listed in table 13 shall be 5 percent of the nominal value of the weight. The tolerance for other weights not listed in the table shall be determined by proportional interpolation between tolerances given for weights of the next larger and the next smaller denomination.

4.8.3. MAINTENANCE TOLERANCES

Classes P, Q, and T. Weights that have been in use shall be correct within twice the acceptance tolerances prescribed in table 13.

4.9. Packaging

This section applies to new or newly adjusted weights.

4.9.1. SEPARATE PACKAGE

Classes P, Q, and T. Each set of weights and each individual weight not part of a set shall be packaged separately.

Classes P and Q. For weights of these classes, the package shall be sealed by the manufacturer or adjuster so that the weights cannot be removed from the package without destroying the seal. The weights shall be packed in accordance with 6.4 before sealing.

4.9.2. MARKING

Classes P, Q, and T. The package shall be marked with the class of weight, the maximum and minimum denominations contained therein, the unit or system of units, the name of the manufacturer, the manufacturer's type number, and any other appropriate information.

TABLE 13. Acceptance tolerances¹ for laboratory weights—Continued

Apothecary			Grain				
Denomination	P	Q	P	Q	Denomination	P	Q
<i>oz ap</i>	<i>mg</i>	<i>mg</i>	<i>mg</i>	<i>mg</i>	<i>Grain</i>	<i>mg</i>	<i>Grain</i>
12	7.5	28	0.12	0.43	10,000	13	36
10	6.2	21	.096	.32	5,000	6.5	22
8	5.0	18	.077	.28	2,000	2.6	11
6	3.8	11	.059	.22	1,000	1.4	6.6
5	3.1	12	.048	.19	500	0.93	4.3
4	2.5	10	.039	.15	200	.57	2.3
3	1.9	8.4	.029	.13	100	.40	1.5
2	1.4	6.5	.022	.10	50	.30	1.0
1	0.90	4.2	.014	.065	20	.22	0.58
<i>dr ap</i>					10	.17	.42
6	.76	3.4	.012	.052	5	.14	.31
5	.69	3.1	.011	.048	2	.11	.22
4	.62	2.6	.0096	.040	1	.090	.17
3	.54	2.2	.0083	.034	0.5	.078	.14
2	.44	1.7	.0068	.026	.2	.064	.11
1	.32	1.1	.0049	.017	.1	.056	.088
1/2	.26	0.72	.0040	.011			
<i>s ap</i>					.05	.052	.072
2	.28	.87	.0043	.013	.02	.050	.0077
1	.22	.58	.0034	.0090	.01	.050	.0077

¹ Maintenance tolerances are twice the acceptance tolerances.

TABLE 13. Acceptance tolerances¹ for laboratory weights—Continued

Metric						Carat			
Denomination	P	Q	T	Denomination	P	Q	T	Denomination	P
kg									
1,000	20	50	150	g	1.2	5.6	62	c	10
500	10	25	75	30	0.90	4.0	44	2,500	mg
200	4.0	10	30	20	.70	3.0	33	2,000	8.0
100	2.0	5.0	15	10	.50	2.0	21	1,000	4.0
50	1.0	2.5	7.5	5	.36	1.3	13	500	2.0
25	500	1.2	4.5	3	.30	0.95	9.4	200	1.0
20	400	1.0	3.8	2	.26	.75	7.0	100	0.70
10	200	500	2.2	1	.20	.50	4.5	50	.50
5	100	250	1.4	mg	.16	.38	3.0	20	.33
3	60	150	1.0	500	.14	.30	2.2	10	.26
2	40	100	750	300	.12	.26	1.8	5	.20
1	20	50	470	200	.10	.20	1.2	2	.15
				100	.10	.20	1.2	1	.12
				50	.085	.16	0.88	0.5	.10
				30	.075	.14	.68	.2	.080
				20	.070	.12	.56	.1	.070
				10	.060	.10	.40		
g	10	30	300	5	.055	.080		.05	.060
500	6.0	20	210	3	.052	.070		.02	.053
300	4.0	15	160	2	.050	.060		.01	.050
200	2.0	9.0	100	1	.050	.050			
100									

¹ Maintenance tolerances are twice the acceptance tolerances.

5. Weight-Calibration Service of the National Bureau of Standards

5.1. Nature of Tests Available

The tests available for precision laboratory standards and laboratory weights are listed in table 14. Special test will be made when necessary.

The nature of tests available for commercial

TABLE 14. Tests available¹ at the National Bureau of Standards for precision laboratory standards and laboratory weights

Class J
<ul style="list-style-type: none"> a. Calibration of microbalance weights that are in a standard series (see 3.4.2); accuracy of calibration: approximately 0.0002 mg. b. Calibration of microbalance weights not in a standard series; accuracy of calibration: approximately 0.0003 mg. c. Qualification test.
Class M
<ul style="list-style-type: none"> a. Calibration (including determination of volume); accuracy of calibration: 1 part in 10^6 for weights 10 g and above, 0.01 mg for weights 10 g to 1 g, inclusive, 0.001 mg for weights below 1 g. b. Constancy test (above high-precision calibration is repeated after 3 months). c. Calibration (volumes determined in a previous calibration); accuracy of calibration: same as for (a). d. Calibration (lower precision); accuracy of calibration: same as for (a) of class S. e. Qualification test.
Class S
<ul style="list-style-type: none"> a. Calibration; accuracy of calibration: 3 parts in 10^6 for weights 10 g and above, 0.03 mg for weights 10 g to 1 g, inclusive, 0.003 mg for weights below 1 g. b. Qualification test.
Class S-1 (other than metric)
<ul style="list-style-type: none"> a. Calibration; accuracy of calibration: 3 parts in 10^6 for weights of 0.3 oz or 155 grain or larger, 0.03 mg for weights 155 grain to 15 grain, inclusive, 0.003 mg for weights below 15 grain. b. Qualification test.
Class S-1 (metric); classes P, Q, and T
<ul style="list-style-type: none"> a. Tolerance test. b. Qualification test.

¹ See 5.1.1 for definitions of calibration, tolerance test, and qualification test.

standards of mass will be listed separately in the proposed revision of specifications for commercial standards. Meanwhile, this information may be obtained from the "Fee Schedule" (see 6.3 below).

Trade weights are tested by the local or State weights and measures officials. They are accepted for test by the Bureau only in exceptional circumstances.

5.1.1. CALIBRATION, TOLERANCE TEST, QUALIFICATION TEST

A "calibration" includes inspection (5.1.2), the tests necessary to guarantee the calibrated values within the accuracy stated, and the determination of calibrated values. The latter determination is based upon more than one standard, with multiple observations and mathematical treatment of the results. Ordinarily, weights of classes J, M, S, and nonmetric S-1 will be accepted for calibration.

A "tolerance test" includes inspection and the determination of accuracy within the specified tolerances.

A "qualification test" includes inspection and the tests necessary to determine whether or not the weights meet all of the requirements of the class for which they are intended. Qualification tests are for the purpose of sampling large lots for acceptance or for establishing the quality of the product of a supplier, and, at the discretion of the National Bureau of Standards, may include destructive tests.

5.1.2. INSPECTION AND TESTING

Weights will be inspected with respect to packaging, sealing, and marking of package, marking and construction of case, construction of lifters, and the marking, surface, design, and corrosion resistance of the weights. Weights will be tested with respect to tolerances, variation with humidity, density, magnetic properties, surface darkening on boiling, corrosion resistance, and hardness, as appropriate for the class and construction of the weights.

In general, work will be discontinued when a substantial number of items in a lot fail to comply with a specification or are found to be unsuitable for use as standards. The sorting of acceptable items from lots not properly graded for certification cannot be undertaken.

5.2. Certificates and reports

A "certificate" or "report" will be issued in those cases in which the weights submitted will serve their proposed use.

When weights conform to the requirements of one of the classes specified herein or to other established standards recognized by the National Bureau of Standards, a "certificate" will be issued.

When weights do not conform to all of the

requirements of the class for which they have been submitted, this will be reported either as a formal "report" or in a letter. Replacement or further instructions will be requested by letter or other communication when the failure to conform may interfere with the proposed use of the weights (such as when a certificate is required for legal reasons, or when the constancy of a weight is in doubt).

When the failure to conform does not interfere with the proposed use of the weights, a "report" will be issued, which will state wherein the weights fail to meet the requirements. For weights intended for classes J, M, S, and nonmetric S-1, the report will list the actual values of the weights

when this is justified by the construction of the weights. Unless otherwise stated, if the corrections given in the report are applied, the results of the weighings will be as acceptable as those made with certified weights.

5.3. Identification of Weights Tested by the National Bureau of Standards

When certified weights are shipped from the Bureau, either the inner wrappings or the shipping case will be sealed, and will bear the Bureau's test number, together with any other numbers or letters that may be necessary for identification.

6. Regulations Governing the Submission of Weights to the National Bureau of Standards for Test

6.1. Application for Test

Written requests shall be made for all tests. The request may be in the form of a letter or purchase order, and shall be signed by an individual authorized to obligate funds.

The request shall state the class under which the weights are to be tested,³ the nature of the test desired (see 5.1), and any other pertinent information. For example, pertinent information would include the fact that the weights have been used as standards in exacting work, and it is important to know what their corrections were at the close of such work. Otherwise, these weights will be cleaned carefully before being tested, thus possibly changing their corrections.

Sufficient information shall be given to enable the Bureau to identify the material (see 6.5).

If a supplier submits weights for a customer, the supplier shall clearly identify the weights. Also unless the supplier expects to defray the cost of calibration, he should arrange for the customer to submit the request for calibration. A request authorizing payment must be received by the Bureau before the work can be started. It is suggested that the weights be identified by tagging with the statement "For (name of customer) on (order number)."

6.2. Priority and Testing Time

In general, tests are undertaken in the order in which the weights are received, provided proper arrangements have been made. However, priority is given to essential basic standards, when the need for priority is clearly established.

Basic standards are understood to include those to be used (1) in the production or calibration of other standards or working instruments, (2) in law enforcement, and (3) for measurements requiring very high accuracy or precision.

³ See the Introduction for effective date of these requirements.

When it is desired that weights be out of normal service for a minimum time, arrangements should be made in advance for the setting of a specific date for the test; shipment then can be made in accordance with this date.

The time necessary for the completion of tests depends on a number of factors. Some tests in themselves require considerable time (as indicated in 5.1.) The condition of the submitted weights, the work load in the NBS Mass Laboratory, and emergency work for the Government are other factors that enter into consideration. Consequently, it is not possible to specify an exact date of completion of tests. However, an estimate of the date of completion will be furnished when the request for test is received. Every effort will be made to meet this estimate.

6.3. Fees

The fees charged for the calibrations and tests listed in 5.1. are given in the "Fee Schedule" of the National Bureau of Standards, a copy of which will be furnished on request.

6.3.1. CALIBRATION FOR GOVERNMENT AGENCIES

The National Bureau of Standards is required by its basic legislation to charge for all calibrations and tests except those performed for the Government of the United States or State governments, the latter being understood to include State universities. Within the limits of appropriated funds, comparisons and calibrations of essential basic standards (6.2) are performed free for Federal and State governments. In order to take advantage of this free service, it is only necessary that a written request for calibration be made to the Bureau by the Government agency or institution concerned at the time the standards are submitted.

Basic laboratory standards of mass, for the purpose here described, include only weights of classes J, M, and S, and the nonmetric weights of class S-1. Metric weights of class S-1 and all weights of classes P, Q, and T, whether submitted by Government or private agencies, will be accepted for calibration by the Bureau only in exceptional circumstances. Tests of these weights to determine compliance with specifications (i. e., tolerance or qualification tests, see 5.1) will be made for Government agencies on the basis of a transfer of funds to cover actual cost.

Free calibration service cannot be rendered on the request of contractors or firms acting as agents for Federal or State governments because such firms are not in fact a part of government. Under some circumstances, however, a Government agency may have occasion to request the free calibration of standards being delivered to such a contractor or agent.

If weights are purchased by a Government agency after they have been calibrated at the supplier's expense, there is no practicable means by which the Bureau can refund the fee collected for calibration.

6.4. Packing

The weights, if shipped in their regular laboratory handling cases, shall be packed tightly in their pockets through the use of extra material. In many cases sheet-metal weights, which are packed under inner covers of the laboratory cases, are bent or otherwise damaged in shipment. For this reason, great care should be given to the packing of these weights. Sufficient extra packing shall be used to hold the inner cover firmly in place.

The regular laboratory handling cases are not suitable for shipping cases, and therefore they should be carefully packed in boxes that can be used in return shipment. Also, unless the laboratory cases are sturdy and designed so as to hold the weights securely in place during shipments, weights larger than about 200 g should be shipped separately from the case. The larger weights

should be wrapped separately and so placed that they cannot damage containers or other weights.

In wrapping the better grades of weights, when they are not shipped in cases containing pockets, some soft but firm material should be used next to the weight and bound tightly against it to avoid the possibility of abrasion. If tissue paper is used, many thicknesses should be wrapped on firmly, and this tissue should be covered with thicker, soft material, such as cotton packing or knit goods. The whole should then be wrapped securely in tough wrapping paper in order to exclude the dust and grit from the rough packing material in which the boxes or bundles are generally packed for shipment.

Manufacturers' sealed packages are required to be packed in accordance with this section (3.10.2 and 4.9.1).

6.5. Shipping and Identification

Shipping charges to the Bureau must be prepaid. Return shipments are made to other than Federal agencies via express, collect. Government bills of lading, when required for return shipment, should be included with the original request for test.

If a test number has been assigned prior to the shipment, this number must appear on the shipping container. If a test number has not been assigned, a packing list, purchase order, or letter should accompany the shipment for identification purposes.

All packages should bear the shipper's name and address, a list of the contents, and any other marks that may be necessary to insure ready identification. The inner wrappings of each package shall bear thereon the denominations of the weight or weights enclosed (see 3.10 or 4.9).

Articles to be tested shall be directed to "National Bureau of Standards, Washington 25, D. C." If shipping cases of wood are used, the covers should be put in place with screws, and the return or forwarding address should be on the underside of the cover.

7. Suggestions for the Purchase of Weights Meeting This Specification

The National Bureau of Standards tests and certifies the values of weights submitted, but it does not manufacture or sell weights nor does it, except in rare instances, correct those that are not sufficiently accurate.

When ordering weights from a supplier, the purchaser should specify (a) that the weights shall conform to *all* of the requirements for National Bureau of Standards class -----, as described in (give title, number, and date of this publication); (b) the maximum and minimum denominations of each set (see tables 5 and 12 for the most useful

sets), or, when ordering individual weights, their nominal values; (c) gold plating (3.1.1.) when required; special material (3.1 or 4.1) when important; the series (3.4.3.1) when a particular series is required; lifters for individual weights (3.6) when required; basis for adjustment (3.8.1) when significant; and (d) whether the weights are to be sent directly to the National Bureau of Standards for test and certification.

If the weights are to be sent to the Bureau, a written request must be made for the desired test, as described in 6.1.

Appendix 1. Conversion Factors for Units of Mass

Values in boldface are exact values

Units of mass less than pounds and kilograms									
Unit	Grains	Apothecaries' scruples	Pennyweights	Avoirdupois drams	Apothecaries' drams	Avoirdupois ounces			
1 grain	1	0.05	0.041 666 67	0.036 571 43	0.016 666 7	0.002 285 71			
1 apothecary scruple	20	1	0.833 333 3	0.731 428 6	0.333 333	0.045 714 3			
1 pennyweight	24	1.2	1.367 187 5	0.877 714 3	0.4	0.054 857 1			
1 avoirdupois dram	27.343 75	3	1.139 323	1	0.455 729 2	0.062 5			
1 apothecary dram	60	21.875	2.5	2.194 286	1	0.137 142 9			
1 avoirdupois ounce	437.5	24	18.229 17	16	7.291 67	1			
1 apothecary or troy ounce	480	288	20	17.554 28	8	1.097 142 9			
1 apothecary or troy pound	5 760	350	240	210.651 4	96	13.165 714			
1 avoirdupois pound	7 000	350	291.666 7	256	116.666 7	16			
1 milligram	0.015 432 356	0.000 771 618	0.000 643 014 8	0.000 564 383 3	0.000 257 205 9	0.000 035 273 96			
1 gram	15.432 356	0.771 618	0.643 014 85	0.564 383 3	0.257 205 9	0.035 273 96			
1 kilogram	15 432.356	771.617 8	643.014 85	564.383 32	257.205 94	35.273 96			

Units of mass greater than avoirdupois ounces									
Unit	Apothecaries' or troy ounces	Apothecaries' or troy pounds	Avoirdupois pounds	Milligrams	Grams	Kilograms			
1 grain	0.002 083 33	0.000 173 611 1	0.000 142 857 1	64.798 918	0.064 798 918	0.000 064 798 9			
1 apothecary scruple	0.041 666 7	0.003 472 222	0.002 857 143	1 295.978 4	1.295 978 4	0.001 295 978			
1 pennyweight	0.05	0.004 166 667	0.003 428 571	1 555.174 0	1.555 174 0	0.001 555 174			
1 avoirdupois dram	0.056 966 146	0.004 747 178 8	0.003 906 25	1 771.845 4	1.771 845 4	0.001 771 845			
1 apothecary dram	0.125	0.010 416 667	0.008 571 429	3 887.935 1	3.887 935 1	0.003 887 935			
1 avoirdupois ounce	0.911 458 3	0.075 954 861	0.062 5	28 349.527	28.349 527	0.028 349 53			
1 apothecary or troy ounce	1	0.083 333 33	0.068 571 43	31 103.481	31.103 481	0.031 103 48			
1 apothecary or troy pound	12	1	0.822 857 1	373 241.77 1	373.241 77	0.373 241 77			
1 avoirdupois pound	14.583 333	1.215 277 8	1	453 592.427 7	453.592 427 7	0.453 592 427 7			
1 milligram	0.000 032 150 74	0.000 002 679 23	0.000 002 204 62	1	1	0.000 001			
1 gram	0.032 150 74	0.002 679 23	0.002 204 62	1 000 000	1 000	1			
1 kilogram	32.150 742	2.679 228 5	2.204 622 341	1 000 000	1 000	1			

Units of mass greater than avoirdupois ounces						
Units	Avoirdupois ounces	Avoirdupois pounds	Short hundred-weights	Short tons	Long tons	Metric tons
1 avoirdupois ounce	1	0.062 5	0.000 625	0.000 031 25	0.000 027 901 79	0.000 028 349 53
1 avoirdupois pound	16	1	0.01	0.000 5	0.000 446 428 6	0.000 453 592 43
1 short hundredweight	1 600	100	1	0.05	45.359 243	0.045 359 243
1 short ton	32 000	2 000	20	1	907.184 86	0.907 184 86
1 long ton	35 840	2 240	22.4	1.12	1 016.047 04	1.016 047 04
1 kilogram	35.273 957	2.204 622 34	0.022 046 223	0.001 102 311 2	0.000 984 206 4	0.000 984 206 4
1 metric ton	35 273.957	2 204.622 34	22.046 223	1.102 311 2	0.984 206 40	1 000

Appendix 2. Units and Abbreviations for Marking Weights

Name of unit	Accepted abbreviation
Assay ton (29.1667 g).....	AT ¹
Carat (200 mg).....	c
Dram, apothecaries'.....	dr ap
Grain.....	GN ¹
Gram.....	g
Kilogram.....	kg
Milligram.....	mg
Ounce, apothecaries' (480 grain).....	oz ap
Ounce, avoirdupois (437.5 grain).....	oz
Ounce, troy (480 grain).....	oz t
Pennyweight.....	dwt
Pound, avoirdupois.....	lb
Scruple, apothecaries'.....	s ap

¹ In descriptive material the abbreviation for "assay ton" should be written A. T., and "grain" should be spelled out.

DEPARTMENT OF COMMERCE

CIRCULAR
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BUREAU OF STANDARDS
S. W. STRATTON, DIRECTOR

No. 3

**DESIGN AND TEST OF STANDARDS
OF MASS**

3d Edition

[Superseding 2d edition entitled "Verification of Standards of Mass,"
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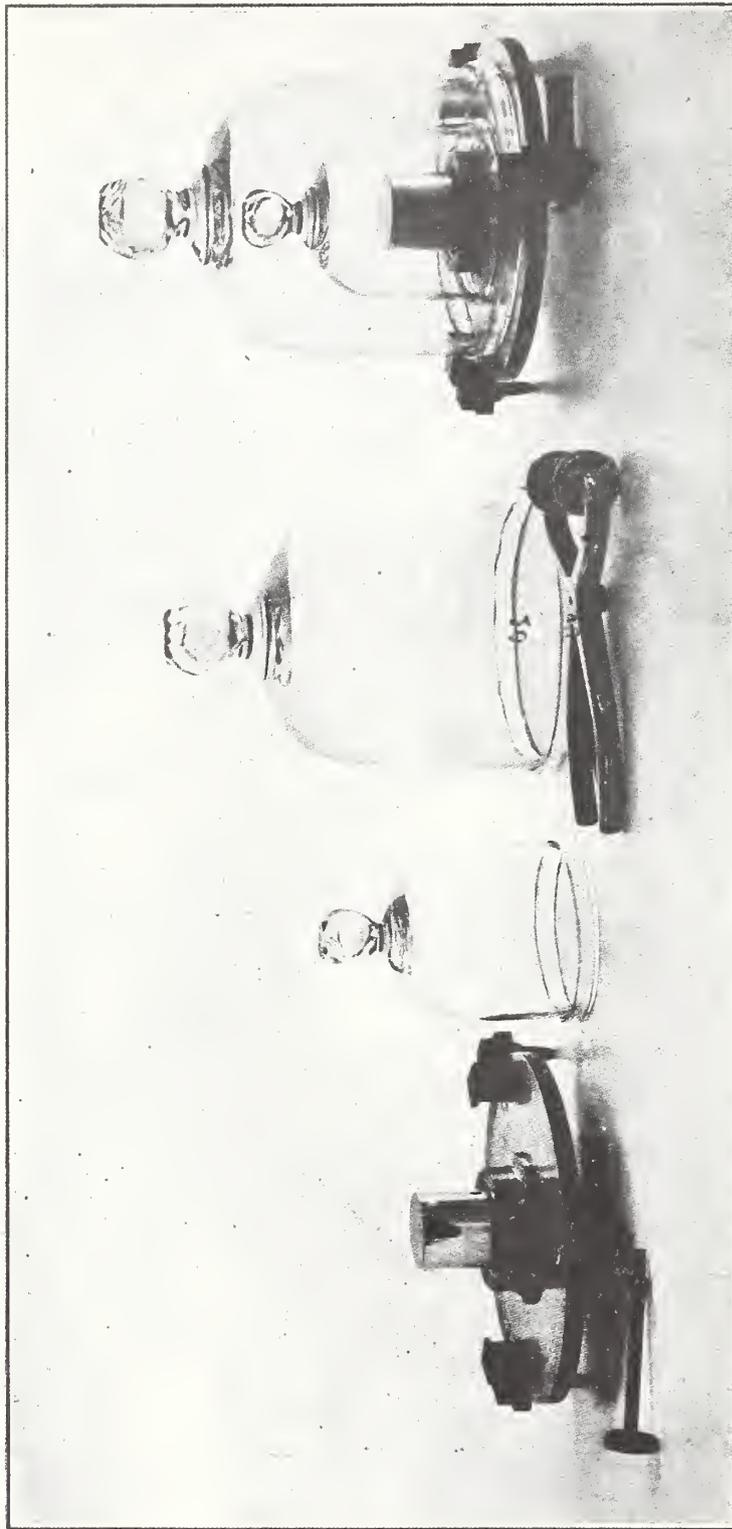


FIG. 1.—National prototype kilograms

From these standards are derived the values of all weights used in this country. These standards were made by an international commission. The material is an alloy of 90 per cent platinum and 10 per cent iridium, which is hard and very resistant to corrosion. It is handled by a special lifter, the faces of which are covered with very soft white plush.

DESIGN AND TEST OF STANDARDS OF MASS

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I. INTRODUCTION

1. SCOPE OF CIRCULAR

This circular is intended to furnish information concerning the testing of standards of mass, and related matters that are frequently the subject of inquiries addressed to the Bureau. It will be impossible to give any but general information within the limited space available. However, any other information at the disposal of this Bureau on the subject of mass standards, bal-

ances, methods of weighing, or the design of special apparatus will be gladly furnished on receipt of specific requests.

Under regulations set forth in subsequent sections, the Bureau tests and certifies the accuracy of standards submitted, but it does not manufacture or sell such standards nor does it, except in rare instances, correct those that are not sufficiently accurate. For further details, see "Regulations governing tests," page 65.

2. FUNDAMENTAL STANDARDS

The fundamental standard of mass adopted by the United States is the international prototype kilogram.¹ This is a cylinder of platinum-iridium kept at the International Bureau of Weights and Measures near Paris in the custody of an international committee. This committee is composed of 14 eminent scientists, elected, each one from a different country, by an international conference that meets every six years. This conference is composed at present of diplomatic representatives of 26 countries, among which are all the leading nations of the world.

Authentic copies of this standard of the same form and composition have been made under the supervision of the international committee and distributed among the countries that support this international organization. Of these authentic copies the United States has two, Nos. 4 and 20. They are kept in a specially constructed vault at the National Bureau of Standards, and are used only when needed to verify the secondary standards of the Bureau.

3. BASIS OF THE CUSTOMARY UNITS

The units of the customary system were based originally on the old brass standard pound of the United States Mint; but the platinum-iridium alloy possesses a constancy far superior to brass and other base-metal alloys, and for this reason the Office of Weights and Measures (now the Bureau of Standards) was authorized in 1893 to base the pound, with its derived and related units, on the international kilogram, using the relation:

One pound avoirdupois = 0.4535924277 kg, the value determined by the joint work of the International Bureau of Weight and Measures and the British Standards Office.

4. DISTINCTION BETWEEN MASS AND WEIGHT

Mass standards are most frequently called "weights," and in accordance with this widely accepted usage, the term will be

¹ For legal status, see B. S. Circular No. 47, p. 62.

freely used in that sense throughout this circular. But confusion often arises from the indiscriminate use of the term "weight" in two other senses; the first, to denote the mass of a body, and the second, to refer to the force with which gravity acts upon such a body. The former is really an incorrect use of the word, due to failure to separate the two ideas. Of course, it is readily understood that when we speak of a 2-ton block of iron, and a tension of 2 tons in a stretched rope, we refer in one case to a mass and in the other to a force. It should be kept in mind that, whereas the mass of a body is a property inherent in the body itself, its weight depends on the attraction of the earth, varying on that account with altitude, latitude, and other circumstances. This variation of weight with locality, though amounting only to about one-half of 1 per cent over the surface of the earth, is perfectly definite, and can be detected by means of a sensitive spring balance.

Weight is measured in units of force. As it is not feasible nor desirable for the purposes of metrology to base the unit of force on some concrete standard force, it is derived from the established units of mass and acceleration. The cgs unit of force, the dyne, is thus derived. It is common, however, to express weight in terms of the gravitational unit of force. This unit is the force of gravity that acts on a body per unit of mass (in vacuo) at any place where the acceleration of gravity is $980.665 \frac{\text{cm}}{\text{sec.}^2} \left(= 32.1740 \frac{\text{ft.}}{\text{sec.}^2} \right)$, in accordance with the definition of standard weight and standard gravity by the International Committee of Weights and Measures.²

5. COMPARISON OF MASSES

Masses are determined by direct or indirect comparison with a standard. The most accurate means of comparing masses is the lever balance. This method involves a comparison, by means of the lever principle, of the respective forces of gravity on the bodies under consideration. Since the masses of bodies are in the same *ratio* as their weights at the same locality, the desired result is obtained. In the same way, any other dynamometer might be

² Procès Verbaux des Séances, Comité Internationale des Poids et Mesures, 1901, p. 173.

The unit was based, as closely as was known at the time (1888), on the intensity of gravity at mean sea level, latitude 45°. This was the specification of standard gravity from the time of the establishment of the metric system until the adoption of $980.665 \frac{\text{cm}}{\text{sec.}^2}$ as an arbitrary value. (See, for example, Delambre *Base du Syst. Met. T., III*, p. 590, 1806.) The value of g at sea level, latitude 45°, as computed from the absolute determination of gravity at Potsdam (1898-1906), differs from the standard value above by 1 part in 30 000.

used as the means of comparison. In all cases the effect of atmospheric buoyancy must be taken into account; for, while the force of gravity is proportional to the mass of a body, the oppositely directed buoyant force of the atmosphere is proportional to its *volume*. In accordance with Archimedes well-known principle of fluid buoyancy, the weight of a body in a fluid (liquid or gas) is diminished by an amount equal to the weight of fluid displaced.

Full details relative to the buoyant effect of the air in its relation to the actual methods used in comparing standards of mass are given in succeeding sections of this circular.

II. CLASSIFICATION OF WEIGHTS

The following classification covers the forms recommended for particular purposes, but weights not falling in any class may under certain conditions be tested. For details applying to this subject, see "Regulations governing tests," page 65.

The following two sections, 1 and 2, indicate roughly the distinctions between the different classes, but the detailed specifications must be consulted for accurate information as to the uses and specifications for each class.

Standards intended for commercial purposes should meet several requirements that are not essential to those intended for scientific work, and the converse also is true. For these reasons, it has been found necessary to divide the weights submitted for test into two general groups: Weights for commercial purposes, and weights for scientific purposes.

1. COMMERCIAL GROUP, CLASSES A, B, C, AND T

This group includes weights that range from the primary standards of the several States to those weights used in ordinary trade. The group is divided into classes A, B, C, and T. These weights have an official or legal status, and the fact that they are correct within the established tolerances will be indicated by a seal impressed on them in some conspicuous place. Weights having screw knobs or other means by which the adjustment is made accessible for alteration without destroying the seal, will not be sealed.³

Each of these classes covers weights that are suitable, among other things, for testing the weights of the next lower class.

³ The verb "to seal," as here used, refers only to affixing a mark as evidence that the weight is correct; it does not mean to make the weight correct and should not be used in that sense. The word "adjust" is employed to express the latter idea.

Class T includes ordinary trade weights, which are not tested by this Bureau except there be no local authority to whom they can be submitted, or in the event of an important controversy.⁴

Class C weights are much more accurate than those of class T, the errors allowed on weights of class C being only one-tenth of those allowed on ordinary trade weights. This class includes, among others, what are commonly called test weights; but these specifications require more reliable types of construction than are sometimes used. On all but the small weights they provide for an adjusting hole closed by a soft metal cap or plug.

Classes A and B both have allowable errors about one-fifth of those of class C, but the correction for each weight of class A is accurately determined when the weight is tested, so that allowance may be made for this error in using the weight. Weights of both classes A and B are without means for easy readjustment, but class B weights may be made with a hard metal adjusting plug, which makes the first adjustment very much easier.

Class A weights are made of a single piece of metal. At present the larger weights of this class are generally made of gold-plated bronze. They are intended for such purposes as primary state standards.

2. SCIENTIFIC GROUP, CLASSES M AND S

This group includes weights used for the more precise weighings in scientific and technical laboratories. It includes such weights as primary laboratory standards, analytical weights, assay weights, etc.⁵ It is divided into classes M and S.

Weights of these classes do not, as a rule, have any legal status, and will not be sealed unless they conform also to the specifications for class A, B, or C.

The small weights of this group are required to be more accurately adjusted than are corresponding weights of the commercial group, but the requirements as to form and material are more liberal.

Class S is intended to cover high-grade analytical weights and similar weights used in scientific and technical laboratories. At present, in sets of this kind the weights of 1 gram and above are universally made with the knobs merely screwed into the body of the weights, and the weights from 20 milligrams down are of aluminum.

⁴Tolerances for these weights are given in Table No. 9, p. 72 of this circular. Complete specifications and tolerances for commercial weights and measures are given in Circular No. 61.

⁵The so-called rough laboratory weights should not be submitted to this Bureau. They can readily be checked in the laboratory or by the local sealer of weights and measures.

Class M is intended to cover weights suitable for scientific work of very high precision where a high degree of constancy is necessary. The accuracy of adjustment required of this class is no higher than that of class S because it is expected that the corrections, which will be determined to a much higher degree of precision, will be applied in using the weights.

Class M weights, like those of class A, are made of a single piece of material, without special provision for adjustment or readjustment.

III. CLASS A—HIGHEST PRECISION COMMERCIAL STANDARDS

Class A comprises commercial standards of the highest type intended for reference standards in work of exacting character. They are recommended for the use of manufacturers of class A or class B weights and as primary standards of States in which the work of inspection is well organized. While these weights are designed with particular reference to reliability and permanence, such qualities can be maintained only by using the weights with extreme care. The soft gold plating ordinarily used as surface protection for this class of weights renders them more susceptible to rapid wear with unskillful or frequent handling than even the unplated brass weights.

1. SPECIFICATIONS

(a) **Material.**—The material of weights of this class must be hard, nonmagnetic, must not be readily susceptible to oxidation and corrosion, and must be of a minimum porosity.

For weights down to 1 g or to 1/32 ounce the density should be between 8.2 and 8.6 g/cm³. For smaller weights there may be used platinum or other suitable material having a density between 5.3 and 22.0 g/cm³. For weights less than 5 mg or 0.1 grain aluminum may be used. All "flat" sheet-metal weights must be of a material sufficiently resistant to need no surface protection against oxidation or corrosion.

(b) **Structure and Form.**—The entire weight must be a single piece, except for the surface plating.

Weights down to and including 1 g or 1/32 ounce must be cylindrical in form with the diameter approximately equal to the height. The difference between the diameter and height must not be over 10 per cent of the latter. Weights of this class must be provided with a rounded knob, small in proportion to the body of the weight. The bottom must be hollowed out symmetrically, leaving a flat ring as a base upon which the weight is to rest.

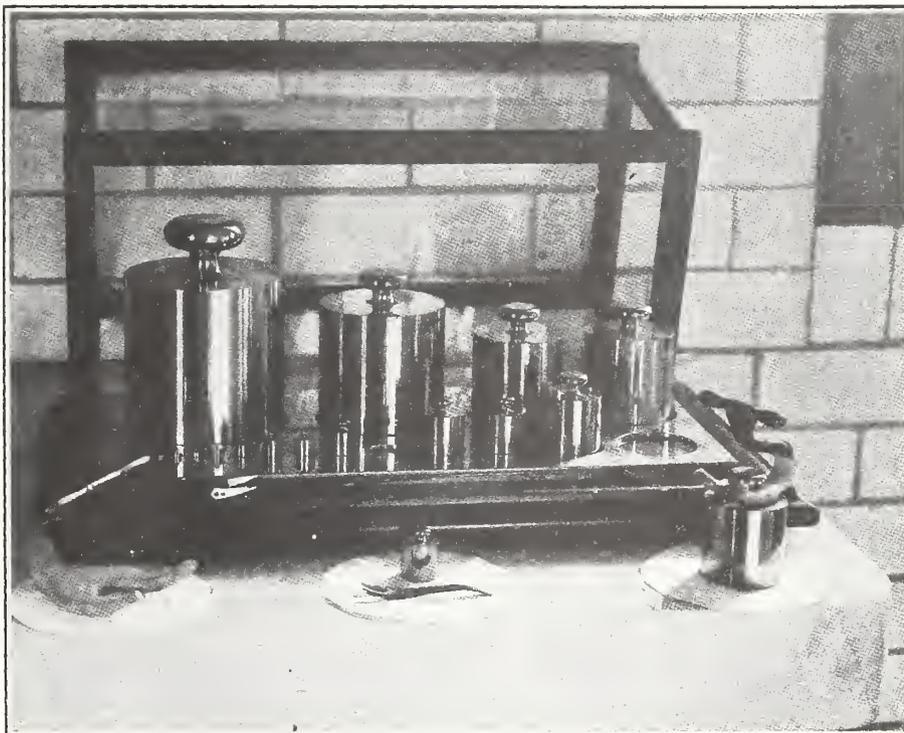


FIG. 2.—*Gold-plated, one-piece standards*

This set of metric standards from 10 kg to 1 g is one of the most important sets in use at this Bureau. It was made in 1893, so that there has been time to test its constancy by many calibrations. More recent Class A weights have the edges rounded much more than these and the knobs of the small weights are taller.

The width of this ring should be from one-tenth to two-tenths of the diameter of the weight.

There must be no other cavities or depressions, except such as are required to designate the value of the weight. All edges and corners must be well rounded.

(For approved designs and dimensions, see p. 27.)

Weights less than 1 g or 1/32 ounce may be "flat," as when cut from sheet metal, with one edge or corner turned up at right angles, to facilitate handling with the forceps. The edges of these weights must be free from sharp or ragged places. Moreover, these small weights must not be cut through by the stamping of the designation, and are not to be unnecessarily thin.

(c) **Surface.**—The entire surface must be smooth and highly polished, and, in the case of new weights, must be free from scratches. All weights not constructed of platinum, aluminum, or metals similarly resistant to atmospheric corrosion, must be protected by a plating of gold, platinum, or similar metal. These weights must be of such material and plating that they will show no discoloration on the surface when placed in boiling water or when dried at a temperature of 110° C (230° F), as is done in preparing them for test.

Nickel plating will not be permitted on class A weights.

(d) **Designation of Value.**—New cylindrical weights down to and including 10 g or 1/4 ounce and sheet-metal weights down to and including 100 mg or 1 grain must be plainly marked with their nominal or intended value. This must include the name of the unit or the accepted abbreviation. Smaller weights must bear the proper number, but the name of the unit may be omitted when the space available does not permit its legible inclusion.

A short table of abbreviations, recommended by this Bureau, is given in Table 8, on page 71. A much more complete list will be found in Circular No. 47.

Duplicate and triplicate weights of a set must be so marked that they can be distinguished with ease and certainty. A common and satisfactory practice is that of using, in addition to the denomination, one and two conspicuous dots on duplicates, and one, two, and three such dots on triplicates. It is preferable to have such marks on each weight rather than to have one weight entirely unmarked. Both the denomination and the distinguishing marks must be on the upper surface of the weights, and these markings must be shallow, broad, and free from burrs and sharp angles.

(e) **Case.**—For weights of this class a suitable dust-proof case should be provided, preferably so arranged that the weights rest upon a base of smooth, hard material such as glass or polished quartz, which can be readily kept clean.

If the case is provided with deep pockets for the cylindrical weights, these pockets must be lined with some soft material, such as plush. The fit must not be tight enough to require any appreciable force in inserting or withdrawing the weights. (See p. 67 in regard to precautions in shipment.)

(f) **Lifter.**—Special lifters should be provided for all weights of class A. The parts that come in contact with the weights may be of wood or ivory, or be covered with velvet or prepared chamois skin, or similar soft material, and must be smooth and free from sharp edges.

(g) **Tolerances.**—Weights of this class must be adjusted within the limits of error prescribed for this class on page 17. All weights must be adjusted so as to balance correct brass standards in air. (See “Buoyant force of the air,” p. 55.) The constancy of the weights must be such that they will not change their values more than one-fifth of the tolerances allowed when subjected to a constancy test of three months.

2. SEALING AND CERTIFICATION

Weights meeting all the requirements will be stamped with the seal shown in Fig. 3.



FIG. 3.—Seal for class A weights

This seal will be impressed on standards tested and approved under this class unless such standards are so small that this is impracticable

The certificate will give the correction to be applied in using each weight. This will be based on the apparent mass as determined by comparison with brass standards in air.

Standards of Mass

TABLE 1.—Precision of Corrections, Class A

[Corrections will be given to the nearest unit in the decimal place indicated in the columns headed "To"]

Metric		Customary									
		Avoirdupois		Grains		Apothecary		Ounces troy		Pennyweights	
De-nomi-nation	To	De-nomi-nation	To	Denomi-nation	To	De-nomi-nation	To	Denomi-nation	To	De-nomi-nation	To
kg	g	ib.	gr.	gr.	gr.	℥	gr.	oz. t.	gr.	dwt.	gr.
20	0.1	50	1	10 000	0.1	12	0.01	1000	1	10 000	1
10	0.01	25	1	5000	0.01	10	0.01	500	1	5000	1
5	0.01	20	0.1	2000	0.01	6	0.01	400	1	4000	0.1
2	0.01	14	0.1	1000	0.01	5	0.01	300	1	3000	0.1
1	0.01	10	0.1	500	0.01	4	0.01	200	0.1	2000	0.1
g	mg	8	0.1	200	0.001	3	0.01	100	0.1	1000	0.1
500	1	7	0.1	100	0.001	2	0.01	50	0.1	500	0.1
200	1	5	0.1	50	0.001	1	0.01	40	0.1	400	0.01
100	1	4	0.1	20	0.001	℥		30	0.1	300	0.01
50	1	3	0.1	10	0.001	6	0.01	20	0.01	200	0.01
20	0.1	2	0.1	5	0.001	4	0.01	10	0.01	100	0.01
10	0.1	1	0.01	2	0.0001	3	0.01	5	0.01	50	0.01
5	0.1	oz.		1	0.0001	2	0.001	4	0.01	40	0.01
2	0.1	10	0.01	0.5	0.0001	1	0.001	3	0.01	30	0.01
1	0.1	8	0.01	0.2	0.0001	℥		2	0.01	20	0.01
mg		5	0.01	0.1	0.0001	2	0.001	1	0.01	10	0.01
500	0.01	4	0.01	0.05	0.0001	1	0.001	0.5	0.01	5	0.001
200	0.01	2	0.01	0.02	0.0001			0.2	0.001	4	0.001
100	0.01	1	0.01	0.01	0.0001			0.1	0.001	3	0.001
50	0.01	$\frac{1}{2}$	0.01	0.005	0.0001			0.05	0.001	2	0.001
20	0.01	$\frac{1}{4}$	0.001	0.002	0.0001			0.02	0.001	1	0.001
10	0.01	$\frac{1}{8}$	0.001	0.001	0.0001			0.01	0.001		
5	0.01	$\frac{1}{16}$	0.001					0.005	0.0001		
2	0.001	$\frac{1}{32}$	0.001					0.002	0.0001		
1	0.001	$\frac{1}{64}$	0.001					0.001	0.0001		
0.5	0.001	$\frac{1}{128}$	0.001					0.0005	0.0001		
0.2	0.001							0.0002	0.0001		
0.1	0.001							0.0001	0.0001		

IV. CLASS B.—PRECISE COMMERCIAL STANDARDS

Class B weights are intended as high-grade commercial standards to be used in cases where extreme precision is not necessary or warranted. They are suitable for primary standards for local inspectors, for manufacturers of commercial weights and measures, and for high-grade working standards such as would be needed in testing class C weights.

1. SPECIFICATIONS

(a) **Material.**—Weights of this class must be made of material that is not readily oxidized or corroded. The material must be moderately hard (for example, the better grades of brass or bronze) and must be nonmagnetic. Nickel, though magnetic, may be used as a plating material.

The material of the weights must be free from pits or pores and must show no effects of porosity, such as the white patches that

sometimes form on porous weights electroplated with gold or nickel.

The density of the weights, including any cavity under the adjusting plug, shall be between 8.2 and 8.6 g/cm³ except in the case of weights below 1 g or 1/32 ounce. For these smaller weights platinum or other suitable material having a density between 5.3 and 22.0 g/cm³ shall be used. For weights below 50 mg aluminum may be used. All flat sheet-metal weights shall be of a material whose surface need not be protected against oxidation or corrosion.

(b) **Structure and Form.**—Metal weights shall consist of a single piece, except as described below under “Means of adjustment.” Weights of this class may be made of glass or similar material filled with shot or otherwise loaded to the proper mass. In such weights the opening to the interior must be closed by fusing the material of which the shell is composed.

Weights down to and including 1 g or 1/32 ounce shall be cylindrical in form with the diameter approximately equal to the height. The difference between the diameter and height must not be over 10 per cent of the latter. Weights of this class must be provided with a rounded knob small in proportion to the body of the weight. The bottom must be hollowed out in a symmetrical manner, leaving a flat ring as a base. The width of the ring should be from one-tenth to two-tenths the diameter of the weight. There must be no other cavity or depression except as specified below. All edges and angles must be well rounded.

Smaller weights may be of flat sheet metal with an edge or corner turned up at right angles to facilitate handling with the forceps, or they may be of wire bent in shape to indicate the denomination. They must be free from sharp or ragged edges, must not be cut through in marking the designation on them, and must not be unnecessarily thin.

ADJUSTING PLUG.—For metal weights of this class, special means of adjustment may be provided. This, if used, shall consist of a single cavity in the top of the weight, closed by a plug of brass or similar hard, tough metal securely driven into place.

This plug shall be air and water tight and shall fit so tightly as to make its removal impossible without the certainty of destroying the seal. When the plug is driven into place its top must be on a level with, or very little below, the surface of the weight, and no cracks, sharp angles, or burrs must remain. The diameter of the

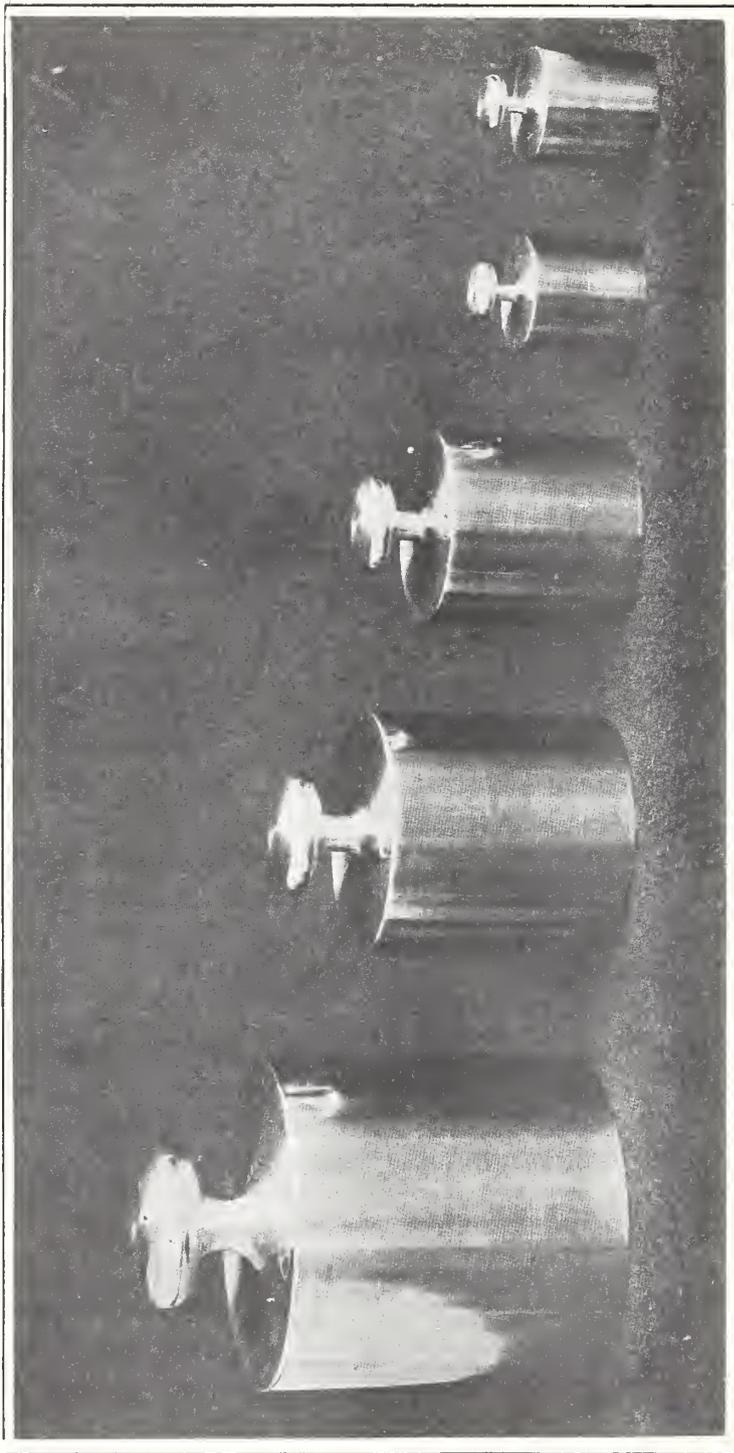


FIG. 4.—*Nickel-plated, class B weights*

These are from a set of Tobin bronze secondary standards made in 1911 and used in this Bureau in a great deal of routine testing. The knob and body of the weight are a single piece of metal. All but the small weights of the set have the typical Class B driven plug. Their constancy has been very satisfactory

plug must conform in size to the dimensions given in the following table:

	Diameter of plug
Under 500 g or 1 pound	2 mm to 5 mm or $\frac{3}{32}$ inch to $\frac{1}{16}$ inch
From 500 g to 10 kg or from 1 to 20 pounds....	5 mm to 10 mm or $\frac{1}{16}$ inch to $\frac{3}{8}$ inch
Above 10 kg or 20 pounds and less than 50 pounds	10 mm to 16 mm or $\frac{3}{8}$ inch to $\frac{5}{8}$ inch
50 pounds and above	16 mm to 25 mm or $\frac{5}{8}$ inch to 1 inch

The transverse cross section of the plug must be circular. The sides of the plug and cavity must be smooth, to insure tightness against leakage of air. The plug should have a slight taper and a driving fit. When inserted in the hole and forced down by a pressure exerted by the fingers, the top of the plug must remain above the surface of the weight by an amount equal to at least one-half the diameter of the plug, so that it is necessary to drive it down into place.

In order that the plug may seat firmly enough to take the impression of the seal, its final position must be determined by a shoulder. A common and satisfactory form is indicated in Fig. 6. The form of cavity shown in Fig. 6 can be easily drilled and accurately counterbored. The fit must not be tight enough to cause the metal about the hole to bulge when the plug is driven into place. The length of the plug from its upper end to the shoulder must not be less than its diameter.

The plug should not be driven into place in the case of new weights submitted to this Bureau for test, first, because the Bureau wishes to inspect the design and workmanship before sealing the weight; and second, because if the weight is not properly adjusted, the adjustment can be more readily corrected by the manufacturer before the plug is driven than afterwards.

The adjusting cavity should not contain any loose material. The adjusting cavities and plugs of metal weights and the volumes of filled glass weights must be so regulated as to keep the average density of the weight within the limits specified under "Material."

(c) **Surface.**—The surface must be smooth and weights, if made of metal, must be carefully polished. If the weights are

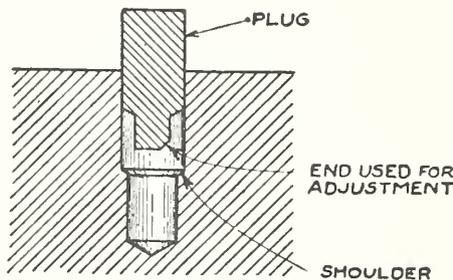


FIG. 6.—Design of class B adjusting plug

This plug should preferably be of the same material as the rest of the weight. It should be made with a good "driving fit," and may be tapered slightly if desired. The plug is shown in place ready to be driven.

made of material that under ordinary conditions tarnishes to a greater extent than nickel or aluminum, the surface must be protected by a plating of gold, platinum, nickel, or other suitable metal, or by lacquer. If lacquer is used it must be hard, of only moderate thickness, transparent, and not easily chipped. Flat sheet-metal weights, however, are not to be lacquered.

(*d*) **Designation of Value.**—New cylindrical weights down to and including 10 g or $\frac{1}{4}$ ounce and sheet-metal weights down to and including 100 mg or 1 grain must be plainly marked with their nominal mass or "value." This must include the name of the unit or the accepted abbreviation. Smaller cylindrical and flat weights must bear the proper number, but where space is limited, the name of the unit may be omitted. Wire weights need not be marked but must be bent in such forms as to suggest their value.

Duplicate and triplicate weights of a set must be so marked that they can be distinguished with ease and certainty. A common and satisfactory practice is that of using in addition to the denomination, one and two conspicuous dots on duplicates, and one, two, and three such dots on triplicates. It is preferable to have such marks on each weight rather than to have one weight entirely unmarked. Both the denomination and the distinguishing marks must be on the upper surface of the weights. These markings must be shallow, broad, and free from burrs and sharp angles.

(*e*) **Case.**—The weights should be kept in a dust-proof case or box, and each weight must have a separate pocket. The pockets, if deep, must be lined with some soft material, such as velvet, or chamois skin from which all grease has been carefully removed. The fit must not be tight enough to require any appreciable force in inserting or removing the weights. (See p. 67 for precautions in shipment.)

(*f*) **Lifters.**—Special lifters must be provided. The parts that come into contact with the weight may be of wood or ivory or may be covered with velvet or prepared chamois skin, or similar soft material, and must be without sharp edges or corners.

(*g*) **Tolerances.**—Weights of this class must be adjusted within the limits of error prescribed below.

All weights must be adjusted according to the apparent mass, as this mass would be determined by comparison with brass standards in air. (See "Buoyant force of the air," p. 55.)

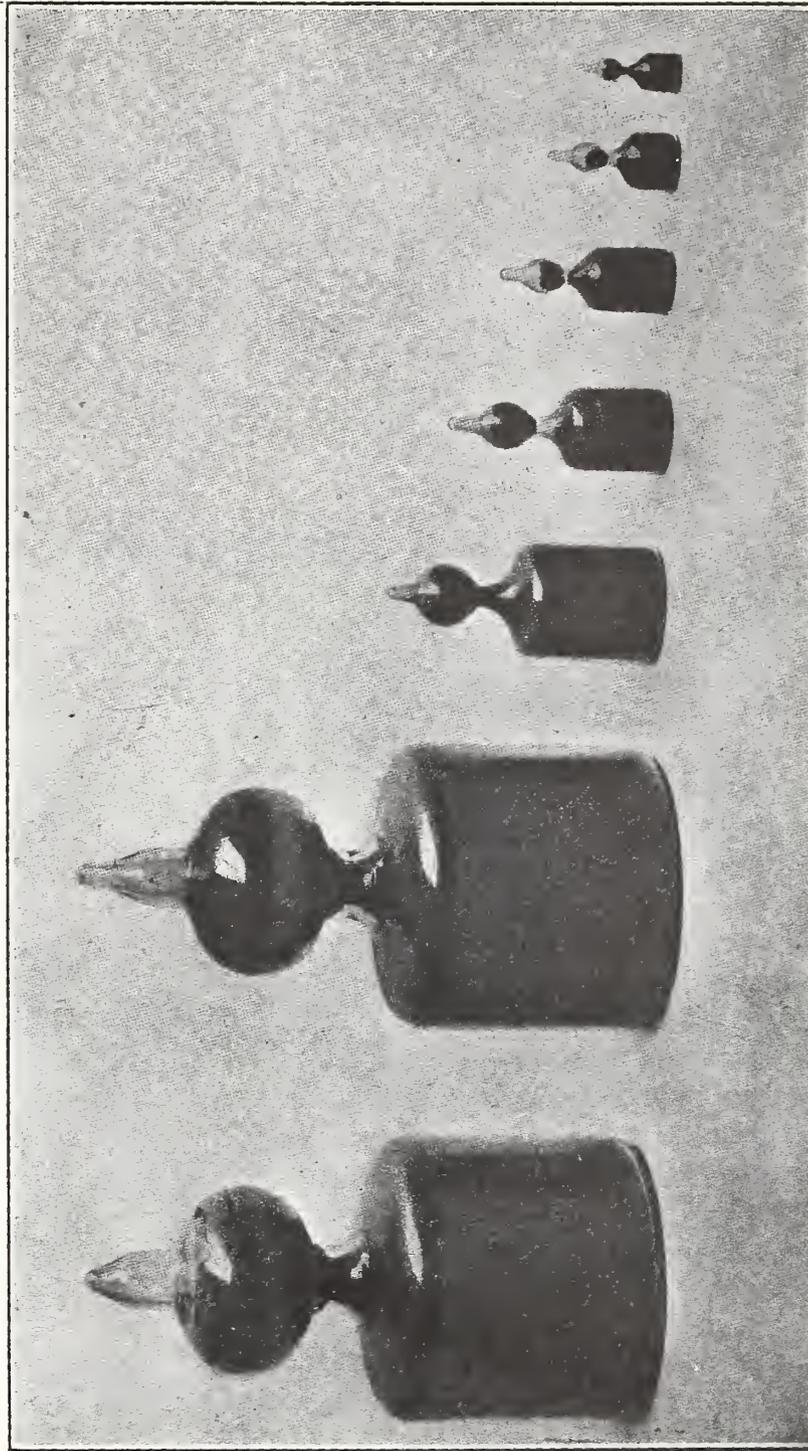


FIG. 5.—*Glass-cased weights*

These weights are made of glass shells filled with shot. The surface is easily cleaned and wears less than that of metal weights under ordinary office handling. These weights are even more reliable than the metal weights of this class because as a rule they are not injured at all or else the injury is so great that there is no doubt about it. They can easily be made strong enough to stand ordinary handling and at a low cost. They are made and used in Austria, and it is hoped that American manufacturers will take up the making of similar weights.

Standards of Mass

TABLE 2.—Tolerances for Classes A and B: Precise Commercial Standards
 [The maximum error allowable on each weight is given in the columns headed "Tolerance"]

Metric		Customary												Carats			
		Avoirdupois			Grains			Apothecary			Troy ounces					Pennyweights	
Denomi- nation	Tolerance	Denomi- nation	Tolerance	Denomi- nation	Tolerance	Denomi- nation	Tolerance										
kg	mg	lb.	gr.	gr.	gr.	oz. ap.	gr.	oz. t.	gr.	dwt.	gr.	c	mg				
20	120	50	2.0	10 000	0.2	12	0.2	1000	2	10000	2	2500	14				
10	60	25	1.2	5000	0.2	10	0.2	500	2	5000	1	2000	14				
5	30	20	1.2	2000	0.1	8	0.1	400	1	4000	1	1000	12				
2	30	10	0.8	1000	0.06	6	0.1	300	1	3000	0.6	500	8				
1	20	5	0.6	500	0.04	5	0.1	200	1	2000	0.4	200	6				
g	14	8	0.6	200	0.03	4	0.1	100	0.6	1000	0.4	100	4				
200	8	4	0.4	100	0.02	3	0.08	50	0.4	500	0.3	50	2				
100	6	3	0.4	50	0.01	2	0.06	40	0.3	400	0.2	20	1.5				
50	4	2	0.3	20	0.006	1	0.04	30	0.3	300	0.2	10	1.0				
20	2	1	0.2	10	0.004			20	0.2	200	0.2	5	0.6				
10	1.5	oz.	0.2	5	0.003	dr. ap.	0.04	10	0.2	100	0.1	2	0.4				
5	1.0	10	0.2	2	0.002	6	0.04	5	0.1	50	0.08	1	0.2				
2	0.6	8	0.1	1	0.001	4	0.04	4	0.1	40	0.06	0.5	0.14				
1	0.4	5	0.1	0.5	0.001	3	0.02	3	0.08	30	0.06	0.2	0.10				
		4	0.1	0.2	0.0005	2	0.02	2	0.06	20	0.04	0.1	0.06				
		2	0.06	0.1	0.0004	1	0.01	1	0.04	10	0.04	0.05	0.04				
mg	0.3	1	0.04	0.05	0.0003	s. ap.	0.01	0.5	0.04	5	0.02	0.02	0.03				
200	0.14	1	0.04	0.02	0.0003	2	0.01	0.4	0.03	4	0.02	0.01	0.02				
100	0.10	1	0.02	0.01	0.0003	1	0.006	0.3	0.02	3	0.01	0.01	0.01				
50	0.07	1	0.01	0.005	0.0002			0.2	0.01	2	0.01	0.01	0.01				
20	0.04	1	0.01	0.002	0.0002			0.1	0.01	1	0.01	0.01	0.01				
10	0.03	1	0.01	0.001	0.0002			0.05	0.006	0.05	0.006	0.006	0.006				
5	0.02	1	0.004					0.05	0.005	0.05	0.005	0.005	0.005				
2	0.01	1	0.002					0.03	0.005	0.03	0.005	0.005	0.005				
1	0.01	1	0.001					0.02	0.004	0.02	0.004	0.004	0.004				
0.5	0.01	1	0.001					0.01	0.002	0.01	0.002	0.002	0.002				
0.2	0.01	1	0.001					0.01	0.002	0.01	0.002	0.002	0.002				
0.1	0.01	1	0.001					0.006	0.006	0.006	0.006	0.006	0.006				

NOTE.—Many of these denominations are not in common use and can not be recommended. They are, however, included here for the sake of completeness. The tolerance on denominations not in this list and not equivalent to any in the list will be that of the next smaller weight listed.

2. SEALING AND CERTIFICATION

Weights meeting all the requirements will be stamped with the seal shown in Fig. 7.

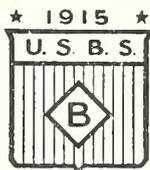


FIG. 7.—Seal for class B weights

This seal will be impressed on standards tested and approved under this class, unless such standards are so small that this is impracticable

The certificate will state that the weights are correct within the tolerances prescribed for this class.

V. CLASS C.—COMMERCIAL TEST WEIGHTS

Class C weights are designed for working standards for sealers of weights and measures for use in testing trade weights and weighing instruments and in work where weights of a superior grade are desired for commercial or industrial purposes.

A very important feature of these weights is that they can easily be readjusted, not, however, without destroying the seal.

1. SPECIFICATIONS

(a) **Material.**—Weights of class C of 5 g or 60 grains and above must be constructed of material at least as hard as brass. Steel may be used for weights of 1 pound or $\frac{1}{2}$ kg, and above, and cast iron for those of 20 pounds, and above. Satisfactory materials are nickel, brass, bronze, Monel metal, the nonrusting nickel steels, and other alloys which are not readily corroded by the atmosphere or by handling with bare hands. The density of the weights as a whole shall be between 7.0 and 9.0 g/cm³; that is to say, the volume, including the cavity and cap for adjustment, must not differ greatly from that of a solid brass or iron weight. Weights below 5 g or 60 gr. may be of the same materials or of aluminum, nickel, platinum, or similar materials, provided that the materials used for sheet-metal weights be such that their surface need not be protected against oxidation or corrosion.

(b) **Structure and Form.**—The construction of the weight must be such that no part of the weight can be replaced or removed without distinctly defacing the weight or destroying the seal. It is to be noted that weights having knobs or handles merely

Standards of Mass

screwed into the body of the weight and those having movable rings for handles or similar loose parts are excluded from this class.⁶

No special form will be required. Manufacturers and dealers should submit to this Bureau samples or detail drawings of the types of weights that they wish to submit for test. Notice of the acceptance of any type will be sent to all manufacturers or dealers who request it, and to the heads of the weights and measures departments of each State. This provision is made in order to allow the acceptance of unusual forms that are suited to certain special lines of work, and yet can not be included in general specifications.

MEANS OF ADJUSTMENT.—Weights above 50 g or 2 ounces must be provided with a single adjusting hole, which must open on the upper surface or side of the weight, but in no case on its bottom face.

Weights of 50 g, or 2 ounces and less, may be provided with an adjusting hole when this is desired and can be done without violating the requirements given below. The adjusting hole must be closed by a cap or plug upon which the regular seal for this class of weights can be stamped.

SEALING CAPS.—The cap or plug must be so constructed that it can not be removed without destroying the seal. A screw, covered by a cap of some soft metal such as lead, in order that readjustments may be easily made, is recommended. The outer surface of this cap or plug must not project above the surface of the weight. It must be either on a level with the surface or very slightly below it.

There must be a flat space equal to at least one-third of its diameter between the edge of the cap or plug and the edge of the weight, the edge of the knob, or the projection of the knob on the upper surface of the weight. In new weights there should be an empty space in the adjusting hole large enough to contain lead equal in mass to at least five times the tolerance applying to the weight.

Samples or drawings showing the method of closing the adjusting holes, the denominations and shapes of the weights on which this method is to be used, and the size of the various parts as they

⁶Weights which have a screw knob or handle will be accepted for test, provided the knob or handle is pinned in place in such manner as to prevent its removal. The pin must fit snugly when driven in to the weight, and must project slightly above the body of the weight. The projecting part of the pin must be rounded.

will be used in weights of each of these denominations, must be submitted for approval in connection with, or as a part of, those submitted to cover the requirement stated above under "Shape."

(c) **Surface.**—The surface must be smooth and well finished. Weights that are constructed of material that might be badly tarnished or corroded by the air, or by handling with bare hands, must be provided with suitable surface protection. Plating with a metal such as nickel may be used, or a coating may be applied of transparent paint or lacquer which will be hard and not likely to chip, provided, however, that opaque paints may be used on weights of 20 pounds and above. Weights of rectangular design need not have their surface machined, on condition that the irregularities of the surface be slight and not of sharp contour. Neither lacquer nor paint of any kind is to be used on the sheet-metal weights.

(d) **Designation of Value.**—The denomination of each weight must be clearly marked upon it. This must include the number in all cases, and the name of the unit in all except cylindrical weights below 10 g or $\frac{1}{4}$ ounce, and sheet-metal weights below 100 mg or 1 gr. (For a list of abbreviations recognized by this Bureau, see p. 71.)

(e) **Case.**—It is recommended that weights of this class, except the rectangular-type 50-pound weights, be provided with a covered case in which they may be kept and carried. A separate pocket lined with velvet, felt, or leather should be provided for each cylindrical weight. The fit must not be tight enough to require any appreciable force in inserting or removing the weights. (See p. 67 in regard to precautions in shipment.)

(f) **Lifters.**—Special lifters need not be provided unless desired, because class C weights are intended to be handled with bare hands. However, such lifters, as well as other means of protecting the weights, should be provided if the weights are to be used where they would otherwise become dirty.

(g) **Tolerances.**—Weights of this class must be adjusted within the limits of error prescribed for this class on page 21. All weights must be adjusted according to the apparent mass, as this mass would be determined by comparison with brass standards in air. (See "Buoyant force of the air," p. 55.)

Standards of Mass

2. SEALING AND CERTIFICATION

Weights meeting all the requirements will be stamped with the seal shown in Fig. 8.



FIG. 8.—Seal for class C weights

This seal will be impressed on standards tested and approved under this class, unless such standards are so small that this is impracticable

The certificate will state that the weights are correct within the tolerances prescribed for this class.

TABLE 3.—Tolerances for Class C—Commercial Test Weights

[The maximum error allowable on each weight is given in the columns headed "Tolerance"]

Metric		Customary										Carats	
		Avoirdupois		Grains		Apothecary		Ounces troy		Pennyweights			
De-nomination	Tolerance												
kg	mg	lb.	gr.	gr.	gr.	oz. ap.	gr.	oz. t.	gr.	dwts.	gr.	c	mg
20	600	50	10	10 000	1	12	1	1000	10	10 000	10	2500	70
10	400	25	6	5000	1	10	1	500	10	5000	5	2000	60
5	250	20	6	2000	0.5	6	0.5	400	5	4000	5	1000	40
2	150	10	4	1000	0.3	5	0.5	300	5	3000	3	500	30
1	100	8	3	500	0.2	4	0.5	200	5	2000	3	200	20
		5	3	200	0.15	3	0.4	100	3	1000	2	100	10
g	70	4	2	100	0.10	2	0.3	50	2	500	1.5	50	7
200	40	3	2	50	0.05	1	0.2	40	1.5	400	1.0	20	5
100	30	2	1.5	20	0.03	dr. ap.		30	1.5	300	1.0	10	3
50	20	1	1.0	10	0.02	6	0.2	20	1.0	200	1.0	5	2
20	10	oz		5	0.015	5	0.2	10	1.0	100	0.5	2	1
10	7	10	1.0	2	0.010	4	0.2	5	0.5	50	0.4	1	0.7
5	5	8	0.5	1	0.005	3	0.1	4	0.5	40	0.3	0.5	0.5
2	3	5	0.5	0.5	0.005	2	0.1	3	0.4	30	0.3	0.2	0.3
1	2	4	0.5	0.2	0.0025	1	0.05	2	0.3	20	0.2	0.1	0.2
mg		2	0.3	0.1	0.0020	s. ap.		1	0.2	10	0.2	0.05	0.15
500	1.5	1	0.2	0.05	0.0015	2	0.05	0.5	0.2	5	0.1	0.02	0.10
200	0.7	½	0.2	0.02	0.0015	1	0.03	0.4	0.15	4	0.1	0.01	0.05
100	0.5	¼	0.1	0.01	0.0015			0.3	0.10	3	0.05		
50	0.35	⅛	0.05					0.2	0.10	2	0.05		
20	0.20	⅜	0.05					0.1	0.05	1	0.03		
10	0.15	⅜	0.05					0.05	0.03				
5	0.10	⅜	0.02					0.04	0.025				
2	0.05	⅜						0.03	0.025				
1	0.04							0.02	0.020				
								0.01	0.015				
								0.005	0.010				
								0.004	0.008				
								0.003	0.007				
								0.002	0.005				
								0.001	0.005				

VI. CLASS M—HIGH-PRECISION LABORATORY
STANDARDS

This class includes weights suitable for reference standards for work of the highest precision, and for use in investigations demanding especially accurate determinations of mass.

1. SPECIFICATIONS

The material of which the weights are composed must be hard, nonmagnetic, and not readily oxidized or corroded by the atmosphere. It must show no traces of porosity. Gold, though a soft material, may be used for plating. Platinum is the material best suited for the smaller weights even as low as 1 mg, but aluminum will serve for those of 20 mg and less. All flat sheet-metal weights must be of a material whose surface need not be protected against oxidation or corrosion.

The entire weight must be a single piece, except for the plating that is permitted on weights of bronze and similar metals. The weights must be free from deep depressions, sharp angles, or other features that jeopardize their constancy. Weights of brass, bronze, or other metals that tarnish on exposure to the atmosphere must be plated with gold, platinum, or other suitable metal. There must be no darkening of the surface, and no spots of any kind may appear when the weights are placed for some time in boiling water and subsequently dried at a temperature of 110° C, as is done in preparing them for test.

The entire surface of the weight shall be smooth and highly polished. If the denomination is marked upon it, the lines of the marking must be shallow and free from burrs and sharp angles. (For list of accepted abbreviations, see Table 8, p. 71.)

Special lifters shall be provided. The parts that come in contact with the weights may be made of wood or ivory, or may be covered with velvet or similar soft material and must be smooth and without sharp edges.

Weights of this class must be adjusted within the limits of error prescribed below. They may be adjusted either according to their true mass or according to their apparent mass as determined by comparison with brass standards in air; that is, according to "weight in air against brass." The latter is generally preferable, chiefly because of the convenience in using small platinum and aluminum weights in connection with brass weights.

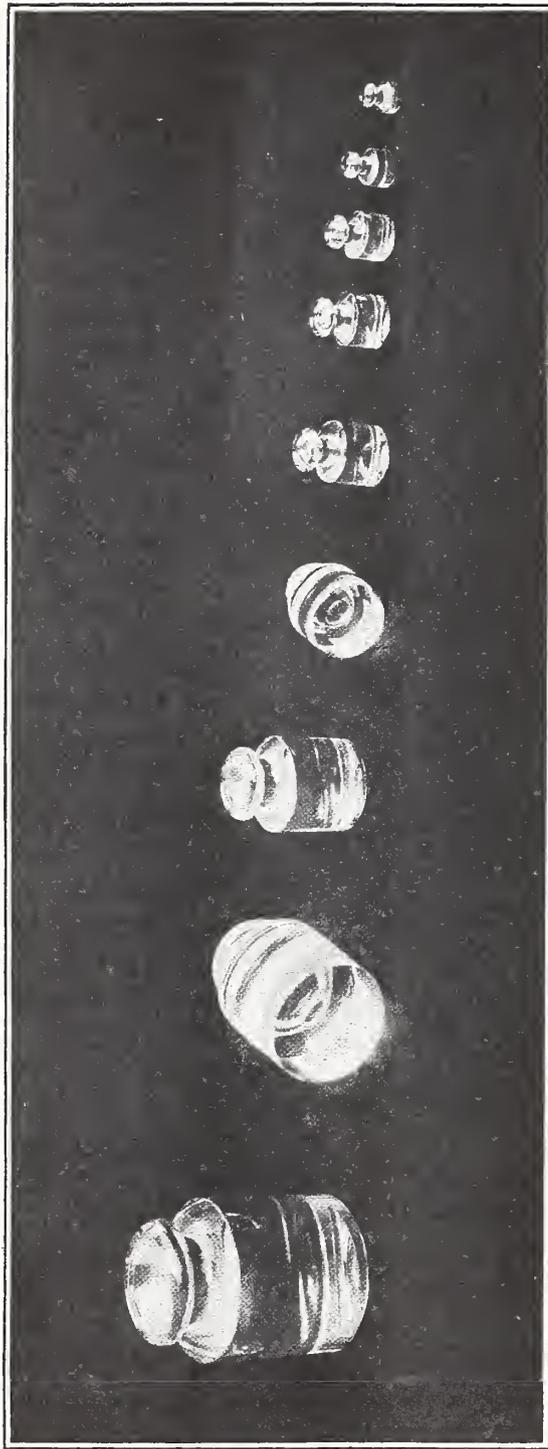


Fig. 9.—*Quartz-crystal weights*

Crystal quartz is occasionally used for weights of the grade covered by Class M. These weights are very constant, but they are easily electrified and have such large volumes that the corrections for the buoyant force of the air are very large when these weights are compared with brass or platinum standards. Platinum-iridium, platinum, or gold-plated bronze (see Figs. 1 and 2) are probably preferable in the majority of cases.

Standards of Mass

2. CERTIFICATION AND SEALING

Class M weights when they incidentally conform to class A, B, or C will be sealed accordingly, when this is requested.

The Bureau will, in general, determine the volumes of all weights except the fractions of a gram. This must be done in order to determine the last figure of the corrections. The volumes, or the mean densities assumed, will be given in the certificate, as will also the atmospheric conditions under which the test was made.

TABLE 4.—Precision of Corrections and Tolerances, Class M: High-Precision Laboratory Standards

[Corrections will be given to the nearest unit in the decimal place indicated.^a The maximum error allowable on each weight is indicated in the column headed "Tolerance"]

Denomination	Precision of corrections	Tolerance	Denomination	Precision of corrections	Tolerance
kg	g	mg	mg	mg	mg
20	0.01	100	500	0.001	0.05
10	0.01	50	200	0.001	0.05
	mg		100	0.001	0.05
5	1	30	50	0.001	0.03
2	1	10	20	0.001	0.03
1	1	5	10	0.001	0.02
g			5	0.001	0.02
500	1	3	2	0.001	0.01
200	0.1	1	1	0.001	0.01
100	0.1	0.5	0.5	0.001	0.01
50	0.1	0.3	0.2	0.001	0.01
20	0.01	0.2	0.1	0.001	0.01
10	0.01	0.15			
5	0.01	0.15			
2	0.01	0.10			
1	0.01	0.10			

^a Weights may in certain cases be certified to a higher degree of precision than that indicated in the table, but this involves additional labor and expense. Arrangements for such work should be made by correspondence before the weights are submitted, and the request must state the character of the weight and of the work for which it is needed, and the degree of precision desired. The Bureau will not undertake to determine weights "as accurately as possible" nor to other similarly indefinite degrees of precision.

Two sets of corrections will be given on the certificate:

One of these will refer to the apparent mass as this would be found by comparison with brass standards in air. These corrections will be calculated for air whose density is 1.2 mg per milliliter. (See p. 55 for complete details.)

The apparent mass of a weight, determined as above, is of course equal to the true mass if the volume of the weight, or its density, is equal to that of the standard when the weight is tested.

In determining the other corrections, full allowance will be made for the buoyant effect of the air; that is, the observations will be "reduced to vacuo." These corrections will therefore refer to the true mass of the weights.

When the density of the weight can safely be assumed, the following values will be used. The coefficients of expansion given in the same table will be assumed in all cases.

Material	Density in grams per cubic centimeter	Coefficient of cubical expansion per degree centigrade
Brass or bronze	8.4 at 0° C	0.000054
Platinum or platinum-iridium.....	21.5 at 0° C	0.000026
Aluminum.....	2.7 at 0° C	0.000069
Quartz (crystal)	2.65 at 20° C	0.000033

VII. CLASS S—ANALYTICAL AND SIMILAR LABORATORY WEIGHTS

This class includes such weights as are commonly used in physical and chemical laboratories for most of the accurate weighing.

1. SPECIFICATIONS

(a) **Material.**—These weights must be made of material that is not rapidly corroded by the air. It must be hard, nonporous, and nonmagnetic. (Nickel, though magnetic, may be used for plating.) The larger weights down to and including 1 g must be of brass or bronze, or of a material having approximately the density of these metals. Below 1 g platinum may be used, and below 50 mg aluminum. All flat sheet-metal weights must be of a material whose surface need not be protected against oxidation or corrosion.

(b) **Structure and Form.**—These weights may have any of the common forms, provided they do not show features that might reduce their reliability. There must be no hole on the underside. Screw-knob weights may be tested under this class.

(c) **Surface.**—The surface must be smooth and polished. Unless the material of the weights is at least as resistant to atmospheric corrosion as nickel or aluminum, the weights must be plated with metals such as platinum, gold, or nickel, or must be lacquered.

LACQUERED WEIGHTS.—Lacquer, if used, must be hard, of moderate thickness, and smooth. Lacquer must not be used, however, on "flat" sheet-metal weights. Lacquer absorbs variable amounts of moisture according to the humidity of the air. The variations due to this cause may be appreciable in weights of this class. Since the character of the lacquer varies greatly, definite figures can not be given; but for moderate ranges of humidity, say 40 to 70 per cent, a change of about 0.1 mg may be

expected on a 100 g weight, and slightly more on an equivalent summation of smaller weights.

(d) **Designation of Value.**—The nominal mass or “value” of each weight must be plainly marked upon it, except in the case of riders and other wire weights. On the largest weight of a set, at least, and on flat sheet-metal weights down to and including 100 mg, this marking should include the name of the unit. For list of abbreviations approved by this Bureau, see page 71. On other weights the number alone will be sufficient.

The lines forming the characters must be shallow, broad, and free from burrs and sharp angles.

Duplicate and triplicate weights of a set must be so marked that they can be distinguished with ease and certainty. A common and satisfactory practice is that of using, in addition to the denomination, one and two conspicuous dots on duplicates, and one, two, and three such dots on triplicates. It is preferable to have such marks on each weight rather than to have one weight entirely unmarked.

(e) **Case.**—It is recommended that a dust-proof case or box be provided and that the pockets for the cylindrical weights be lined with some soft material, such as velvet. When a case is provided there must be a separate pocket for each weight and these pockets must be large enough so that no appreciable force is required in removing or inserting the weights. (See p. 67 for precautions in shipment.)

(f) **Lifters.**—Special lifters must be provided. The parts that come in contact with the weights may be of wood or ivory, or may be covered with velvet or similar soft material, and must be smooth and without sharp edges.

(g) **Tolerances.**—Weights of this class must be adjusted within the tolerances given below. They may be adjusted either according to their true mass or according to their apparent mass as determined by comparison with brass standards in air—that is, according to “weight in air against brass.” The latter is generally preferable, chiefly because of the convenience in using small platinum and aluminum weights in connection with brass weights.

2. CERTIFICATION AND SEALING

Class S weights, when they incidentally conform to the requirements of class A, B, or C, will be sealed accordingly when such sealing is requested.

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Two sets of corrections will be given on the certificate. One of these will refer to the apparent mass as this would be found by comparison with brass standards in air. These corrections will be calculated for air whose density is 1.2 mg per milliliter. (For details see p. 55.)

In determining the other corrections allowance will be made for the buoyant effect of the air—that is, the observations will be “reduced to vacuo.” For this purpose an approximately correct density will be assumed for weights of each different material. These latter corrections will therefore refer to the true mass of the weights.

For weights of brass or of materials of the same density as brass the two sets of corrections are the same. The densities and coefficients of expansion given on page 24 will be assumed for weights of the materials there listed.

TABLE 5.—Precision of Corrections and Tolerances, Class S: Analytical and Similar Laboratory Weights

[Corrections will be certified to the degree of precision indicated in the column headed “Precision of corrections.” Where the figure 1 is used the values are rounded off to the nearest unit in the decimal place indicated; where the figure 5 is used the values are rounded off to the nearest 5 or 0. The maximum error allowable on each weight is indicated in the column headed “Tolerances”]

Denomination	Precision of corrections	Tolerance	Denomination	Precision of corrections	Tolerance
kg					
20	0.1 g	100 mg	500	0.01 mg	0.05 mg
10	0.01	50	200	0.01	0.05
5	0.01	30	100	0.01	0.05
2	0.01	10	50	0.01	0.03
			20	0.01	0.03
a 1	5 or 1 mg	5	10	0.01	0.02
g			5	0.01	0.02
500	1	3	2	0.01	0.01
200	1	1	1	0.01	0.01
a 100	0.5 or 0.1	0.5	0.5	0.01	0.01
50	0.1	0.3	0.2	0.01	0.01
20	0.1	0.2	0.1	0.01	0.01
10	0.05	0.15			
5	0.05	0.15			
2	0.05	0.10			
a 1	0.05 or 0.01	0.10			

^a Single reference standards of these denominations will be certified to the higher degree of precision indicated. In this case variations in the density of the air may change the values of brass weights by a little more than 1 in the last place given in the corrections. When weights of these denominations are the largest weights of a set, they may also be certified to the higher degree of precision, but this will be done only on special request.

VIII. NOTES ON THE DESIGN AND CONSTRUCTION OF WEIGHTS

1. CLASSES A, B, AND M

(a) **Form.**—It is advantageous to fix rather rigidly the design of weights of classes A and B.

Fig. 10 shows satisfactory shapes that are already in use. In order to allow room for the tweezers or other lifters with which weights are handled, the necks of the knobs on the smaller weights

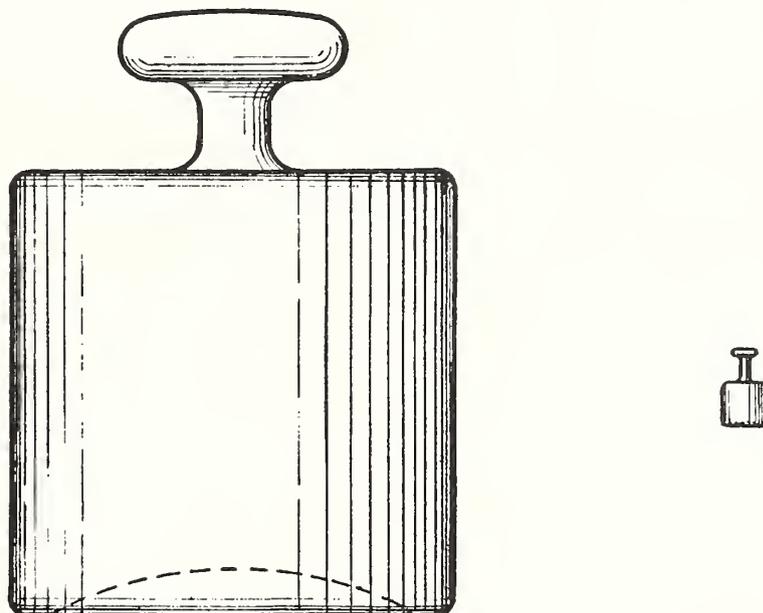


FIG. 10.—*Typical forms: Classes A, B, and M*

This shape of body and a well-rounded knob are required in classes A and B. A wide range of shape is allowed in class M; for example, that shown in Fig. 1, or that shown in Fig. 14, if the adjusting plug is omitted

must be taller in proportion to the body of the weights than on the larger ones.

In machining class A and class M weights the knob, top, and sides should be finished first, then the outer rim of the bottom, and then the central portion of the bottom is hollowed out by an amount equal approximately to the volume of the knob. The preliminary adjusting is completed in the last operation, and if the weights are to be plated they must be left sufficiently light to allow for such plating.

(b) **Size.**—The following table shows the value for height and diameter for the bodies of avoirdupois and metric weights of these

classes, computed for metal having a density of 8.4 g/cm³, which is an average value of the density of dense brass or Tobin bronze:

TABLE 6.—Representative Values of Height and Diameter, Classes A and B

Avoirdupois		Metric	
Denomi- nation	Height and diameter	Denomi- nation	Height and diameter
lb.	in.	kg	mm
50	5.94	20	144.7
20	4.38	10	114.9
10	3.48	5	91.2
5	2.76	2	67.2
2	2.03	1	53.3
1	1.61	g	
oz.		500	42.3
8	1.28	200	31.2
4	1.02	100	24.7
2	0.81	50	19.6
1	0.64	20	14.47
$\frac{1}{2}$	0.51	10	11.49
$\frac{1}{4}$	0.40	5	9.12
$\frac{1}{8}$	0.32	2	6.72
$\frac{1}{16}$	0.25	1	5.33
$\frac{1}{32}$	0.20		

(c) **Plating.**—Plated weights should have a much heavier coating than is generally used for ordinary commercial plated work. There is a distinct advantage also in polishing or possibly burnishing the weights once or twice during the plating, as this tends to make the coating less porous. Serious spotting is probably due in all cases to very minute pores in the metal under the plating, and can not be avoided except by using metal that is free from pores. Tobin bronze has generally been found to be very satisfactory in this regard. Gold plating, though soft, has the advantage that it can be repeatedly polished and plated without difficulty. Besides the usual electroplating process there are the newer methods of plating—by cathode discharge in vacuum, and by the so-called “spray process,” described by Schoop⁷—but these methods have not yet attained a sufficient commercial development to be definitely recommended. Such processes are, however, well worth investigation, as they avoid some of the difficulties encountered in the ordinary processes.

2. CLASS C, COMMERCIAL TEST WEIGHTS

(a) **Means of Adjustment.**—It is of great practical importance that the means of closing the adjusting hole be such that the weights can be readily readjusted, while permitting such

⁷ M. U. Schoop, *Metallurgical and Chemical Engineering*, 8, pp. 404-406 (July, 1910); 11, pp. 89-91 (February, 1913). John Calder, *Journal of the American Society of Mechanical Engineers*, July, 1915, p. 378.

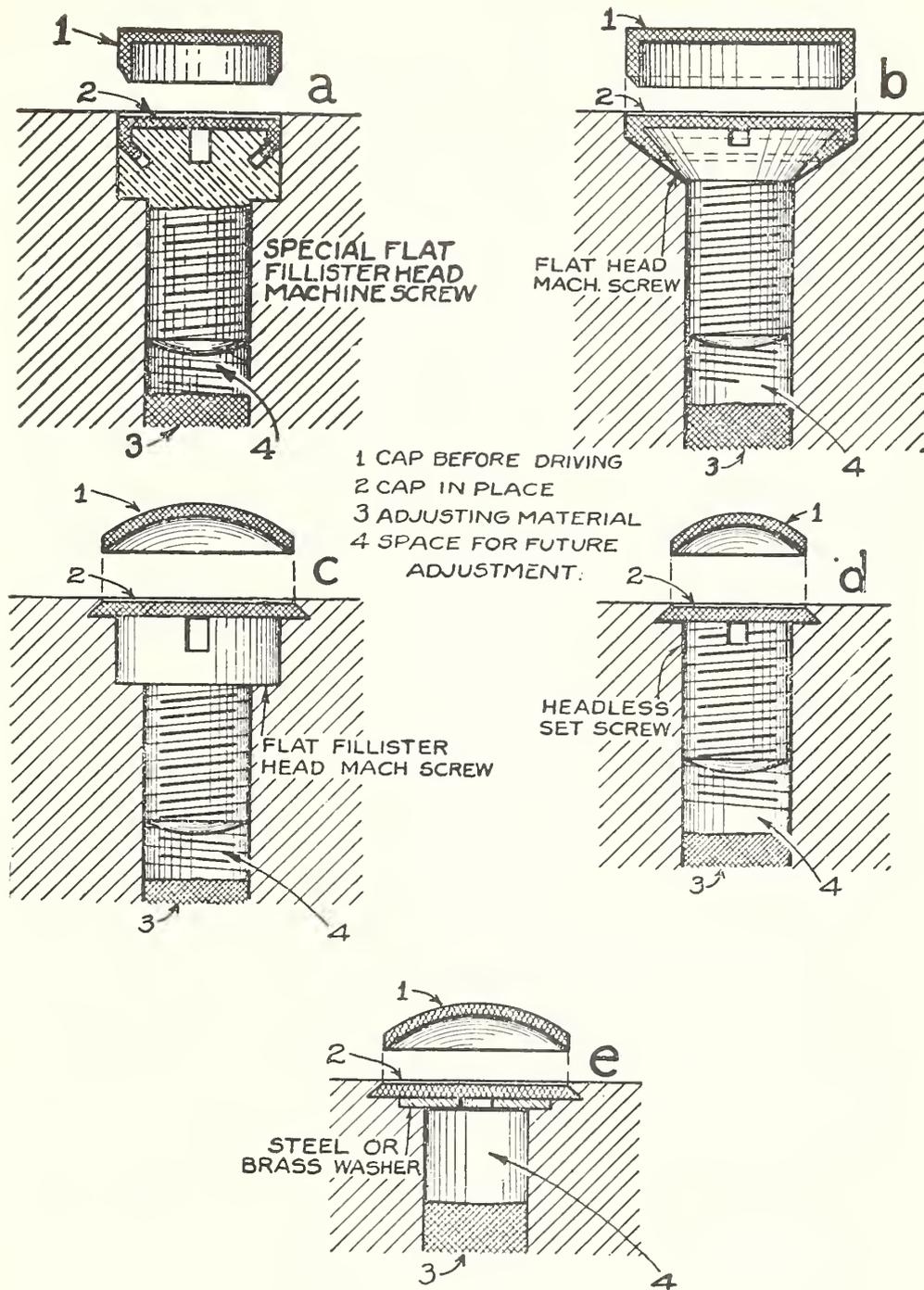


FIG. II.—Sealing caps for class C weights

These are some of the satisfactory methods of closing the adjusting holes. In forms *a* and *b*, especially in *a*, care must be taken in driving the caps not to allow the material to flow up beside the punch, but the skirts of the caps must be long enough to clinch securely. If lead caps for forms *c*, *d*, and *e* are the proper size and shape, they will be secure as soon as they are driven flat

readjustment only after mutilation or destruction of the seal. It is essential that the working standards of a local sealer be so constructed that they may be readjusted and sealed as often as there is need.

Five sealing devices are illustrated in Fig. 11. Type *c* is the most satisfactory of these, though the undercutting required in its construction is somewhat more difficult than the shaping of the heads of the screws of types *a* and *b*. In type *c* and in type *d* the head of the screw should be smaller than the hole at the surface of the weight by an amount sufficient to permit of the

cap being withdrawn by merely turning the screw without cutting off the edge of the cap.

Careful selection should be made of a design, which should be regularly adhered to, thereby facilitating the supplying at any time of new caps to those desiring to readjust their weights.

(b) **Acceptable Forms for the Weights.**—For class C there are a number of acceptable forms. The inverted cone form (Fig. 12) has a distinct advantage in the ease with which it can be replaced in the pocket of the carry-

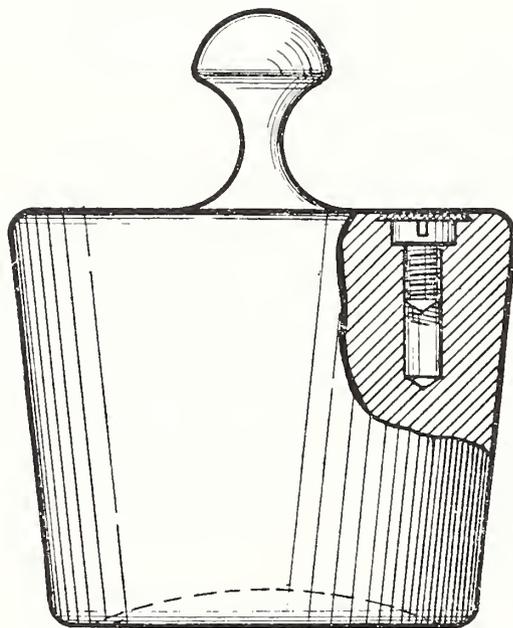


FIG. 12.—Conical form, class C

The small base allows weights of this form to be very quickly inserted into the pockets of the carrying base

ing case. The smaller sizes must, however, be broader, resembling the form shown in Fig. 13, so as to provide the necessary stability.

As a rule the knobs of class C weights should be taller than those of classes A or B in order to facilitate handling with the fingers.



FIG. 13.—Broad form, class C

Small weights of class C should be made broad so that they will not continually be falling over and rolling about

The knobless cylindrical form, Fig. 14, is a good form for weights of 2 kg or 5 pounds and less. Such weights can be readily stacked, and being cylindrical in shape and having the adjusting hole in the center, they can be formed with a minimum of machine work.

The form shown in Fig. 15, having a cup-shaped depression in the top, has these two advantages: First, that weights of this form can be stacked easily; and, second, that large weights can readily be turned from dense uniform steel such as bars of cold-rolled shafting. This form is not recommended, however, except for indoor use and in places where foreign material is not likely to gather in the depression in the top.

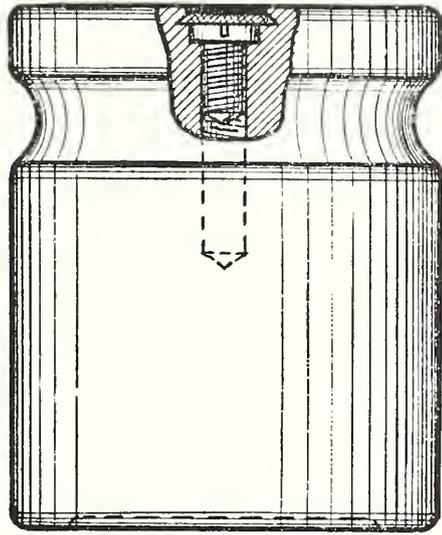


FIG. 14.—*Knobless cylindrical form, class C*

This is a very satisfactory form for weights of 5 pounds or less. It is specially recommended for reducing the cost of making weights of monel metal, high percentage nickel-steels, and other materials that are less easily worked than brass

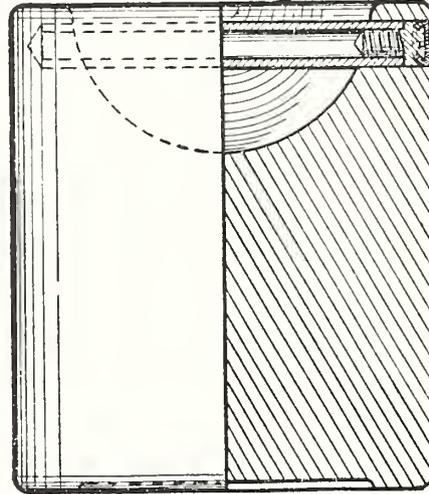


FIG. 15.—*Mint form*

The handle should not extend clear through the weight. This form is suitable for large weights for clean work. A similar form has long been used to some extent for weighing bullion in mints, assay offices, etc.

The forms here illustrated are not to be considered as especially recommended; they merely serve as examples of common and satisfactory types.

(c) **Pockets.**—Attention should be given to providing for ease of handling of these weights into and out of the pockets of the case. It is desirable to have the pockets slightly larger than apparently necessary, both to facilitate handling of the weights and to allow for possible shrinkage of the case.

(d) **Lifters.**—Special attention is called to the specifications in regard to lifters. Some forms of lifters, especially tweezers, are often well rounded where it is not needed but very sharp on edges that seize the weights. In a few cases metal tweezers with specially roughened surfaces have been noted. The tips of the tweezers used in handling the small weights can not be rounded very much, but can be rounded enough to be a great benefit. The other edges that may come in contact with the weights should be well rounded.

(e) **Tolerances.**—Manufacturers and dealers should bear in mind that the tolerances are the largest allowable errors, and it is to be expected that no large proportion of the weights of the set shall have errors near these limits. A properly equipped shop should ordinarily have no difficulty in working within one-half of these limits, larger errors resulting only from the accidental combination of several errors.

Care taken in adjusting, so as to keep the errors of the weights well within the tolerance limit, will reduce delays and eliminate the expense of readjusting weights having such large errors as to require rejection and return to the maker for correction.

Tables of tolerances for each class will be found under the specifications for the corresponding classes. Tolerances for trade weights, in accordance with those given in Circular 61, are given as Tables 9, 10, and 11 on pages 72, 73, and 74.

IX. TESTING OF WEIGHTS

In the small space available it will be possible to give but a brief outline of the common methods used for different grades of weighing and to describe only a few of the simpler methods of procedure for the calibration of weights by intercomparison of the weights of a set.

For the testing of ordinary trade weights by a local sealer the only practicable method is the comparison of each weight directly with a standard. For work of high precision and for testing sets of weights that come within the scientific group, some of the intercomparisons outlined below are preferable.

When this latter method of testing is used there is no gain in the purchase of complete sets of standards if this must be done, as is often the case, at the expense of quality. Probably the best denominations for reference standards for most scientific laboratories are one 100 g or 50 g weight, one 1 g weight, and one 10 mg weight.⁸ The 1 g weight should be of platinum, as it is used as the starting point for the calibration of the fractions of a gram, which are usually of platinum.

1. WEIGHING

(a) **Zero Reading and Equilibrium Point.**—The phrase “zero point” is often used to designate the center of the scale over which the pointer of a balance moves, or, in general, the place at

⁸ It is often advisable to add a smaller weight, generally 1 or 2 mg, for determining the sensitiveness of the balance; or even a small set, from 5 to 1 mg, which will also serve to measure any large differences that may exist among the larger weights.

which the pointer should come to rest without load if the instrument were perfectly constructed and balanced. It is sometimes used to designate the rest point when there is no load on the pans, whether this point is at the center of the scale or not, but this idea is better expressed by the phrase "zero reading."

The phrase "rest point" or "equilibrium point" is then used to indicate the point on the scale at which the pointer would come to rest in any particular case. In very rough testing this may be found by waiting until the pointer comes to rest and then noting the reading, but it is seldom advisable to adopt this method. In careful work the rest point must be calculated from the extreme positions to which the pointer swings on each side of this point. The number of readings taken when the rest point is to be calculated will depend upon the accuracy required and the sensitiveness of the balance. For one determination of a rest point there must be one more reading made on one side than on the other, and all of the readings used must be consecutive. Simple illustrations of the method of calculating the rest point are given in Fig. 16, in the column headed "Equilibrium Points $\times 2$." It should be noted that the figures there given are twice the true rest point. In the later work shown in this figure the factor 2 cancels out.

(b) **Sensitiveness—Use of Deflections.**—In any of the following methods of weighing it is often necessary to know the sensitiveness of the balance. This may be found by adding a small weight to one of the pans and noting how far the rest point is deflected from its former position. In weighing it is often preferable, instead of adding small weights to the pans until the rest point is brought to the desired place, merely to note the deflection from this point and to calculate from the sensitiveness the weight that would be needed to bring about a reading at the appropriate rest point. The sensitiveness will, in general, be different at different loads, and as it may change with use of the balance and even with change of temperature, especially in very sensitive balances, it must be redetermined from time to time. In precision testing it is advisable to determine the sensitiveness at each weighing. On the other hand, a balance suitable for testing ordinary trade weights, being less sensitive, should not change noticeably from day to day. Illustrations showing the use of the deflection of the pointer in weighing by substitution and by transposition are given in Figs. 17 and 18.

Test Date Bal. Observer
 Ther, Hygr. Bar. at

PANS		SCALE READINGS		EQUILIB. POINTS × 2	COMPUTATIONS OBSERVED DIFFERENCE			
LEFT	RIGHT	LEFT	RIGHT					
Transposition	Σ(50)	(50g)	6.8	10.1	16.90	$\begin{array}{r} .10 \times 2.5 \\ \hline 2.85 \\ \hline \end{array}$		
	"	"	6.8					
	"	"		9.0				
	"	"	7.8	9.0				
Substitution	"	"		11.4	19.65	$\begin{array}{r} -6.00 \\ \hline -6.09 \text{ mg} \end{array}$		
	105.mg	.	8.3	11.3				
	C	85kg.	7.0	14.1			21.05	
		Δ20mg.	7.0	14.0				
	C	(5kg)	6.0	13.9			19.90	$\begin{array}{r} \text{Correction} \\ \text{to } 135\text{kg.} = +36.0 \\ \hline +75.2 \text{ mg.} \end{array}$
	"	"	6.1	13.7				
	Δ50.mg	8.9	14.0	22.90				
"	"	9.0	13.8					

11-576

FIG. 17.—Record of weighings—condensed form

This shows observations and computations for weighings by transposition and also by substitution. In these examples small weights were used in addition to the deflection of the pointer. Such readings as those given in the first weighing would indicate either that the zero reading of the balance was 13.43 (6.72 in actual scale divisions) or that the arms of the beam were very unequal. With the usual adjustment of a balance and the middle point of the scale taken as 10, the first rest point would indicate that after interchanging the weights, small weights would be needed on the right-hand pan instead of on the left

(c) **Three Methods of Weighing.**—There are three distinct methods of weighing. These will be designated in the following discussion as direct weighing, weighing by substitution, and weighing by transposition.

DIRECT WEIGHING.—In direct weighing the weight to be tested is placed on one pan of the balance and the standard on the other. The pointer may then be brought to the zero reading by adding small weights to one of the pans, or by the use of a rider on the beam; or the weight necessary to bring the pointer to the zero reading may be calculated from the deflection of the pointer and the sensitiveness.

To secure satisfactory results by this method the arms of the balance must be accurately equal, if it is nominally an equal-arm balance, or accurately in the proper ratio if it is an unequal-arm balance. The latter case will not be considered here, as such balances are not extensively used for testing purposes. The lengths and ratio of the arms may change with age and use, though these lengths are not so subject to change as the sensitiveness. Therefore, the ratio of arms of a balance that is to be used for direct weighing must be tested occasionally. If two accurately equal weights are available, this test may be made by noting whether the rest point is the same with these weights applied, as when there is no load in the pans. Two weights not accurately equal may be used by noting the rest point with one on each pan, then interchanging them and again noting the rest point. The point midway between these is the true rest point, or the point where the pointer would come to rest if the weights had been equal.⁹ The error due to inequality of arms is then the weight required to bring the rest point (with equal loads on the pans) back to the zero reading. This error is proportional to the load.

The direct method of weighing is so much simpler and quicker that it is worth while for a sealer to demand that his portable balance at least have its arms so nearly equal as to make interchanging of weights on the pans unnecessary in his work. For balances used in testing class C weights this equality of arms is not so important, since other methods can be used without serious inconvenience. In work of higher precision it is generally necessary to use one of the other methods of weighing.

For most, if not all, chemical analysis and for some other scientific and technical operations where accuracy of proportion

⁹ This is not theoretically exact, but is sufficiently accurate for testing any balance the arms of which are approximately equal.

rather than actual quantity is needed, direct weighing may be used even when the arms of the balance are not very accurately equal, because the errors are proportional to the load, and therefore the proper proportion will be maintained in the results.

SUBSTITUTION WEIGHING.—This is the simplest method of eliminating the effect of inequality of the arms of a balance, and it is a method applicable either to a balance having nearly equal arms or to one having one arm several times as long as the other.

The best order of procedure for testing by this method is as follows: Place the standard on one pan and counterpoise weights on the other pan sufficient to balance it approximately; note the position of the pointer on the scale; remove the standard and substitute the weight to be tested. If it is necessary to place additional weights with the weight being tested, in order to bring the rest point to its former position, then it is evident that the weight being tested is lighter than the standard by the amount of the weight added, and vice versa. If the weight is to be adjusted, this can be done at once by adding or subtracting sufficient adjusting material without again using the standard. Nevertheless, it is best to replace the standard after the adjustment is completed in order to be sure that the rest point of the balance has not changed appreciably during the work.

If the balance used is known to be very nearly constant, several weights of the same denomination may be tested before replacing the standard. How far this may be carried can be ascertained in no way except by experience with each individual balance and knowledge of the conditions under which it is being used.

TRANSPOSITION WEIGHING.—When the arms of a balance are nearly equal, but not sufficiently equal for direct weighing, the method of transposition furnishes a more accurate comparison than that of substitution. It requires about the same amount of time for the observations, but the error of the weight being tested is not shown in quite so direct a manner.

In weighing by transposition the weight to be tested is first placed on one pan and the standard on the other. It is expedient then to bring the rest point near the center part of the scale, by adding any convenient material (an extra set of cheap weights may be used) to one of the pans; this material may be left in place and disregarded during the rest of the weighing. The rest point is then noted, and the standard and weight under test are transposed, or interchanged. Known weights may then be added to

the "light" side of the balance to bring the rest point back to its former position. The difference between the standard and the weight under test is then one-half of these added weights. In work of high precision, however, it is generally impracticable to do this. In such work, after the standard and weight under test have been transposed, the new rest point is noted and the amount of added weights that would be needed is calculated from the sensitiveness obtained as mentioned in (b) above and illustrated in Figs. 16 and 17. Whether the weight tested is too light or too heavy can be readily ascertained from the direction of motion of the rest point.

Weighing by transposition is recommended for work of high precision, in which it is also desirable to calculate the rest point from several swings of the pointer. In other cases substitution is generally to be preferred.

Illustrations of the calculations, which are in general very simple, are given in Figs. 16 and 17.

2. RECORDING

Figs. 16 and 17 show forms used at this Bureau for recording the readings taken in weighing by transposition or substitution. Complete weighings are shown therein to illustrate the methods of recording and calculating. Both forms shown have essentially the same arrangement. The one in Fig. 17 is somewhat condensed, that in Fig. 16 being used when more room is needed for auxiliary calculations, such as buoyancy corrections. In taking readings of the swings of the pointer the center of the scale (often called the zero point) is numbered 10 or 100 in order to avoid having negative readings on the left of this point.

It should be noted in these figures that the denomination of the standard is preceded by a letter which indicates the set to which it belongs, while that of the weight being tested is inclosed in parentheses. These special designations serve not only to distinguish the standard from the weight being tested, but also to prevent them from becoming confused with the numbers used in the calculations. The corrections to the nominal values are used, instead of the total values of the weights, in order to avoid a confusing succession of nines or zeros. The plus sign for a correction indicates that the mass of a weight is greater than its nominal value, the minus sign that it is less than the nominal value. In calculations these signs may be treated in the ordinary algebraic manner.

3. TOLERANCE TESTING

When it is sufficient to know that the error of a weight is less than a prescribed amount, as is nearly always the case in commercial testing, the deflection of the pointer may in some cases be used to advantage. However, the most direct and safe method of tolerance testing is that of placing a weight equal to the tolerance on the pan with the weight being tested if the weight is too light, the standard being on the other pan, or on the pan with the standard if the weight being tested is too heavy. This shows directly whether or not the error is greater than the tolerance.

In using deflections it is not generally necessary to know the sensitiveness very closely, but merely that a certain weight is "correct" if it does not deflect the pointer beyond a certain position.

4. THE TEST OF A SINGLE WEIGHT

In testing a single weight it is generally compared with a standard of the same denomination. A weight can, however, be tested by comparing it with a standard of twice its mass, using an additional weight equal to it. The two together are compared with the standard and the correction for their sum (which is the sum of their corrections) is thus determined. The weights are then compared with each other and their difference determined. From the sum and differences of the corrections the correction for each weight can readily be computed, and proper attention to the algebraic signs will secure the proper sign for the individual corrections.

There are two distinct advantages in this latter plan. In the first place, whatever uncertainty or error there may be in the correction for the standard will be divided by 2 in calculating the correction for the weight tested. In the second place, the additional weight used may be one whose correction is known at least approximately, and the redetermination of this correction gives a valuable check on the accuracy of the work.

5. THE TEST OF SEVERAL EQUAL WEIGHTS

(a) **Methods and Computations.**—When a large number of equal weights are to be tested, and the highest precision of which the balance is capable is not necessary, the substitution method is greatly to be preferred for comparing each weight individually with the standard. For more accurate work, and especially in order to secure checks that will detect any serious error in record-

ing, each weight may be compared separately with the standard, and then with each of the other weights. This is not practicable when testing a very large number of equal weights, but in that case the weights may be divided into groups and each group treated in the above manner. Although the calculation of the corrections for each weight from such a series of observations would ordinarily be a long process, it happens that even the solution by the method of least squares, which gives the most reliable results, can be put into very simple form for an intercomparison of a few weights. The formulas for two of the simplest cases are given below.

Let w_1 represent the first weight, w_2 the second, and s the standard. Then if two weights are to be tested, three weighings can be made, which may be represented by $w_1 - s$, $w_2 - s$, and $w_1 - w_2$. The difference must be taken in the order indicated. For example, if s is heavier than w_1 , its mass is greater than w_1 , and therefore the difference will be negative. Let the differences found be represented by a_1 , a_2 , and a_3 . The complete weighings may then be represented as:

$$\begin{aligned} w_1 - s &= a_1 \\ w_2 - s &= a_2 \\ w_1 - w_2 &= a_3 \end{aligned}$$

Using all three weighings the best values for $w_1 - s$ and $w_2 - s$ are shown by the method of least squares to be

and
$$\begin{aligned} w_1 - s &= \frac{1}{3} (2a_1 + a_2 + a_3) \\ w_2 - s &= \frac{1}{3} (a_1 + 2a_2 - a_3) \end{aligned}$$

Thus, knowing the correction for s , the errors of w_1 and w_2 may be found algebraically. Similarly for three weights to be tested we have the weighings

$$\begin{aligned} w_1 - s &= a_1 \\ w_2 - s &= a_2 \\ w_3 - s &= a_3 \\ w_1 - w_2 &= a_4 \\ w_2 - w_3 &= a_5 \\ w_1 - w_3 &= a_6 \end{aligned}$$

and from these we find—

$$\begin{aligned} w_1 - s &= \frac{1}{4} (2a_1 + a_2 + a_3 + a_4 + a_6) \\ w_2 - s &= \frac{1}{4} (a_1 + 2a_2 + a_3 - a_4 + a_5) \\ w_3 - s &= \frac{1}{4} (a_1 + a_2 + 2a_3 - a_5 - a_6) \end{aligned}$$

Circular of the Bureau of Standards

(1 pound) ₂	
+	-
+ a ₁ =	0.014
+2 a ₂ =0.006	
+ a ₃ =	.014
- a ₄ = .019	
+ a ₅ = .016	

	.041 .028
	.028

sum=+	.013

$\frac{1}{4}$ sum=(1 pound) ₂ -1 pound ₇ =+	.003
Correction for 1 pound ₇	=- .033

Correction for (1 pound) ₂	=- .030 grain

(1 pound) ₃	
+	-
+ a ₁ =	0.014
+ a ₂ =0.003	
+2a ₃ =	.028
- a ₅ =	.016
- a ₆ =	.004

	.003 .062
	.003

sum=-	.059

$\frac{1}{4}$ sum=(1 pound) ₃ -1 pound ₇ =-	.015
Correction for 1 pound ₇	=- .033
Correction for (1 pound) ₃	=- .048 grain

FIG. 18.—(Above and preceding page)

Arrangement of computations for comparison of four equal weights, three unknown and one standard.
The standard is designated as 1 pound₇.

(b) **Residuals.**—After the final correction for each weight has been obtained the values of $w_1 - s$, $w_2 - s$, etc., are computed. Since the weighings can not be made with perfect accuracy, these computed values will, in general, differ slightly from the observed values a_1, a_2 , etc. The differences are called the residuals and might be considered as representing the errors of the weighings if the final corrections were perfectly accurate. If any residual is larger than the error to be expected in a weighing, it is good evidence that a mistake has been made either in the weighings or in the computations. If the mistake is in a weighing, the residual for that weighing will generally be much larger than for the others, but with some of the computation methods given in this circular one or two other residuals may sometimes be made practically as large by the one

mistake. If a very large number of weighings were made for a few weights, the accuracy of the weighings could be estimated from the residuals, but with the few weighings used in all of the inter-comparisons shown in this circular, the residuals serve only to indicate serious mistakes. However, if most or all of the residuals are unusually large and there is no error in recording or in the computations, it is safe to assume that some condition has rendered the weighings less accurate than usual.

6. INTERCOMPARISON OF THE WEIGHTS OF A SET

(a) **Methods and Computations.**—When the weights to be tested form what is ordinarily called a set, then instead of comparing each weight with a standard, the largest weight or the sum of several of the weights may be compared with a standard and the weights of the set then intercompared. From these weighings the value of each individual weight can then be calculated as described later.

Especial advantages of this mode of procedure are: In the first place it does not require the use of a standard of each denomination; in the second place, it makes it possible to base the determination of the values of the small weights on a much larger standard, thus practically eliminating so far as these small weights are concerned any unknown uncertainty or error of the standard used. It is best, as a rule, to base the work on as large a standard as possible, using other standards in the comparisons as checks only. In addition, the methods given in this section have the advantage that a sufficient number of combinations are used so that the checks and residuals serve to show not only when a mistake has been made in the calculations but also when one has been made in the weighings, and in general to indicate in which weighing it has been made. (See paragraph above on "Residuals.") The results are obtained from a "least-square" solution of the observations and are therefore the best to be obtained from any given set of observations, but the use of the forms requires no knowledge of "least squares"—merely sufficient mathematical training to use the positive and negative signs properly.

The first two forms shown, 52 C and 52 B (Figs. 19 and 20), have been filled in from one of the tests made at this Bureau, as this may assist in making clear the use of the forms. The first equation on these sheets is for the value of the summation 100 g, generally indicated as $\Sigma(100\text{ g})$. This may be obtained by simply comparing it with a 100-g standard, or if the set contains a 100-g

Standards of Mass

Sheet 2

MASS COMPUTATION SHEET
Combination: 5, 2, 2, 1

DEPARTMENT OF COMMERCE
BUREAU OF STANDARDS
Form 51 B

Weights submitted by _____
Original Record: Vol. _____, pp. _____ to _____, inclusive.
Computer: _____ Date: _____ B. S. Test No. _____

MEAN.		OBSERVATIONS.	
(5) + (2) ₁ + (2) ₂ + (1) = 10	-		
(5) - (2) ₁ - (2) ₂ - (1) =	-		
(5)	= 5		
(2) ₁ + (2) ₂ + (1) = 5	+	.002 = W [From Sheet (1)]	
		Observed.	Obs. - Cal.
(1)	- Σ(1) = a ₁ = -	.083 mg	.004
(1) - (2) ₁	+ Σ(1) = a ₂ = +	.104	.001
(1) - (2) ₂	+ Σ(1) = a ₃ = +	.129	.001
(2) ₁ - (2) ₂	= a ₄ = +	.015	.010
(1) + (2) ₁ - (2) ₂ - Σ(1)	= a ₅ = -	.048	.006
(1) - (2) ₁ + (2) ₂ - Σ(1)	= a ₆ = -	.107	.003
		(0.-C.) ²	
-	+	-	-
- a ₁		.104	- .155
+ a ₂	.129		- .238
+ 2 a ₃	.30		- .79
+ 2 a ₄		.96	- .159
- 2 a ₅	.214		+ .233
	.373	.200	+ .74
	.200		+ .8
Sum.	.173		- .66
5 × Sum.	.865		+ .124
(5 × Sum) + 7 = N	+ .124		+ .006
W	+	.2	- .019
m	+	.74	+ .015
W + m = 5(1)	+	.76	+ .2
(1)	+	.015	
-S	+	.79	
(1) - S = Σ(1)	+	.094	

Note: Σ(1) is Vig
Check: Ev. Vig = +.100

FIG. 20.—Computation form (52 B)

Intercomparison of weights of a set, series 5, 2, 2, 1, Σ1. The numbers inserted in this form continue the work shown in Fig. 19. This form applies to sets in which the (2) is duplicated and in which it is not desired to divide the Σ(1) into (0.5) and Σ(0.5) as would be required by the use of the form shown in Fig. 22 (52 D). If (2) is not duplicated and (1) is duplicated or triplicated, then this form may be replaced by that shown in Fig. 21 (52 A)

weight, the (100 g) and Σ (100 g) may be compared with the standard with and each other as described above for the testing of two equal weights. In these first equations it should be noted that the numbers not inclosed in parentheses are merely nominal values. In the later portion of the sheets they do not appear, and the final values calculated are the corrections to the various weights. The second equation indicates the next weighing to be made, and from these first two equations the corrections for the 100-g and 50-g weights are calculated. Special care should be taken in determining these values of (50 g) and Σ (50 g), as there are no residuals to expose immediately any mistake made. The calculation may be checked by computing Σ (100) and (50) - Σ (50) from the values found. Sometimes the work can be so arranged that the Σ (50) is involved in a preceding inter-comparison, as in the case of the Σ (5) of the next sheet (Fig. 20), the value for which was determined on the first sheet. The next weighings are indicated in the following series of equations. Each of these weighings determines the difference between two weights or groups of weights. The weights to be grouped together are not always printed together, but can readily be distinguished by the plus and minus signs. The columns headed "Obs. - Cal." or (O. - C.)² are for the residuals. The balance of the computation consists in merely following the operations indicated. Practically all of the operations can be done mentally and all results entered directly on the computation sheet.

The quantities marked "Check" or "Computed" are always computed from the final corrections, and serve to check the accuracy of the computations. The weight marked Vig on the second sheet is a known weight inserted partly in order to complete the number of required weights, but a known weight was chosen in order to serve as a further check on the latter part of the calibration.

The final results of this calibration may be summarized as follows:

Designation:	Correction, mg	Designation:	Correction, mg
(50 g).....	+0.341	(5 g).....	+0.029
(20 g).....	+ .036	(2 g) ₁	+ .006
(10 g) ₁	+ .021	(2 g) ₂	- .019
(10 g) ₂	+ .040	(1 g).....	+ .015

Each sheet is applicable to any multiple or submultiple of the denominations indicated on the sheet. The decimal multiples are most commonly used, but it is sometimes convenient to use binary multiples, especially in testing avoirdupois weights.

Standards of Mass

No arrangements have been made for the sale of these printed forms, but they can readily be copied or printed. Sample copies will be supplied to persons interested in them.

It is sometimes convenient to base the calibration on a standard substituted in place of one of the weights indicated in the equations. This is illustrated in the work inserted on Form 52A (Fig. 21). Here the weight corresponding to $(1)_2$ was the standard. In making the computation the ordinary procedure was followed until the unknown value of $4 K$ was needed to complete the computations. The known correction for 1 kg_7 , -2.0 mg , was then entered at the bottom of the column headed 1 kg_7 . The sum of the quantities above $4 K$ was then found and the value of $4 K$ necessary to give -2.0 as a correction to 1 kg_7 was easily determined and entered. This furnished the missing value of $4 K$ and $8 K$ to the other columns and the computation was completed. Another method of procedure would be to assume that the correction to 1 kg_7 was zero and enter 0.00 in place of -2.0 . A further correction of -2.0 mg per kg is then added to the corrections computed as before. For example, we have by application of the first method—

for (2 kg) a correction $+1.0 \text{ mg}$

by the second method we would have:

for (2 kg) a correction of $+5.0 + 2(-2.0) = +1.0 \text{ mg}$

The first method is generally preferable.

(b) **Check equations.**—As previously mentioned, mistakes usually disclose themselves by causing a large residual. The residuals, however, are not obtained until after completing the computations. By means of “check equations” mistakes in the weighings can generally be discovered before the computation is started. These equations express the relations that exist between the observations of a given series.

The check equations for the sheet 52C and their application to the accompanying data appear as follows:

Equations:	Particular values
$a_1 - a_2 = a_5 - a_6$	$-0.063 = -0.061$
$a_1 - a_3 = a_5 - a_7$	$-0.014 = -0.015$
$a_4 - a_1 = a_6 + a_8$	$+0.076 = +0.087$
$a_4 - a_2 = a_5 + a_8$	$+0.013 = +0.026$
$a_3 - a_4 = a_8 - a_7$	$-0.062 = -0.053$
$a_3 - a_2 = a_7 - a_6$	$-0.049 = -0.046$

The agreement among the particular values is sufficiently good to indicate that no mistake was made and that the weighings are

Circular of the Bureau of Standards

MASS COMPUTATION SHEET
Combination: 5, 2, 1, 1, 1

Department of Commerce
BUREAU OF STANDARDS
Form 52 A

Weights submitted by
Original Record: Vol., pp. to, inclusive.
Computer: Date: B. S. Test No.

Based on a standard, 1 kg_T, that takes the place of (1)₂ in the equations

		MEAN.	OBSERVATIONS.	
(5)	(2) + (1) ₁ + (1) ₂ + Σ(1) = 10	-	[]
(5)	(2) - (1) ₁ - (1) ₂ - Σ(1) =	-	[]
(5)	= 5	-		
(2)	(1) ₁ + (1) ₂ + Σ(1) = 5	-	= K	
		Observed.	Obs. - Cal.	(O. - C.) ² .
(1)	(1) ₁ - (1) ₂ = a ₁ = +4.0 mg	.0	.	.
(1)	(1) ₁ - Σ(1) = a ₂ = +.7	.1	.	.
(1)	(1) ₂ - Σ(1) = a ₃ = -3.5	.1	.	.
(1)	(1) ₁ + (1) ₂ - (2) = a ₄ = -1.0	.0	.	.
(1)	(1) ₁ + Σ(1) - (2) = a ₅ = -1.4	.2	.	.
(1)	(1) ₂ + Σ(1) - (2) = a ₆ = +2.3	.1	.	.

	(1) ₁			(1) ₂ = 1 kg _T			Σ(1)	
	+	-		+	-		+	-
+5a ₁	20.0		-5a ₁		20.0	0a ₁		
+5a ₂	3.5		0a ₂			-5a ₂		3.5
0a ₃			+5a ₃		17.5	-5a ₃	17.5	
+3a ₄		3.0	+3a ₄		3.0	-2a ₄	2.0	
-2a ₅	2.8		+3a ₅		4.2	+3a ₅		4.2
+3a ₆	6.9		-2a ₆		4.6	+3a ₆	6.9	
+4K	9.3		+4K	9.3		+4K	9.3	
	42.5	3.0		9.3	49.3		35.7	7.7
	3.0				9.3		7.7	
Sum	39.5		Sum		40.	Sum	28.0	
1/2 sum	2.0		1/2 sum	known	2.0	1/2 sum	1.4	

Check: +9.3 ÷ 4 = +2.3 = K

		(2)	Check:	
		+	-	(2) + (1) ₁ + (1) ₂ + Σ(1) = 5 +2.4 = K.
-4a ₄	4.0			
-4a ₅	5.6			
-4a ₆		9.2		
+8K	18.6			
	28.2	9.2		
	9.2			
Sum	19.0			
1/2 sum	1.0			

FIG. 21.—Computation form (52 A)

Intercomparison of weights of a set, series 5, 2, 1, 1, 1. This form applies to sets or parts of a set in which the (100), the (10), or the (1) are duplicated instead of the (200), the (20), or the (2), and in which it is not desired to divide the (100), the (10), or the (1) into two parts as would be required in the form shown in Fig. 19 (52 C). The computations inserted in this figure illustrate a calibration based on a standard which is treated in the weighings as if it were one of the weights of the set. Any of the computation forms shown in this circular may be used in a similar manner

Circular of the Bureau of Standards

MASS COMPUTATION
COMBINATION: 4, 2, 2, 1, Σ 1

Department of Commerce
BUREAU OF STANDARDS
Form 52 F

Original Record: Vol., pp. to, inclusive

Computer: Date: B. S. Test No.

$$(4) + (2)_1 + (1) + \Sigma(1) = 8 \quad - \quad = K$$

	Observed.	Obs. - Cal.
$(4) - (2)_1 - (2)_2 + (1) - \Sigma(1) = a_1 = -$.	.
$(4) - (2)_1 - (2)_2 - (1) + \Sigma(1) = a_2 = -$.	.
$(4) - (2)_1 - (1) - \Sigma(1) = a_3 = -$.	.
$(4) - (2)_2 - (1) - \Sigma(1) = a_4 = -$.	.
$(2)_1 - (2)_2 + (1) - \Sigma(1) = a_5 = -$.	.
$(2)_1 - (2)_2 - (1) + \Sigma(1) = a_6 = -$.	.

(4)		(2) ₁				(2) ₂			
		+	-	+	-	+	-	+	-
0a ₁				-5a ₁				-5a ₁	
0a ₂				-5a ₂				-5a ₂	
+3a ₃				+a ₃				+5a ₃	
+2a ₄				+4a ₄				0a ₄	
-a ₅				+3a ₅				-5a ₅	
-a ₆				+3a ₆				-5a ₆	
+5K				+5K				+5K	
Sum				Sum				Sum	
1/8 sum				1/8 sum				1/8 sum	
(1)				Σ(1)				Partial Check: (Computed values)	
				+	-	+	-		
+10a ₁						0a ₁		$(4) + (2)_1 + (1) + \Sigma(1) =$ $- \quad \quad \quad = K$	
0a ₂						+10a ₂			
-7a ₃						-7a ₃			
-8a ₄						-8a ₄			
+4a ₅						-6a ₅			
-6a ₆						+4a ₆			
+5K						+5K			
Sum						Sum			
1/8 sum						1/8 sum			

FIG. 25.—Computation form (52 F)

Intercomparison of weights of a set, series 4, 2, 2, 1, Σ 1. This form may be used, by inserting an extra (2), for calibrating the ordinary sets of avoirdupois pound and ounce weights; sets in which each weight is one-half of the next larger weight. Without the insertion of some extra weights there is no very satisfactory way of intercomparing the weights of such sets

satisfactory. Since the first three of these check equations involve all of the observations, a satisfactory agreement in these three is generally considered sufficient evidence to warrant proceeding with the computations.

The check equations for the various schemes are inserted here as they do not appear upon the respective computation sheets.

TABLE 7.—Check Equations

Sheet	Series	Check equations	Sheet	Series	Check equations
52A	5, 2, 1, 1, $\Sigma 1$	$a_1 + a_3 = a_2$ $a_2 + a_5 = a_4$ $a_3 + a_6 = a_4$ $a_1 + a_5 = a_6$	52D	50, 20, 20, 10, 5, $\Sigma 5$	$a_1 - a_8 = a_5$ $a_2 + a_8 = a_3$ $a_3 - a_9 = a_5$ $a_4 + a_9 = a_5$ $a_6 - a_7 = a_5$
52B	5, 2, 2, 1, $\Sigma 1$	$a_3 - a_2 = a_4$ $a_5 - a_1 = a_4$ $a_1 - a_6 = a_4$ $a_5 - a_6 = a_4$ $\frac{a_5 - a_6}{2} = a_4$	52E	4, 3, 2, 1, $\Sigma 1$	$a_1 - a_2 = a_9$ $a_4 - a_3 = a_9$ $a_7 - a_6 = a_9$ $a_1 + a_8 = a_4$ $a_2 + a_8 = a_3$ $a_5 - a_6 = a_4$ $a_5 - a_7 = a_3$
52C	50, 20, 10, 10, 5, $\Sigma 5$	$a_1 - a_2 = a_5 - a_6$ $a_1 - a_3 = a_5 - a_7$ $a_4 - a_1 = a_6 + a_8$ $a_4 - a_2 = a_5 + a_8$ $a_3 - a_4 = a_8 - a_7$ $a_3 - a_2 = a_7 - a_6$	52F	4, 2, 2, 1, $\Sigma 1$	$a_4 - a_3 = \frac{a_5 + a_6}{2}$ $a_1 - a_2 = a_5 - a_6$

X. BUOYANT FORCE OF THE AIR

1. GENERAL DISCUSSION

As the buoyant force of the air and its effect on weighing are often not fully appreciated, it is deemed advisable to consider the relation of atmospheric buoyancy to the testing of weights. A knowledge of this subject is essential in order that the principles of buoyancy corrections in weighings may be fully understood, and that in weighings in which the buoyant force is neglected an estimate may be formed of the degree of the approximations that are obtained. For example, in weights of the commercial group either the tolerances are large enough or the densities of the materials permissible under the specifications lie within a narrow enough range that the buoyant force of the air may be neglected.

(a) **Cause and Amount of the Buoyant Force.**—The buoyant force of the air is exactly similar to the buoyant or floating effect familiar to all in the case of liquids, although, to be sure, in the case of the air the force is much less in amount. If pound weights, respectively, of wood, aluminum, and brass are held under water, those of aluminum and brass will appear lighter, while that of wood will actually exert an upward force, thus having, so to speak, a negative weight. Moreover, the aluminum will appear to lose

more weight than the brass. Now the amount by which the weight of an object is diminished when it is submerged is exactly equal to the weight of the water displaced. A pound of aluminum has a volume of 10 cubic inches, and a mass of water having this volume weighs 0.36 pound. The buoyant effect of the water on the mass of aluminum is therefore 0.36 pound, and, when the aluminum is submerged, it will appear to weigh 0.36 pound less than its true weight; similarly the brass weight will appear to weigh 0.12 pound less than its true value.

Now assume that on the pans of an equal-arm balance which has been completely submerged in a tank of water 1-pound weights of aluminum and brass are placed, one weight being on each pan. Clearly the balance will indicate a difference in the value of the weights, the brass one appearing to weigh the more, since its volume, and therefore the buoyant force upon it, is less than that of the aluminum weight. This apparent difference in weight will amount to 0.24 pound.

Now assume that common salt is dissolved in the water of the tank. The effect of this will be to increase the density of the water, and consequently to increase the apparent difference in the values of the two weights. If we increase the salinity of the solution to the point of saturation, the difference in the apparent values of the weights will increase to 0.29 pound.

Now a similar phenomenon occurs in the case of weights weighed in air, every weight being buoyed up by an amount equal to the weight of the air which it displaces. The weight of 1 cubic yard of air is about $2\frac{1}{2}$ pounds; any object, therefore, having a volume of 1 cubic yard will be buoyed up by the air with a force of $2\frac{1}{2}$ pounds with the effect of reducing by that amount the weight which would be obtained for the object were it weighed in a vacuum. Since most bulky objects are porous, the size of an article gives only a rough estimate of the amount of the buoyant force of the air upon it.

The buoyant force of the air on a 1-pound weight of brass is about 1 grain; on a 50-pound weight of brass, about 50 grains. Either of these latter figures would, of course, be too large to neglect in the test or adjustment of a test weight. However, the discrepancies introduced by the buoyant force of the air usually occur merely as differences, two weights of the same mass apparently having different weights when weighed in air, due to the *difference* in the magnitudes of the forces by which the two weights are respectively buoyed up.

Moreover, as the air changes in density, which it does constantly under changing weather conditions, the difference in the buoyant forces will vary for weights of two materials being compared. In any given locality the density of the air will vary by about 10 per cent as weather conditions change; under constant weather conditions the density of the air will vary about 20 per cent, due to a difference in altitude from sea level to 6000 feet above sea level.

For example, a cast-iron 50-pound weight, if adjusted to the same apparent weight in air at sea level as a brass standard, will appear about 2 grains heavier than the brass standard at an altitude of 6000 feet, and an apparent variation of nearly 1 grain due to variations in air density may be expected at any given place.

The above changes are usually negligible in commercial work, but for some scientific work, and for the purposes of adjusting standards from which commercial weights are derived, the buoyancy correction must be carefully considered and either eliminated or properly corrected for.

These effects of changes in the buoyant force of the air can not be neglected in work of high precision, even in the case of brass weights, for brass weights of the same denomination do not have exactly the same size on account of slight differences in the metal. A 2-pound brass weight whose density would come within the limits specified for class A might appear to be 0.01 grain heavier or lighter at an altitude of 6000 feet than it does at sea level.

It is on the basis of these considerations of the effect of variation in the buoyant force of the air that in many cases the kinds of material allowable and the degree of precision to which the corrections could be certified, etc., have been determined. In a few cases extreme variations in the buoyant force of the air will change corrections of class A or class S weights by about one unit in the last place.

(b) **Apparent Mass and "Weight in Air Against Brass."**—Since, as has been explained, the mass of the object (or of the weight) being tested is determined by comparison of its weight with that of the standard, it is evident that the buoyant force of the air, by changing the apparent weight of both of them, prevents exact comparison of the masses unless the effect of the buoyant force is eliminated or proper corrections are made for it.

It is often convenient, however, to refer to results obtained by the ordinary comparison in air as "apparent mass," in cases where no allowance is made for the effect of this buoyant force.

As has been stated, the buoyant force of the air may be neglected on weights of the commercial group. However, it is necessary to establish the values of commercial weights on a definite basis, and to this end all corrections and tolerances apply to the apparent mass as determined in air having a density of 1.2 mg per milliliter, against (brass) standards having a density of 8.4 g per cubic centimeter at 0° C, whose coefficient of cubical expansion is 0.000054 per degree centigrade, and whose values are based on their true mass or weight in vacuo. The value of any brass weight adjusted or tested in the ordinary way is therefore practically its true mass, for all commercial purposes.

In scientific work, and especially in chemical analysis, weighings are often recorded as being made according to "weight in air against brass." Since this is generally understood as meaning the apparent mass as this would be determined against brass standards in air, it will be taken as exactly equivalent to the commercial basis, as just defined.

All weights of the commercial group (classes A, B, C, and T) are tested and certified by this Bureau on this commercial basis; and weights of classes M and S have corrections certified both on this basis and according to the true mass of the weights.

2. ELIMINATION OF BUOYANCY CORRECTIONS

The error due to the buoyancy of the air is completely eliminated in cases where the volumes of the two weights being compared are exactly equal. Since absolute equality is impossible of attainment, it is desirable to determine how nearly equal the volumes must be in order to make the difference in the buoyant forces negligible. This question is chiefly of importance in cases where the volumes are not measured but are calculated from the known or assumed densities of the material. The matter is fully discussed under "Effect of error in assumed density of the weight" on page 63.

In testing weights of the commercial group the effect of the buoyant force of the air is practically eliminated by the restrictions as to material and by the relatively large tolerances which apply.

In ordinary chemical analysis, and in scientific work of similar character, it is desirable to be able to use the platinum and alumi-

num fractions of a gram with the brass weights without the necessity of making continual buoyancy corrections. It is recommended that for such purposes these fractions of the gram be adjusted according to their apparent mass as determined in air against brass standards. The values of the platinum and aluminum weights on this basis may be considered as values determined from weighings in air using the density of brass as the *assumed density* of these weights. If the corrections of these weights have already been determined on an assumed density of 21.5 and 2.7, the change to the other assumed density may be readily calculated in the same manner as would be done in testing them.

Similar reductions are made in primary standards for use as reference standards for commercial weights and in some other special instances as well.

Table 12, page 75, gives the reduction terms needed to reduce the values of small platinum and aluminum weights from those based on an assumed density of 21.5 or 2.7 to those based on an assumed density of 8.4 or 21.5.

As will be explained later, the error in the assumed density for the weight would cause no discrepancies whatever in the use of the weight if the density of the air were to remain constant. A discussion of the errors arising from variations in the density of the air will be given subsequently.

3. CALCULATION OF BUOYANCY CORRECTIONS

(a) **General Principle — Direct Method.**—The correction for buoyancy will be the difference between the buoyant forces acting on the two objects or weights compared.

If the weighing is expressed as

$$w_1 - w_2 = a$$

and the volumes of the weights are v_1 and v_2 , respectively, and the density of the air ρ , the buoyancy correction will be

$$\rho v_1 - \rho v_2 = \rho(v_1 - v_2)$$

and if the correction is computed in the manner indicated, this correction is to be added algebraically to the apparent difference as observed on the balance.

The degree of precision to be attained in the buoyancy correction depends upon the precision with which the volume of the weights and the density of the air can be determined. The

volume of the weights can be very accurately determined by hydrostatic weighing, a method familiar in its application to the determination of the density of solids. In such cases, of course, the density of the weight is not determined or used, the value of the volume sufficing for all purposes in the determination of the buoyancy corrections to the weights.

(b) **Density of the Air.**—The density of the atmosphere depends upon the temperature, pressure, and humidity, each of which factors must be separately determined.¹⁰

DETERMINATION OF TEMPERATURE.—The temperature may be determined by a thermometer, and although the corrections of a good thermometer are usually small, assurance should be had that this is the case and that the differences arising from thermometer errors are negligible.

DETERMINATION OF PRESSURE.—The pressure of the air is determined by a barometer. The readings of a mercurial barometer may need correction for instrumental errors,¹¹ such as residual gas pressure, capillary depression, and errors of the scale. The barometer height must be reduced to the height which would obtain at zero degree centigrade and under a standard value of gravity. The temperature reduction tables commonly given in books of physical and chemical tables are satisfactory. A brief table for this purpose is given on page 76. The value of standard gravity adopted by the International Conference on Weights and Measures is 980.665 dynes. For buoyancy corrections, however, the ordinary tables for reductions to sea level at latitude 45° are satisfactory, as extreme accuracy is not needed in this term.

DETERMINATION OF HUMIDITY.—The humidity may be determined by a hair hygrometer, a dew-point hygrometer, or a psychrometer. The various forms of the hair hygrometer are almost the only instruments that can be used inside of a closed balance case. They are not reliable under all conditions, however, and on that account must be calibrated frequently by comparison with a more reliable type of instrument.¹²

¹⁰ Although the composition of the air, aside from its contents of water vapor, may change very slightly, the effect of such change on the density of the air is entirely negligible even in work of high precision unless the carbon-dioxide content becomes large, as might occur when several persons are working in a poorly ventilated room.

¹¹ In most barometers the error due to capillary depression is eliminated to a large extent in the maker's calibration, the scale errors are negligible, and the determination of the residual gas pressure is very difficult of performance. On this account the instrumental corrections can best be determined directly by comparing the barometer with one whose corrections are accurately known.

¹² A hair hygrometer should not be tested by being placed in saturated vapor, as is often advised, for this leaves it almost worthless for some time and is likely to produce permanent changes in it.

To allow for the water vapor in the atmosphere a third reduction is made in the barometer. Representing by f , the actual pressure of the water vapor present in the air, the reduction term is $-0.378f$.

When a psychrometer is used, f is found from the usual psychrometer tables. But for the more common atmospheric conditions $0.378f$ may be found directly from the condensed table given on page 77. When the dew point is determined, f is equal to the saturation pressure at the dew point. When the relative humidity is found (as with a hair hygrometer), this per cent of the *saturation pressure* at the observed temperature gives f . A table of the pressures of saturated water vapor at different temperatures is given on page 78.

USE OF TABLES.—The actual density of the air may then be found by taking from a table of the densities of dry air the value corresponding to the temperature and the barometric height reduced as above. A condensed table of densities of dry air is given on page 79. The density of the air may be more conveniently, though with less precision, determined from Table 17, page 80. This table is, however, accurate enough for a great deal of precise weighing, since it gives values that are correct within a few tenths of 1 per cent.

(c) **Calculation Based on Density of Objects Compared: Indirect Method.**—If the volume of a weight has not been measured, it may be calculated with a fair degree of accuracy from the approximately known density of the material of which it is composed.¹³

The buoyancy corrections may be calculated directly in terms of the density of the weights and that of air. This may be done conveniently for nearly all work by using the buoyancy correction per gram of material, assuming the mass of the two weights to be the same. This is what is done in the usual approximate formula which is given below as formula No. 3.

COMPLETE FORMULA.—The complete formula for calculating the buoyancy corrections from densities is derived below; in this

¹³ Since the density of brass and some other materials is subject to considerable uncertainty and since, moreover, many weights have an interior cavity, care must be taken in forming a judgment of the accuracy of the calculation based on assumed densities. In the case of screw-knob weights or others having a closed cavity in the interior, the density of the material will not give an accurate value for the volume of the weight, since the cavity and the air inclosed within it are practically a part of the weight. However, if the cavity is connected by an opening or crevice with the exterior air, the weight in regard to buoyancy will act as though its volume were the net volume of metal which it contains. In the case of screw-knob weights it is practically impossible to determine whether or not the interior cavity is sealed off from the outer air, as tightness of the cavity is largely a matter of accident in the manufacture of the weight.

equation the mass is expressed directly in terms of the result of the weighing and the buoyancy corrections.

Let M_s = the *mass* of the standard,

M_n = the mass of the weight tested,

D_s = the density of the standard,¹⁴

D_n = the density of the weight tested,¹⁴

ρ = the density of the air,

g = the acceleration due to gravity;

then when the two weights are in equilibrium¹⁵ we have

$$\left(M_s - \frac{M_s \rho}{D_s}\right)g = \left(M_n - \frac{M_n \rho}{D_n}\right)g$$

Solving for M_n , we obtain

$$M_n = M_s \frac{\left(1 - \frac{\rho}{D_s}\right)}{\left(1 - \frac{\rho}{D_n}\right)} \quad (1)$$

Expanding, we obtain

$$M_n = M_s \left[1 + \frac{\rho}{D_n} - \frac{\rho}{D_s} + \left(\frac{\frac{\rho^2}{D_n^2} - \frac{\rho^2}{D_n D_s}}{1 - \frac{\rho}{D_n}} \right) \right] \quad (2)$$

APPROXIMATE FORMULA: THE USUAL METHOD.—The term in parentheses can usually be neglected because $\frac{\rho}{D_n}$ and $\frac{\rho}{D_s}$ are small. Omitting this term we have the usual expression

$$M_n = M_s \left(1 + \frac{\rho}{D_n} - \frac{\rho}{D_s} \right) \text{ or } M_n = M_s + \rho \left(\frac{1}{D_n} - \frac{1}{D_s} \right) M_s \quad (3)$$

The usual derivation may be seen in the second form of equation (3). $\left(\frac{1}{D_n} - \frac{1}{D_s}\right)$ is the difference in volume per unit of mass, therefore, $M_s \left(\frac{1}{D_n} - \frac{1}{D_s}\right)$ is the total difference in volume and

¹⁴ When there are weights of different densities on either pan, the masses and densities must be split up into the proper values; or as can generally be done safely, the weights of one density (generally the platinum and aluminum fractions of a gram) may first be reduced to values on an assumed density equal to that of the larger weights, as is recommended for general use in the case of ordinary analytical weights.

¹⁵ When part or all of the difference between the two weights is determined by the deflection of the pointer the weights are not in exact equilibrium. The difference determined by the deflection of the pointer must be added to the proper side of the equation and may be treated as material of the same density as that assumed for the weight by which the sensitiveness of the balance was determined. In all ordinary cases, however, this difference is so small that it may be added without regard to density.

$\rho M_s \left(\frac{1}{D_n} - \frac{1}{D_s} \right)$ is the difference in buoyant force, or the buoyancy correction.

In computing the buoyancy correction the errors of the weights may be safely neglected, as these errors cause such an exceedingly small difference in the volume of the weights that any difference arising from this small term is practically inappreciable. For all usual purposes, therefore, the nominal or designated values of the masses may be used.

In assuming, as has been done in the calculation of Table 18, "Buoyancy correction factors for materials of different densities," that the weights are of equal mass, a slight difference in volume has been neglected in addition to that arising from the neglect of the correction to the weights. This difference in volume exists on account of the fact that the weights are in equilibrium in air, and on this account the less dense weights have a slightly greater mass, as is necessary to counterbalance the greater buoyant force acting on them. In equation (2) the last term in the brackets, viz,

$$\frac{\frac{\rho^2}{D_n^2} - \frac{\rho^2}{D_n D_s}}{1 - \frac{\rho}{D_n}} \text{ which equals } \frac{\rho^2(D_s - D_n)}{(D_n - \rho)D_n D_s}$$

appears on this account. When the value of this term is calculated, it is seen to be negligible for weights having densities lying between 2.6 and 21 mg per cubic centimeter (this range of densities including practically all weights) except for weighings requiring precision higher than 1 in 1 000 000.

EFFECT OF VARIATIONS IN THE DENSITY OF THE AIR.—Since the value of a weight, or the corrections to it, are determined in order to permit the use of that weight in ascertaining the mass of some other object, the effect of errors in any density assumed for the weight is to be measured by the errors which arise in the use of the weight. Were the density of the air invariable, any desired value of density could be assumed for the weight in determining its value without causing error in the results obtained in using the weight, provided the same value of density were used in both cases; for, in the use of the weight in weighing, any such error originating in the determination of the mass of the weight would be automatically eliminated. On this account

it is in the variations in the density of the air that we are interested in determining the effect of errors in the values of density assumed. A case of particular importance is that in which weights of platinum (generally the fractions of a gram) are adjusted or determined on the basis of an assumed density of 8.4, the value usually used for brass. This is the adjustment usually made in order that platinum weights may be used with sets of brass weights without the necessity of constantly making buoyancy corrections.

The approximate formula for the effect of variations in the density of the air in such cases is given below in equation (5) and the two following paragraphs, the "correction" being equal to the variation in the value of the weight caused by a change of $\Delta\rho$ in the density of the air.

When a weight is tested under the assumption of a certain value for its density, D , in air of a certain density, ρ , the value found under those conditions may be called $M_{D,\rho}$. This quantity, which may be termed the "effective mass," is equal to the mass of an ideal standard of the assumed density that will just balance the weight considered, in air of the same density as that in which $M_{D,\rho}$ was determined. The error caused by using one value of $M_{D,\rho}$ when the weight is being used in air of a different density may then be calculated by the necessary changes in this ideal weight.

The following formula gives the correction to be applied to one value of $M_{D,\rho}$ to reduce it to that for a different density of the air, and similarly, the error caused in using one value of $M_{D,\rho}$ for a weight that is being used as a standard in air having a density different from that in which its effective mass was determined.

Let M_s = the mass of the ideal standard of the assumed density, whose true mass is equal to the value of $M_{D,\rho}$ of the weight under discussion,

M_n = the true mass of this weight,

D_s = the density of the ideal standard, this density being equal to that assumed for the weight.

D_n = the actual density of the weight,

ρ = the density of the air in which the effective mass was determined,

$\Delta\rho$ = the change in the density of the air.

From the same equation as that used in deriving the preceding formulae, we obtain,

$$M_n \left(1 - \frac{\rho}{D_n} \right) = M_s \left(1 - \frac{\rho}{D_s} \right)$$

When the density of the air is changed by an amount $\Delta\rho$, a change C will be required in the mass of the ideal weight in order to produce equilibrium. We then obtain

$$M_n \left(1 - \frac{\rho + \Delta\rho}{D_n} \right) = (M_s + C) \left(1 - \frac{\rho + \Delta\rho}{D_s} \right)$$

and C will be the correction sought. Expanding and rearranging the terms, we have

$$M_n \left(1 - \frac{\rho}{D_n} \right) - M_n \frac{\Delta\rho}{D_n} = M_s \left(1 - \frac{\rho}{D_s} \right) - M_s \frac{\Delta\rho}{D_s} + C \left(1 - \frac{\rho + \Delta\rho}{D_s} \right)$$

Solving for C we have

$$C = \frac{\Delta\rho \left(\frac{M_s}{D_s} - \frac{M_n}{D_n} \right)}{1 - \frac{\rho + \Delta\rho}{D_s}} \quad (4)$$

Simplifying this by the introduction of approximations sufficiently accurate, we obtain

$$C = \Delta\rho M_s \left(\frac{1}{D_s} - \frac{1}{D_n} \right) \quad (5)$$

From this it is to be observed that the correction may be calculated approximately as the change in the difference between the buoyant forces acting on the two weights, considering their masses as being equal. For determining this correction quickly, Table 18, "Buoyancy correction factors for materials of different densities," may be used.

As a rough estimate of the maximum variation per gram, we may take 0.1 times the total difference in buoyancy, for weights to be retained in one locality, and twice that amount if they are to be taken from sea level to an altitude of 6000 feet.

EFFECT OF ERROR IN ASSUMED DENSITY OF THE WEIGHT.—The actual error in the mass of the weight caused by an error in the assumed density of the weight is seldom of importance, since the weight is generally used for determining the mass of other objects in air not differing greatly in density from that in which the weight

was calibrated. As it may be of importance in some work to know the amount of this error, the complete formula for determining the correction is derived below.

- Let M_s = the approximate mass determined,
- M_n = the actual mass of the weight,
- D_s = the density assumed for the weight,
- D_n = the actual density of the weight,
- ρ = the density of the air,
- g = the acceleration due to gravity.

The weight in question may be considered as just balancing in air an ideal standard of the assumed density, whose true mass is the approximate mass found for the former. Therefore

$$\left(M_n - \frac{M_n}{D_n} \rho\right) g = \left(M_s - \frac{M_s}{D_s} \rho\right) g$$

Solving for M_n , we obtain

$$M_n = M_s \frac{1 - \frac{\rho}{D_s}}{1 - \frac{\rho}{D_n}}$$

Let $D_n = D_s + \Delta D_s$

Substituting for D_n its value, we have

$$M_n = M_s \left(1 - \frac{\rho}{D_s}\right) \left(\frac{1}{1 - \frac{\rho}{D_s + \Delta D_s}}\right)$$

$$M_n = M_s \left(1 - \frac{\rho}{D_s}\right) (D_s + \Delta D_s) \frac{1}{D_s + \Delta D_s - \rho}$$

$$M_n = M_s \frac{D_s - \rho + \Delta D_s - \frac{\rho \Delta D_s}{D_s}}{D_s - \rho + \Delta D_s}$$

$$M_n = M_s \left[1 - \frac{\rho \Delta D_s}{D_s(D_s - \rho + \Delta D_s)}\right]$$

Let ΔM_s = the correction that is to be added to the approximate mass M_s , that is, let $M_n = M_s + \Delta M_s$.

Substituting this value for M_n in the preceding equation, dividing through by M_s , and rearranging the signs of the second term, we have

$$1 + \frac{\Delta M_s}{M_s} = 1 + \frac{\rho \Delta D_s}{D_s(\rho - D_s - \Delta D_s)}$$

Putting $R = \frac{\Delta M_s}{M_s}$, which is the correction per unit of mass, we have, after subtracting 1 from each member,

$$R = \frac{\rho \Delta D_s}{D_s(\rho - D_s - \Delta D_s)} \quad (6)$$

or, solving for the difference in density ΔD_s , we have

$$\Delta D_s = \frac{RD_s(\rho - D_s)}{\rho + RD_s} \quad (7)$$

From this the allowable difference in density of the weight for a given difference in R can readily be found.

XI. REGULATIONS GOVERNING THE TESTING OF WEIGHTS

1. GENERAL REQUIREMENTS

Standards conforming to the general requirements given below will be accepted for regular test and certification. These include all weights that conform to the specifications for classes A, B, C, M, and S, and a few special weights worthy of test that do not come within the regular classification.

The Bureau reserves the right to reject any or all weights that do not conform effectively and in good faith to the foregoing specifications or following general requirements.

(a) Weights must be composed of material that is not readily oxidized or corroded by the atmosphere¹⁶ or else must be protected by a suitable coating of such material.

(b) All but the small weights must be composed of material at least as hard as brass. Weights below 5 g, or 60 grains, may be composed of aluminum or metals of similar hardness.

(c) Weights provided with a cavity for making adjustments, closed with a plug or removable knob or handle, must have but one cavity, and it must be small in proportion to the weight.¹⁷

(d) There must be no hole or aperture in the bottom of weights of any form, or in the sides of weights having a cylindrical or conical body, except where such form is specifically approved in the case of class C weights.

(e) Weights must be regular in shape and simple in design.

(f) The nominal values of the weights must be plainly and permanently marked on them (see list of abbreviations on page 71),

¹⁶ Brass or bronze oxidizes too rapidly to be used without surface protection.

¹⁷ Such weights as metal shells filled with lead will not be tested.

except on weights so small that this is impracticable. This requirement may be waived in the case of weights of class M and of old standards sent to be retested.

(g) With the exception of large cast-iron weights (class C grade, 20 pounds or over, only), all metal surfaces must be smooth and sufficiently well polished to show readily scratches or other injuries. The cast-iron weights must not have deep pits or sharp projections.

(h) Weights submitted for test and not in conformity with the requirements applying to any of the classes A, B, C, M, and S, as outlined in this circular, must be adjusted at least within the limits of error of class C

2. REJECTION OF WEIGHTS

Weights that do not conform to the specifications for the class under which they were submitted, or that do not conform to the general requirements, will be rejected. Notice of this fact will be sent to the apparent owner of the weights; that is, in general to the person to whom the weights are to be shipped from the Bureau.

If weights are rejected for inaccuracy or other faults that can not be detected without considerable labor, the fee for testing them will be the same as though they had been submitted separately.

When weights are bought under contract to conform to the specifications of this Bureau, much time and trouble may be saved to the purchaser by notifying the Bureau of this fact and having the weights shipped to the Bureau directly from the manufacturer or dealer. Under these conditions, rejected weights will be returned directly to the manufacturer or dealer, and the bill for testing rejected weights will also be sent to him.

Rejected weights may, upon special request, be tested and their corrections determined to the degree of precision that seems to be justified by the nature of the weights. In this case a "report" will be issued in place of the regular certificate. It will state definitely the cause of the rejection, as well as the corrections found. Weights rejected for inaccuracy will not be corrected by the Bureau unless it would involve unwarranted expense or delay to have them sent away for adjustment, nor unless it can be done without delaying the regular testing work of the Bureau. In no case will the Bureau make such corrections without specific

request by the person or firm submitting the weights. Fees for this work are given in Schedule 14, page 69.

3. IDENTIFICATION OF WEIGHTS TESTED BY THE BUREAU

The shipping case or the inner wrappings will always be sealed when weights that have been tested are shipped from the Bureau and will bear the Bureau's test number.

4. SUBMITTING WEIGHTS FOR TEST

(a) **Application for Test.**—Written requests should be made for all tests, even when the weights are delivered in person or by messenger. The request should state the class under which the weights are to be tested, together with the other information necessary for their proper test and identification.

If weights have already been used as standards in exacting work, and it is important to know what their corrections were at the close of such work, this fact should be stated; otherwise, weights are carefully cleaned before being tested.

(b) **Packing.**—The weights must be carefully packed in boxes that can be used in returning them. The weights, if shipped in their regular cases, should be tightly packed in their pockets by the use of extra material of some sort. The larger weights, when they are not shipped in cases containing pockets, should have the packing next the weight bound tightly against it to avoid the possibility of abrasion, and a liberal amount of the packing should be used.

In wrapping the better grades of weights some soft but firm material should be used next to the weight. If tissue paper is used, many thicknesses of it should be firmly wrapped on, and this should then be covered with thicker, tougher material such as cotton packing or knit goods. The whole should then be wrapped securely in wrapping paper in order to exclude the dust and grit from the rough packing in which the boxes or bundles are generally packed for shipment.

In many cases the milligram weights that are packed under the glass covers of the regular cases reach the Bureau bent or otherwise damaged. For this reason greater care should be given to the packing of these weights. Sufficient extra packing should be used to hold the glass cover firmly in place.

(c) **Consignor's Identification.**—All packages should bear the shipper's name and address, a list of the contents, and any other marks that may be necessary to insure ready identification. The

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(c) **Consignor's Identification.**—All packages should bear the shipper's name and address, a list of the contents, and any other marks that may be necessary to insure ready identification. The

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Schedule 12. SCIENTIFIC GROUP

Class M and weights given a similar test:	Per weight
(a) Determination of volume, single weights.	\$2. 00
(b) Determination of volume, two or more weights.	1. 50
(c) Determination of corrections, single weights. 80
(d) Determination of corrections, equal weights and sets made up in the series shown above. 50
(e) For constancy test add one-half of the fee for determining the corrections.	
Class S and weights given a similar test:	
(f) Regular test, single weights. 60
(g) Regular test, equal weights and sets made up in the series shown above. 40
(h) For constancy test add one-half of the fee for the regular test.	

Schedule 14. SPECIAL WORK (done only in unusual circumstances, as noted on page 66)

For adjusting gold-plated one-piece weights:

(a) Adjustment within the tolerances for classes A, B, and M when no plating is required, minimum fee.	1. 00
(b) Same as above, but plating required, minimum fee.	2. 00

For adjusting weights that have special means for adjusting as allowed in classes B, C, and S:

(c) Adjustment within the tolerances for class B or C when plugs or caps have not been inserted. 50
(d) Adjustment within the tolerances for class S, screw-knob weights or plugged weights in which the plugs or caps have not been inserted	1. 00

Special fees may be made to cover work not provided for in above schedules.

(f) **Remittances.**—Fees in accordance with the foregoing schedules should be sent with the request for test whenever practicable. Otherwise a bill will be rendered before the testing is begun. Certificates are not given, nor are the weights returned until the fees due thereon have been received. Remittances may be made by money order or check drawn to the order of the Secretary of Commerce.

S. W. STRATTON,
Director.

Approved:
WILLIAM C. REDFIELD,
Secretary.

APPENDIX

TABLE 8.—Short List of Abbreviations Recommended by this Bureau

Kilogram.....	kg
Gram.....	g
Centigram.....	cg
Milligram.....	mg
Pound, avoirdupois.....	lb.
Ounce, avoirdupois.....	oz.
Grain (avoirdupois and troy grains are identical).....	gr.
Pound, troy.....	lb. t.
Ounce, troy.....	oz. t.
Pennyweight.....	dwt.
Pound, apothecaries' weight.....	lb. ap.
Ounce, apothecaries' weight.....	oz. ap. <i>or</i> ℥
Dram, apothecaries' weight.....	dr. ap. <i>or</i> ℥
Scruple, apothecaries' weight.....	s. ap. <i>or</i> ℥
Carat (200 mg).....	c

This table contains practically all of the abbreviations needed for weights, but a more complete list will be found in Bureau Circular No. 47.

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TABLE 9.—Class T—Trade Weights: Tolerances, Customary System

Tolerances for trade weights as adopted by the Annual Conference on the Weights and Measures of the United States, and recommended by the Bureau of Standards for adoption by the several States. Complete specifications and tolerances for weights of this class will be found in Circular 61.

The tolerances to be allowed in excess or deficiency on commercial weights shall not be greater than the following values: Provided, however, that the manufacturers' tolerances or the tolerances to be allowed on new commercial weights shall not be greater than one-half of the values given.

Avoirdupois System

Weight	Tolerance, ordinary weights (ratio 1:1)	Tolerance, counterpoise weights for multiplying-lever scales		
		Ratio less than 100:1	Ratio 100:1 and less than 1000:1	Ratio 1000:1 and over
lbs.	gr.	gr.	gr.	gr.
50	100.0	60.0	40.0	20.0
25	60.0	36.0	24.0	12.0
20	60.0	36.0	24.0	12.0
15	40.0	24.0	16.0	8.0
10	40.0	24.0	16.0	8.0
8	30.0	18.0	12.0	6.0
5	30.0	18.0	12.0	6.0
4	20.0	12.0	8.0	4.0
3	20.0	12.0	8.0	4.0
2	15.0	9.0	6.0	3.0
1	10.0	6.0	4.0	2.0
oz.				
10	10.0	6.0	4.0	2.0
8	5.0	3.0	2.0	1.0
5	5.0	3.0	2.0	1.0
4	5.0	3.0	2.0	1.0
2	3.0	1.8	1.2	0.6
1	2.0	1.2	0.8	0.4
1/2	2.0	1.2	0.8	0.4
1/4	1.0	0.6	0.4	0.2
1/8	0.5	0.3	0.2	0.1
1/16	0.5	0.3	0.2	0.1
1/32	0.5	0.3	0.2	0.1
1/64	0.2	0.12	0.08	0.04

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TABLE 10.—Class T—Trade Weights: Tolerances, Metric System

The tolerances to be allowed on commercial weights of the metric system should be equivalent to those in the avoirdupois system given above, and the following table has been computed on that basis. The manufacturers' tolerances or the tolerances to be allowed on new commercial weights shall not be greater than one-half of the values given.

Weight	Ordinary weights (ratio 1:1)	Counterpoise weights for multiplying-lever scales		
		Ratio less than 100:1	Ratio 100:1 and less than 1000:1	Ratio 1000:1 and over
kg	g	g	g	g
20	6.0	3.6	2.4	1.2
10	4.0	2.4	1.6	0.8
5	2.5	1.5	1.0	0.5
2	1.5	0.9	0.6	0.3
1	1.0	0.6	0.4	0.2
g	mg	mg	mg	mg
500	700	420	280	140
200	400	240	160	80
100	300	180	120	60
50	200	120	80	40
20	100	60	40	20
10	70	42	28	14
5	50	30	20	10
2	30	18	12	6
1	20	12	8	4
mg				
500	15	9.0	6.0	3.0
200	7	4.2	2.8	1.4
100	5	3.0	2.0	1.0

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TABLE 11.—Prescription Weights: Tolerances

Tolerances adopted by the Annual Conference on the Weights and Measures of the United States, and recommended by the Bureau of Standards for adoption by the several States.

The tolerances to be allowed in excess or deficiency on apothecaries' prescription weights shall not be greater than the following values: Provided, however, That the manufacturers' tolerances or the tolerances to be allowed on new apothecaries' prescription weights shall not be greater than one-half of the values given.

Apothecaries' System

Weight	Tolerance	Weight	Tolerance
oz. ap.	gr.	s. ap.	gr.
12	4.0	3	0.3
10	4.0	2	0.25
8	3.0	1	0.15
5	3.0	gr.	
4	2.0	20	0.15
3	2.0	10	0.12
2	2.0	5	0.08
1	1.0	2	0.04
dr. ap.		1	0.03
8	1.0	0.5	0.02
6	1.0	0.2	0.015
4	0.7	0.1	0.010
3	0.6		
2	0.5		
1	0.3		
0.5	0.2		

Metric System

Weight	Tolerance	Weight	Tolerance
g	mg	mg	mg
500	350.0	500	7.0
200	200.0	200	4.0
100	150.0	100	3.0
50	100.0	50	2.0
20	50.0	20	1.0
10	40.0	10	1.0
5	25.0		
2	15.0		
1	10.0		

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TABLE 12.—Buoyancy Reduction Terms

This table gives buoyancy reduction terms for reducing corrections for weights from the basis of one assumed density to that of another assumed density. The reduction terms are for platinum, aluminum, and brass, for weighings made in air having a density of 1.2 mg per milliliter, and are based on the approximate formula

$$C = M\rho \left(\frac{1}{D_n} - \frac{1}{D_s} \right)$$

where C = the buoyancy reduction term.

ρ = the density of the air.

M = the mass of weight.

D_n = the assumed density to which the corrections are to be reduced.

D_s = the assumed from which the corrections are to be reduced.

The densities of the weights indicated in the table are the "standard densities," or the values corresponding to zero degree centigrade, but since in ordinary work the weights will usually be nearer a temperature of 20° C, the difference in volume and the buoyancy corrections are calculated for weights at 20° C.

These reduction terms are to be added algebraically to the corrections determined on the first basis. For example, if the correction for a platinum 500-mg weight is -0.054 mg, assuming its true density to be 21.5, then to obtain its apparent weight in air against brass weights, or its correction under an assumed density of 8.4, the reduction term +0.044 must be added to the former correction, giving -0.010 mg.

By using -0.010 as its correction, the weight may be considered as equivalent to a brass weight, and the errors arising from such use are only those consequent upon variations in the density of the air. Any such error will not under ordinary circumstances be more than one-tenth of the reduction term, or twice that amount if the weight is used in air having a density as low as that found at an altitude of 6000 feet.

Milligrams				Grains			
Denomi- nation	Platinum (21.5) to brass (8.4)	Aluminum (2.7) to brass (8.4)	Aluminum (2.7) to platinum (21.5)	Denomi- nation	Platinum (21.5) to brass (8.4)	Aluminum (2.7) to brass (8.4)	Aluminum (2.7) to platinum (21.5)
g	mg	mg	mg	gr.	gr.	gr.	gr.
1	+0.087	-0.302	-0.389	20	+0.00174	-0.00604	-0.00778
mg				10	+0.00087	-0.00302	-0.00389
500	+0.044	-0.151	-0.195	5	+0.00044	-0.00151	-0.00195
200	+0.017	-0.060	-0.078	2	+0.00017	-0.00060	-0.00078
100	+0.009	-0.030	-0.039	1	+0.00009	-0.00030	-0.00039
50	+0.004	-0.015	-0.019	0.5	+0.00004	-0.00015	-0.00019
20	+0.002	-0.006	-0.008	0.2	+0.00002	-0.00006	-0.00008
10	+0.001	-0.003	-0.004	0.1	+0.00001	-0.00003	-0.00004
5	+0.000	-0.002	-0.002	0.05	+0.00000	-0.00002	-0.00002
2	+0.000	-0.001	-0.001	0.02	+0.00000	-0.00001	-0.00001
1	+0.000	-0.000	-0.000				

Circular of the Bureau of Standards

TABLE 13.—Reduction of Barometric Height to Standard Temperature, Zero Degree Centigrade (Values are Subtractive and Given in Millimeters of Mercury)

This table is taken from the Smithsonian meteorological tables, Table No. 11, and is based on the formula

$$C = -B \frac{(m-l)t}{1+mt}$$

where C = correction for temperatures, in millimeters of mercury

B = observed height of the barometric column in millimeters of mercury

m = coefficient of expansion for mercury (0.0001818)

l = coefficient of linear expansion of brass (0.000184)

t = temperature of the barometer indicated by an attached thermometer

Tem- pera- ture of barom- eter	Height of mercury column in millimeters			Tem- pera- ture of barom- eter	Height of mercury column in millimeters			Tem- pera- ture of barom- eter	Height of mercury column in millimeters		
	740	760	780		740	760	780		740	760	780
° C				° C				° C			
15.0	1.81	1.86	1.91	20.0	2.41	2.47	2.54	25.0	3.01	3.09	3.17
.5	1.87	1.92	1.97	.5	2.47	2.54	2.60	.5	3.07	3.15	3.24
16.0	1.93	1.98	2.03	21.0	2.53	2.60	2.67	26.0	3.13	3.21	3.30
.5	1.99	2.04	2.10	.5	2.59	2.66	2.73	.5	3.19	3.28	3.36
17.0	2.05	2.10	2.16	22.0	2.65	2.72	2.79	27.0	3.25	3.34	3.42
.5	2.11	2.16	2.22	.5	2.71	2.78	2.86	.5	3.31	3.40	3.48
18.0	2.17	2.23	2.29	23.0	2.77	2.84	2.92	28.0	3.37	3.46	3.55
.5	2.23	2.29	2.35	.5	2.83	2.90	2.98	.5	3.43	3.52	3.62
19.0	2.29	2.35	2.41	24.0	2.89	2.97	3.05	29.0	3.49	3.58	3.68
.5	2.35	2.41	2.48	.5	2.95	3.03	3.11	.5	3.55	3.64	3.74

TABLE 14.—Reduction of Barometric Height for Atmospheric Humidity (Millimeters of Mercury)

This table is used for obtaining the proper reduction terms for humidity directly from the wet and dry bulb temperatures of a psychrometer, and is based on the formula

Reduction term = $0.378f$

where f is the actual ("partial") pressure of the water vapor in the air computed from the wet and dry bulb temperature readings of a psychrometer having rapid, forced ventilation, at a barometric height of 755 mm of mercury. A variation of 55 mm in the barometer height produces an error in the values of this table, varying from 0 at saturation to about 0.1 or 0.2 mm at low humidities.

		Temperature of wet bulb, degrees centigrade																														
Tem- pera- ture of air, de- grees centi- grade	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	
15	0.6	0.9	1.3	1.7	2.1	2.5	3.0	3.4	3.9	4.3	4.8	5.2																				
16	0.4	0.8	1.1	1.5	1.9	2.3	2.8	3.2	3.7	4.2	4.6	5.0																				
17	0.2	0.5	0.9	1.3	1.7	2.2	2.6	3.0	3.5	4.0	4.5	5.0	5.5																			
18		0.4	0.8	1.1	1.6	2.0	2.4	2.8	3.3	3.8	4.3	4.8	5.3	5.8																		
19		0.2	0.6	1.0	1.4	1.8	2.2	2.7	3.1	3.6	4.1	4.6	5.1	5.7	6.2																	
20			0.4	0.8	1.2	1.6	2.0	2.5	2.9	3.4	3.9	4.4	4.9	5.5	6.0	6.6																
21			0.2	0.6	1.0	1.4	1.8	2.3	2.7	3.2	3.7	4.2	4.7	5.3	5.8	6.4	7.0															
22				0.4	0.8	1.2	1.6	2.1	2.5	3.0	3.5	4.0	4.5	5.1	5.7	6.2	6.9	7.5														
23					0.6	1.0	1.5	1.9	2.4	2.8	3.3	3.8	4.4	4.9	5.5	6.1	6.7	7.3	8.0													
24						0.2	0.4	0.8	1.3	1.7	2.2	2.6	3.1	3.6	4.2	4.7	5.3	5.9	6.5	7.1	7.8	8.5										
25							0.6	1.1	1.5	2.0	2.5	2.9	3.4	4.0	4.5	5.1	5.7	6.3	6.9	7.6	8.3	9.0										
26								0.9	1.3	1.8	2.3	2.8	3.3	3.8	4.3	4.9	5.5	6.1	6.7	7.4	8.1	8.8	9.5									
27									1.1	1.6	2.1	2.6	3.1	3.6	4.1	4.7	5.3	5.9	6.5	7.2	7.9	8.6	9.3	10.1								
28										1.4	1.9	2.4	2.9	3.4	4.0	4.5	5.1	5.7	6.4	7.0	7.7	8.4	9.2	9.9	10.7							
29											1.7	2.2	2.7	3.2	3.8	4.3	4.9	5.5	6.2	6.8	7.5	8.2	9.0	9.7	10.5	11.4						
30												2.0	2.5	3.0	3.6	4.1	4.7	5.3	6.0	6.6	7.3	8.0	8.8	9.5	10.3	11.2	12.0					
31													2.3	2.8	3.4	4.0	4.5	5.2	5.8	6.5	7.1	7.8	8.6	9.4	10.1	11.0	11.8	12.7				
32														2.7	3.2	3.8	4.4	5.0	5.6	6.3	6.9	7.7	8.4	9.2	10.0	10.8	11.6	12.5	13.5			
33															3.0	3.6	4.2	4.8	5.4	6.1	6.8	7.5	8.2	9.0	9.8	10.6	11.5	12.4	13.3	14.3		
34																3.4	4.0	4.6	5.2	5.9	6.6	7.3	8.0	8.8	9.6	10.4	11.3	12.2	13.1	14.1	15.1	
35																	3.8	4.4	5.0	5.7	6.4	7.1	7.8	8.6	9.4	10.2	11.1	12.0	12.9	13.9	14.9	15.9

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TABLE 15.—Pressure of Saturated Water Vapor (Millimeters of Mercury)

This table gives pressures of saturated water vapor for different temperatures. The values for whole degrees were calculated by F. Henning (*Annalen der Physik*, **22**, 1907, Table 3, p. 626) from a formula by M. Thiesen, and the values for intermediate tenths of degrees were obtained at the Bureau by interpolation from Henning's values.

The later values for the pressure of saturated water vapor obtained by Scheel and Heuse in 1910 do not differ from the values in this table by more than 1 part in 2000

Temperature, degrees centigrade	Tenths of degrees									
	0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
0	4.58	4.61	4.65	4.68	4.71	4.75	4.78	4.82	4.85	4.88
1	4.92	4.96	4.99	5.03	5.07	5.10	5.14	5.18	5.21	5.25
2	5.29	5.33	5.37	5.40	5.44	5.48	5.52	5.56	5.60	5.64
3	5.68	5.72	5.76	5.80	5.85	5.89	5.93	5.97	6.01	6.06
4	6.10	6.14	6.19	6.23	6.27	6.32	6.36	6.41	6.45	6.50
5	6.54	6.59	6.63	6.68	6.72	6.77	6.82	6.87	6.91	6.96
6	7.01	7.06	7.11	7.16	7.21	7.26	7.31	7.36	7.41	7.46
7	7.51	7.56	7.61	7.66	7.72	7.77	7.82	7.88	7.93	7.98
8	8.04	8.10	8.15	8.21	8.26	8.32	8.38	8.44	8.49	8.55
9	8.61	8.67	8.73	8.79	8.84	8.90	8.96	9.03	9.09	9.15
10	9.21	9.27	9.33	9.39	9.46	9.52	9.58	9.65	9.71	9.77
11	9.84	9.91	9.97	10.04	10.11	10.18	10.25	10.31	10.38	10.45
12	10.52	10.59	10.66	10.73	10.80	10.87	10.94	11.01	11.08	11.16
13	11.23	11.30	11.38	11.45	11.52	11.60	11.67	11.75	11.83	11.90
14	11.98	12.06	12.14	12.22	12.29	12.37	12.45	12.53	12.62	12.70
15	12.78	12.86	12.95	13.03	13.11	13.20	13.28	13.37	13.46	13.54
16	13.63	13.72	13.81	13.89	13.98	14.08	14.17	14.26	14.35	14.44
17	14.53	14.62	14.72	14.81	14.90	15.00	15.09	15.19	15.29	15.38
18	15.48	15.58	15.68	15.77	15.87	15.97	16.07	16.17	16.28	16.38
19	16.48	16.58	16.69	16.79	16.89	17.00	17.10	17.21	17.32	17.42
20	17.53	17.64	17.75	17.86	17.97	18.08	18.19	18.31	18.42	18.53
21	18.65	18.77	18.88	19.00	19.12	19.23	19.35	19.47	19.59	19.71
22	19.83	19.95	20.07	20.19	20.32	20.44	20.57	20.69	20.82	20.94
23	21.07	21.20	21.33	21.45	21.58	21.72	21.85	21.98	22.11	22.25
24	22.38	22.51	22.65	22.79	22.92	23.06	23.20	23.33	23.47	23.61
25	23.75	23.89	24.03	24.18	24.32	24.47	24.61	24.76	24.91	25.06
26	25.21	25.36	25.51	25.66	25.82	25.97	26.12	26.28	26.43	26.58
27	26.74	26.90	27.05	27.21	27.37	27.53	27.69	27.86	28.02	28.18
28	28.35	28.52	28.68	28.85	29.02	29.18	29.35	29.52	29.69	29.87
29	30.04	30.21	30.39	30.56	30.74	30.92	31.09	31.27	31.45	31.64
30	31.82	32.00	32.19	32.37	32.56	32.75	32.93	33.12	33.31	33.50
31	33.69	33.88	34.07	34.27	34.46	34.66	34.86	35.06	35.26	35.46
32	35.66	35.86	36.07	36.27	36.48	36.68	36.89	37.10	37.31	37.52
33	37.73	37.94	38.15	38.37	38.58	38.80	39.02	39.24	39.46	39.68
34	39.90	40.12	40.35	40.57	40.80	41.03	41.25	41.48	41.71	41.94
35	42.17									

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TABLE 16.—Density of Dry Air (Milligrams per Milliliter)

This table gives the density at different temperatures of dry air containing about 0.04 per cent of CO₂ (which is an average value for the CO₂ content), the values being computed from the formula

$$C = \frac{1.293052}{1 + 0.00367 t} \times \frac{h}{760}$$

where *h* is pressure in millimeters of mercury at 0° C and standard gravity, and *t* is temperature in degrees centigrade.

Temperature in degrees centigrade.	Pressure in millimeters of Hg (0° C, standard gravity)											
	720	725	730	735	740	745	750	755	760	765	770	775
15	1.1611	1.1691	1.1772	1.1853	1.1933	1.2014	1.2095	1.2175	1.2256	1.2336	1.2417	1.2498
16	1.1571	1.1651	1.1731	1.1812	1.1892	1.1972	1.2053	1.2133	1.2213	1.2294	1.2374	1.2454
17	1.1531	1.1611	1.1691	1.1771	1.1851	1.1931	1.2011	1.2091	1.2171	1.2251	1.2331	1.2411
18	1.1491	1.1571	1.1650	1.1730	1.1810	1.1890	1.1970	1.2049	1.2129	1.2209	1.2289	1.2369
19	1.1451	1.1531	1.1611	1.1690	1.1770	1.1849	1.1929	1.2008	1.2088	1.2167	1.2247	1.2326
20	1.1412	1.1492	1.1571	1.1650	1.1729	1.1809	1.1888	1.1967	1.2046	1.2126	1.2205	1.2284
21	1.1373	1.1452	1.1531	1.1610	1.1689	1.1768	1.1847	1.1926	1.2005	1.2084	1.2163	1.2242
22	1.1335	1.1414	1.1492	1.1571	1.1650	1.1728	1.1807	1.1886	1.1965	1.2043	1.2122	1.2201
23	1.1296	1.1375	1.1453	1.1532	1.1610	1.1689	1.1767	1.1846	1.1924	1.2002	1.2081	1.2159
24	1.1258	1.1337	1.1415	1.1493	1.1571	1.1649	1.1727	1.1806	1.1884	1.1962	1.2040	1.2118
25	1.1220	1.1298	1.1376	1.1454	1.1532	1.1610	1.1688	1.1766	1.1844	1.1922	1.2000	1.2078
26	1.1183	1.1261	1.1338	1.1416	1.1494	1.1571	1.1649	1.1727	1.1804	1.1882	1.1959	1.2037
27	1.1146	1.1223	1.1300	1.1378	1.1455	1.1533	1.1610	1.1687	1.1765	1.1842	1.1920	1.1997
28	1.1108	1.1186	1.1263	1.1340	1.1417	1.1494	1.1571	1.1648	1.1726	1.1803	1.1880	1.1957
29	1.1072	1.1149	1.1225	1.1302	1.1379	1.1456	1.1533	1.1610	1.1687	1.1764	1.1840	1.1917
30	1.1035	1.1112	1.1188	1.1265	1.1342	1.1418	1.1495	1.1571	1.1648	1.1725	1.1801	1.1878
31	1.0999	1.1075	1.1151	1.1228	1.1304	1.1381	1.1457	1.1533	1.1610	1.1686	1.1762	1.1839

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TABLE 17.—Density of Air in Milligrams per Milliliter; Values Given Directly from the Temperature and the (Unreduced) Reading of the Barometer

This table is from *Theorie, Konstruktion und Gebrauch der Feineren Hebelwage*, by W. Felgentraeger. It is computed for air of 50 per cent relative humidity and for a place where "g" equals 981.288 cm/sec². Ordinary changes from these conditions may be expected to introduce errors of about five units in the last decimal place given. If more accurate results are required, reductions or corrections must be applied¹⁸ as noted below the table, on page 82.

The barometer readings should be corrected for instrumental errors such as errors of the scale or residual gas pressure if these errors are not negligible. The reductions for temperature and for gravity (latitude and elevation) are included in the computation of the table. The temperature used must be that at the balance case; the temperature of the barometer should not differ from this by more than 5°.

T b	16°	17°	18°	19°	20°	21°	22°	23°	24°	25°	26°	27°	28°
690	1.103	099	094	090	086	082	077	073	069	065	061	057	052
1	104	100	096	092	087	083	079	075	071	066	062	058	054
2	106	102	097	093	089	085	081	076	072	068	064	060	055
3	108	103	099	095	091	086	082	078	074	070	065	061	057
4	109	105	101	096	092	088	084	080	075	071	067	063	059
5	111	106	102	098	094	089	085	081	077	073	068	064	060
6	112	108	104	099	095	091	087	083	078	074	070	066	062
7	114	110	105	101	097	093	088	084	080	076	072	067	063
8	116	111	107	103	098	094	090	086	082	077	073	069	065
9	117	113	109	104	100	096	092	087	083	079	075	070	066
700	1.119	115	110	106	102	097	093	089	085	080	076	072	068
1	120	116	112	107	103	099	095	090	086	082	078	074	069
2	122	118	113	109	105	100	096	092	088	084	079	075	071
3	124	119	115	111	106	102	098	094	089	085	081	077	072
4	125	121	117	112	108	104	099	095	091	087	082	078	074
5	127	122	118	114	110	105	101	097	092	088	084	080	076
6	128	124	120	115	111	107	103	098	094	090	085	081	077
7	130	126	121	117	113	108	104	100	096	091	087	083	078
8	132	127	123	118	114	110	106	101	097	093	089	084	080
9	133	129	125	120	116	112	107	103	099	094	090	086	082
710	1.135	130	126	122	117	113	109	105	100	096	092	087	083
1	136	132	128	123	119	115	110	106	102	097	093	089	085
2	138	134	129	125	121	116	112	108	103	099	095	090	086
3	140	135	131	127	122	118	114	109	105	101	096	092	088
4	141	137	132	128	124	119	115	111	106	102	098	094	089
5	143	138	134	130	125	121	117	112	108	104	099	095	091
6	144	140	136	131	127	123	118	114	110	105	101	097	092
7	146	142	137	133	128	124	120	115	111	107	102	098	094
8	148	143	139	134	130	126	121	117	113	108	104	100	095
9	149	144	140	136	132	127	123	119	114	110	106	101	097
720	1.151	146	142	138	133	129	124	120	116	111	107	103	099
1	152	148	144	139	135	130	126	122	117	113	109	104	100
2	154	150	145	141	136	132	128	123	119	114	110	106	102
3	156	151	147	142	138	134	129	125	120	116	112	107	103
4	157	153	148	144	140	135	131	126	122	118	113	109	105
5	159	154	150	145	141	137	132	128	124	119	115	111	106
6	160	156	151	147	143	138	134	130	125	121	116	112	108
7	162	158	153	149	144	140	136	131	127	122	118	114	109
8	164	159	155	150	146	141	137	133	128	124	120	115	111
9	165	161	156	152	147	143	139	134	130	125	121	117	112
730	1.167	162	158	153	149	145	140	136	131	127	123	118	114
1	169	164	160	155	151	146	142	137	133	129	124	120	115
2	170	166	161	157	152	148	143	139	135	130	126	121	117
3	172	167	163	158	154	149	145	140	136	132	127	123	119
4	173	169	164	160	155	151	147	142	138	133	129	124	120
5	175	170	166	161	157	153	148	144	139	135	130	126	122
6	177	172	168	163	159	154	150	145	141	136	132	128	123
7	178	174	169	165	160	156	151	147	142	138	133	129	125
8	180	175	171	166	162	157	153	148	144	139	135	131	126
9	181	177	172	168	163	159	154	150	145	141	136	132	128
740	1.183	178	174	169	165	160	156	151	147	142	138	134	129

¹⁸When a large number of corrections or reductions must be introduced it is generally as easy and often preferable for other reasons to determine the density from other tables.

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Interpolation Table

$\Delta\tau$	Δb									
Δb	0.0°	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
mm										
0.0	0	- 0	- 1	- 1	- 2	- 2	- 3	- 3	- 4	- 4
0.1	+ 0	- 0	- 1	- 1	- 2	- 2	- 3	- 3	- 4	- 4
0.2	+ 0	- 0	- 1	- 1	- 2	- 2	- 3	- 3	- 4	- 4
0.3	+ 0	+ 0	- 0	- 1	- 1	- 2	- 2	- 3	- 3	- 4
0.4	+ 1	+ 0	- 0	- 1	- 1	- 2	- 2	- 3	- 3	- 4
0.5	+ 1	+ 0	- 0	- 1	- 1	- 2	- 2	- 2	- 3	- 3
0.6	+ 1	+ 0	+ 0	- 0	- 1	- 1	- 2	- 2	- 3	- 3
0.7	+ 1	+ 1	+ 0	- 0	- 1	- 1	- 2	- 2	- 3	- 3
0.8	+ 1	+ 1	+ 0	- 0	- 1	- 1	- 2	- 2	- 2	- 3
0.9	+ 1	+ 1	+ 0	+ 0	- 0	- 1	- 2	- 1	- 2	- 3

τ	b												
b	16°	17°	18°	19°	20°	21°	22°	23°	24°	25°	26°	27°	28°
740	1.183	178	174	169	165	160	156	151	147	142	138	134	129
1	185	180	176	171	166	162	157	153	148	144	140	135	131
2	186	182	177	173	168	164	159	155	150	146	141	137	132
3	188	183	179	174	170	165	161	156	152	147	143	138	134
4	189	185	180	176	171	167	162	158	153	149	144	140	136
5	191	186	182	177	173	168	164	159	155	150	146	141	137
6	193	188	183	179	174	170	165	161	156	152	147	143	139
7	194	190	185	180	176	171	167	162	158	153	149	144	140
8	196	191	187	182	177	173	168	164	159	155	150	146	142
9	197	193	188	184	179	175	170	166	161	157	152	148	143
750	1.199	194	190	185	181	176	172	167	163	158	154	149	145
1	201	196	191	187	182	178	173	169	164	160	155	151	146
2	202	198	193	188	184	179	175	170	166	161	157	152	148
3	204	199	195	190	185	181	176	172	167	163	158	154	149
4	205	201	196	192	187	182	178	173	169	164	160	155	151
5	207	202	198	193	189	184	179	175	170	166	161	157	152
6	209	204	199	195	190	186	181	177	172	167	163	158	154
7	210	206	201	196	192	187	183	178	174	169	164	160	155
8	212	207	203	198	193	189	184	180	175	171	166	161	157
9	213	209	204	200	195	190	186	181	177	172	168	163	158
760	1.215	210	206	201	196	192	187	183	178	174	169	165	160
1	217	212	207	203	198	193	189	184	180	175	171	166	162
2	218	214	209	204	200	195	190	186	181	177	172	168	163
3	220	215	211	206	201	197	192	187	183	178	174	169	165
4	221	217	212	208	203	198	194	189	184	180	175	171	166
5	223	218	214	209	204	200	195	191	186	181	177	172	168
6	225	220	215	211	206	201	197	192	188	183	178	174	169
7	226	222	217	212	208	203	198	194	189	184	180	175	171
8	228	223	219	214	209	204	200	195	191	186	181	177	172
9	230	225	220	215	211	206	201	197	192	188	183	178	174
770	1.231	226	222	217	212	208	203	198	194	189	185	180	175
1	233	228	223	219	214	209	205	200	195	191	186	182	177
2	234	230	225	220	215	211	206	201	197	192	188	183	178
3	236	231	227	222	217	212	208	203	198	193	189	185	180
4	238	233	228	223	219	214	209	205	200	195	191	186	182
5	239	234	230	225	220	216	211	206	202	197	192	188	183
6	241	236	231	227	222	217	212	208	203	198	194	189	185
7	242	238	233	228	223	219	214	209	205	200	195	191	186
8	244	239	234	230	225	220	216	211	206	202	197	192	188
9	246	241	236	231	227	222	217	213	208	203	199	194	189
780	247	242	238	233	228	223	219	214	209	205	200	195	191

Humidity Correction

t	Per cent	10	20	30	40	50	60	70	80	90	100
°C											
10	+2	+2	+1	+1	0	-1	-1	-2	-2	-3	
20	+4	+3	+2	+1	0	-1	-2	-3	-4	-5	
30	+7	+6	+4	+2	0	-2	-4	-6	-7	-9	

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To insure the accuracy of the last figure given in the table, greater attention must be given to the temperature of the barometer, to the reduction of the barometer for gravity, and to the relative humidity of the air.

The temperature of the barometer must be within 2° of the temperature of the balance case, which is the temperature to be used; otherwise the barometer height may be reduced to what it would be at that temperature.

If the local value of "g" differs from 981.288 cm/sec² by 1.000 unit, then the usual reduction for gravity may be made but -0.5 mm must be added to this reduction term in order to eliminate the reduction that is included in the table. As this gravity reduction term is a constant for any one locality, it need be calculated only once for any one place.

Corrections for variations from 50 per cent relative humidity may be found in the appended "Humidity correction table."

TABLE 18.—Buoyancy Correction Factors for Materials of Different Densities,¹⁹ Milligrams per Gram

In this table, above the diagonal line are given the buoyancy corrections per gram for material of several different densities, the proper correction being found at the intersection of the vertical column under the density of one of the materials, with the horizontal row corresponding to the density of the other material. The density of air is taken as 1.2 mg per milliliter. The differences in volume are also given below the diagonal line for use where 1.2 mg per milliliter is not a sufficiently close approximation to the density of the air.

Density grams per milliliter		21.5	21.1	8.4	8.3	7.8	7.4	2.7	2.6	
21.5	Difference in volume, milliliter		0.00108	0.0870	0.0888	0.0980	0.1063	0.389	0.406	Buoyancy correction, milligrams per gram
21.1		0.0009		0.0859	0.0877	0.0970	0.1052	0.388	0.405	
8.4		0.0725	0.0716		0.0018	0.0110	0.0193	0.302	0.319	
8.3		0.0740	0.0731	0.0015		0.0092	0.0175	0.300	0.317	
7.8		0.0817	0.0808	0.0092	0.0077		0.0083	0.291	0.308	
7.4		0.0886	0.0877	0.0161	0.0146	0.0069		0.282	0.299	
2.7		0.3239	0.3230	0.2514	0.2499	0.2422	0.2353		0.299	
2.6		0.3381	0.3372	0.2656	0.2641	0.2564	0.2495	0.0142	0.0170	

¹⁹ On account of differences in the coefficients of expansion of different materials, the difference in volume observed or computed will not be constant, but will vary with the temperature. For example, if a platinum and a brass weight are changed in temperature from 0° to 20° C the difference in volume of the weights will change by 0.1 ml per kilogram and consequently the buoyancy correction will be altered by a little more than 0.1 mg per kilogram. Errors from this source, however, are negligible for the range of accuracy for which this table is suitable.

DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS
Washington 25, D. C.

Errata to Accompany

NBS Handbook 77 - Volume III

Precision Measurement and Calibration

Optics, Metrology, and Radiation

Printed on the reverse side of this sheet is a supplementary page numbered 699/82 for inserting in NBS Handbook 77, Volume III, Optics, Metrology, and Radiation.

This page was omitted in the preparation of this volume for printing. The page printed in the volume is a duplicate of page 698/81.

March 4, 1963
USCOMM-NBS-DC

Standards of Mass

Interpolation Table

$\Delta\tau$	Δb									
Δb	0.0°	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
mm										
0.0	0	-0	-1	-1	-2	-2	-3	-3	-4	-4
0.1	+0	-0	-1	-1	-2	-2	-3	-3	-4	-4
0.2	+0	-0	-1	-1	-2	-2	-3	-3	-4	-4
0.3	+0	+0	-0	-1	-1	-2	-2	-3	-3	-4
0.4	+1	+0	-0	-1	-1	-2	-2	-3	-3	-4
0.5	+1	+0	-0	-1	-1	-2	-2	-3	-3	-4
0.6	+1	+0	+0	-0	-1	-1	-2	-2	-3	-3
0.7	+1	+1	+0	-0	-1	-1	-2	-2	-3	-3
0.8	+1	+1	+0	-0	-1	-1	-2	-2	-3	-3
0.9	+1	+1	+0	+0	-0	-1	-2	-2	-3	-3

τ	b												
b	16°	17°	18°	19°	20°	21°	22°	23°	24°	25°	26°	27°	28°
740	1.183	178	174	169	165	160	156	151	147	142	138	134	129
1	185	180	176	171	166	162	157	153	148	144	140	135	131
2	186	182	177	173	168	164	159	155	150	146	141	137	132
3	188	183	179	174	170	165	161	156	152	147	143	138	134
4	189	185	180	176	171	167	162	158	153	149	144	140	136
5	191	186	182	177	173	168	164	159	155	150	146	141	137
6	193	188	183	179	174	170	165	161	156	152	147	143	139
7	194	190	185	180	176	171	167	162	158	153	149	144	140
8	196	191	187	182	177	173	168	164	159	155	150	146	142
9	197	193	188	184	179	175	170	166	161	157	152	148	143
750	1.199	194	190	185	181	176	172	167	163	158	154	149	145
1	201	196	191	187	182	178	173	169	164	160	155	151	146
2	202	198	193	188	184	179	175	170	166	161	157	152	148
3	204	199	195	190	185	181	176	172	167	163	158	154	149
4	205	201	196	192	187	182	178	173	169	164	160	155	151
5	207	202	198	193	189	184	179	175	170	166	161	157	152
6	209	204	199	195	190	186	181	177	172	167	163	158	154
7	210	206	201	196	192	187	183	178	174	169	164	160	155
8	212	207	203	198	193	189	184	180	175	171	166	161	157
9	213	209	204	200	195	190	186	181	177	172	168	163	158
760	1.215	210	206	201	196	192	187	183	178	174	169	165	160
1	217	212	207	203	198	193	189	184	180	175	171	166	162
2	218	214	209	204	200	195	190	186	181	177	172	168	163
3	220	215	211	206	201	197	192	187	183	178	174	169	165
4	221	217	212	208	203	198	194	189	184	180	175	171	166
5	223	218	214	209	204	200	195	191	186	181	177	172	168
6	225	220	215	211	206	201	197	192	188	183	178	174	169
7	226	222	217	212	208	203	198	194	189	184	180	175	171
8	228	223	219	214	209	204	200	195	191	186	181	177	172
9	230	225	220	215	211	206	201	197	192	188	183	178	174
770	1.231	226	222	217	212	208	203	198	194	189	185	180	175
1	233	228	223	219	214	209	205	200	195	191	186	182	177
2	234	230	225	220	215	211	206	201	197	192	188	183	178
3	236	231	227	222	217	212	208	203	198	193	189	185	180
4	238	233	228	223	219	214	209	205	200	195	191	186	182
5	239	234	230	225	220	216	211	206	202	197	192	188	183
6	241	236	231	227	222	217	212	208	203	198	194	189	185
7	242	238	233	228	223	219	214	209	205	200	195	191	186
8	244	239	234	230	225	220	216	211	206	202	197	192	188
9	246	241	236	231	227	222	217	213	208	203	199	194	189
780	247	242	238	233	228	223	219	214	209	205	200	195	191

Humidity Correction

Per cent	10	20	30	40	50	60	70	80	90	100
t										
°C										
10	+2	+2	+1	+1	0	-1	-1	-2	-2	-3
20	+4	+3	+2	+1	0	-1	-2	-3	-4	-5
30	+7	+6	+4	+2	0	-2	-4	-6	-7	-9

Standards of Mass

TABLE 19.—Definitions of Units of Mass

FUNDAMENTAL UNITS

A KILOGRAM (kg) is a unit of mass equivalent to the mass of the international prototype kilogram at the International Bureau of Weights and Measures.

AN AVOIRDUPOIS POUND (lb. av.) is a unit of mass equivalent to 0.453 592 427 7 kilogram.

A GRAM (g) is a unit of mass equivalent to one-thousandth of the mass of the international prototype kilogram at the International Bureau of Weights and Measures.

A TROY POUND (lb. t.) is a unit of mass equivalent to $\frac{5760}{7000}$ of that of the avoirdupois pound.

MULTIPLES AND SUBMULTIPLES

1 metric ton (t) = 1000 kilograms.

1 hectogram (hg) = 100 grams = 0.1 kilogram.

1 dekagram (dkg) = 10 grams = 0.01 kilogram.

1 decigram (dg) = 0.1 gram.

1 centigram (cg) = 0.01 gram.

1 milligram (mg) = 0.001 gram.

1 avoirdupois ounce (oz. av.) = $\frac{1}{16}$ avoirdupois pound.

1 avoirdupois dram (dr. av.) = $\frac{1}{256}$ avoirdupois pound = $\frac{1}{16}$ avoirdupois ounce.

1 grain (gr.) = $\frac{1}{7000}$ avoirdupois pound = $\frac{1}{537.5}$ avoirdupois ounce = $\frac{1}{3750}$ troy pound.

1 apothecaries' pound (lb. ap.) = 1 troy pound = $\frac{5760}{7000}$ avoirdupois pound.

1 apothecaries' or troy ounce (oz. ap. or ℥, or oz. t.) = $\frac{1}{12}$ troy pound = $\frac{480}{7000}$ avoirdupois pound = 480 grains.

1 apothecaries' dram (dr. ap. or ℥) = $\frac{1}{36}$ apothecaries' pound = $\frac{1}{8}$ apothecaries' ounce = 60 grains.

1 pennyweight (dwt.) = $\frac{1}{20}$ troy ounce = 24 grains.

1 apothecaries' scruple (s. ap. or ℥) = $\frac{1}{3}$ apothecaries' dram = 20 grains.

1 metric carat²⁰ (c) = 200 milligrams = 0.2 gram.

1 short hundredweight (sh. cwt.) = 100 avoirdupois pounds.

1 long hundredweight (l. cwt.) = 112 avoirdupois pounds.

1 short ton (sh. tn.) = 2000 avoirdupois pounds.

1 long ton (l. tn.) = 2240 avoirdupois pounds.

²⁰ See Bureau of Standards Circular No. 43, The Metric Carat, for a discussion of the metric carat and tables of the relations of the metric carat to the old carat of 205.3 mg in use in this country previous to July 7, 1913.

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TABLE 20.—Conversion Factors for Units of Mass

UNITS OF MASS LESS THAN POUNDS AND KILOGRAMS

Units	Grains	Apothecaries' scruples	Pennyweights	Avoirdupois drams
1 grain =	1	0.05	0.041 666 67	0.036 571 43
1 apoth. scruple =	20	1	0.833 333 3	0.731 428 6
1 pennyweight =	24	1.2	1	0.877 714 3
1 avoird. dram =	27.343 75	1.367 187 5	1.139 323	1
1 apoth. dram =	60	3	2.5	2.194 286
1 avoird. ounce =	437.5	21.875	18.229 17	16
1 apoth. or troy ounce =	480	24	20	17.554 28
1 apoth. or troy pound =	5760	288	240	210.6514
1 avoird. pound =	7000	350	291.6667	256
1 milligram =	0.015 432 356	0.000 771 618	0.000 643 014 8	0.000 564 383 3
1 gram =	15.432 356	0.771 618	0.643 014 85	0.564 383 3
1 kilogram =	15 432.356	771.6178	643.014 85	564.383 32

Units	Apothecaries' drams	Avoirdupois ounces	Apothecaries' or troy ounces	Apothecaries' or troy pounds
1 grain =	0.016 666 7	0.002 285 71	0.002 083 33	0.000 173 611 1
1 apoth. scruple =	0.333 333	0.045 714 3	0.041 666 7	0.003 472 222
1 pennyweight =	0.4	0.054 857.1	0.05	0.004 166 667
1 avoird. dram =	0.455 729 2	0.0625	0.056 966 146	0.004 747 178 8
1 apoth. dram =	1	0.137 142 9	0.125	0.010 416 667
1 avoird. ounce =	7.291 66	1	0.911 458 3	0.075 954 861
1 apoth. or troy ounce =	8	1.097 142 9	1	0.083 333 33
1 apoth. or troy pound =	96	13.165 714	12	1
1 avoird. pound =	116.6667	16	14.583 333	1.215 277 8
1 milligram =	0.000 257 205 9	0.000 035 273 96	0.000 032 150 74	0.000 002 679 23
1 gram =	0.257 205 9	0.035 273 96	0.032 150 74	0.002 679 23
1 kilogram =	257.205 94	35.273 96	32.150 742	2.679 228 5

Units	Avoirdupois pounds	Milligrams	Grams	Kilograms
1 grain =	0.000 142 857 1	64.798 918	0.064 798 918	0.000 064 798 9
1 apoth. scruple =	0.002 857 143	1295.9784	1.295 978 4	0.001 295 978
1 pennyweight =	0.003 428 571	1555.1740	1.555 174 0	0.001 555 174
1 avoird. dram =	0.003 906 25	1771.8454	1.771 845 4	0.001 771 845
1 apoth. dram =	0.008 571 429	3887.9351	3.887 935 1	0.003 887 935
1 avoird. ounce =	0.0625	28 349.527	28.349 527	0.028 349.53
1 apoth. or troy ounce =	0.068 571 43	31 103.481	31.103 481	0.031 103 48
1 apoth. or troy pound =	0.822 857.1	373 241.77	373.241 77	0.373 241 77
1 avoird. pound =	1	453 592.4277	453.592 427 7	0.453 592 427 7
1 milligram =	0.000 002 204 62	1	0.001	0.000 001
1 gram =	0.002 204 62	1000	1	0.001
1 kilogram =	2.204 622 341	1 000 000	1000	1

UNITS OF MASS GREATER THAN AVOIRDUPOIS OUNCES

Units	Avoirdupois ounces	Avoirdupois pounds	Short hundred-weights	Short tons
1 avoirdupois ounce =	1	0.0625	0.000 625	0.000 031 25
1 avoirdupois pound =	16	1	0.01	0.0005
1 short hundredweight =	1600	100	1	0.05
1 short ton =	32 000	2000	20	1
1 long ton =	35 840	2240	22.4	1.12
1 kilogram =	35.273 957	2.204 622 34	0.022 046 223	0.001 102 311 2
1 metric ton =	35 273.957	2204.622 34	22.046 223	1.102 311 2

Units	Long tons	Kilograms	Metric tons
1 avoirdupois ounce =	0.000 027 901 79	0.028 349 53	0.000 028 349 53
1 avoirdupois pound =	0.000 446 428 6	0.453 592 427 7	0.000 453 592 43
1 short hundredweight =	0.044 642 86	45.359 243	0.045 359 243
1 short ton =	0.892 857 1	907.184 86	0.907 184 86
1 long ton =	1	1016.047 04	1.016 047 04
1 kilogram =	0.000 984 206 4	1	0.001
1 metric ton =	0.984 206 40	1000	1

Standards of Mass

TABLE 21.—Comparison of the Various Tons and Pounds in Use in the United States (from 1 to 9 Units)

Troy pounds	Avoirdupois pounds	Kilograms	Short tons	Long tons	Metric tons
1	0.822 857	0.373 24	0.000 411 43	0.000 367 35	0.000 373 24
2	1.645 71	0.746 48	0.000 822 86	0.000 734 69	0.000 746 48
3	2.468 57	1.119 73	0.001 234 29	0.001 102 04	0.001 119 73
4	3.291 43	1.492 97	0.001 645 71	0.001 469 39	0.001 492 97
5	4.114 29	1.866 21	0.002 057 14	0.001 836 73	0.001 866 21
6	4.937 14	2.239 45	0.002 468 57	0.002 204 08	0.002 239 45
7	5.760 00	2.612 69	0.002 880 00	0.002 571 43	0.002 612 69
8	6.582 86	2.985 93	0.003 291 43	0.002 938 78	0.002 985 93
9	7.405 71	3.359 18	0.003 702 86	0.003 306 12	0.003 359 18
1.215 28	1	0.453 59	0.0005	0.000 446 43	0.000 453 59
2.430 56	2	0.907 18	0.0010	0.000 892 86	0.000 907 18
3.645 83	3	1.360 78	0.0015	0.001 339 29	0.001 360 78
4.861 11	4	1.814 37	0.0020	0.001 785 71	0.001 814 37
6.076 39	5	2.267 96	0.0025	0.002 232 14	0.002 267 96
7.291 67	6	2.721 55	0.0030	0.002 678 57	0.002 721 55
8.506 94	7	3.175 15	0.0035	0.003 125 00	0.003 175 15
9.722 22	8	3.628 74	0.0040	0.003 571 43	0.003 628 74
10.937 50	9	4.082 33	0.0045	0.004 017 86	0.004 082 33
2.679 23	2.204 62	1	0.001 102 31	0.000 984 21	0.001
5.358 46	4.409 24	2	0.002 204 62	0.001 968 41	0.002
8.037 69	6.613 87	3	0.003 306 93	0.002 952 62	0.003
10.716 91	8.818 49	4	0.004 409 24	0.003 936 83	0.004
13.937 50	11.023 11	5	0.005 511 56	0.004 921 03	0.005
16.075 37	13.227 73	6	0.006 613 87	0.005 905 24	0.006
18.754 60	15.432 36	7	0.007 716 18	0.006 889 44	0.007
21.433 83	17.636 98	8	0.008 818 49	0.007 873 65	0.008
24.113 06	19.841 60	9	0.009 920 80	0.008 857 86	0.009
2430.56	2000	907.18	1	0.892 87	0.907 18
4861.11	4000	1814.37	2	1.785 71	1.814 37
7291.67	6000	2721.55	3	2.678 57	2.721 55
9722.22	8000	3628.74	4	3.571 43	3.628 74
12 152.78	10 000	4535.92	5	4.464 29	4.535 92
14 583.33	12 000	5443.11	6	5.357 14	5.443 11
17 013.89	14 000	6350.29	7	6.250 00	6.350 29
19 444.44	16 000	7257.48	8	7.142 86	7.257 48
21 875.00	18 000	8164.66	9	8.035 71	8.164 66
2722.22	2240	1016.05	1.12	1	1.016 05
5444.44	4480	2032.09	2.24	2	2.032 09
8166.67	6720	3048.14	3.36	3	3.048 14
10 888.89	8960	4064.19	4.48	4	4.064 19
13 611.11	11 200	5080.24	5.60	5	5.080 24
16 333.33	13 440	6096.28	6.72	6	6.096 28
19 055.56	15 680	7112.32	7.84	7	7.112 32
21 777.78	17 920	8128.38	8.96	8	8.128 38
24 500.00	20 160	9144.42	10.08	9	9.144 42
2679.23	2204.62	1000	1.102 31	0.984 21	1
5358.48	4409.24	2000	2.204 62	1.968 41	2
8037.69	6613.87	3000	3.306 93	2.952 62	3
10 716.91	8818.49	4000	4.409 24	3.936 83	4
13 937.50	11 023.11	5000	5.511 56	4.921 03	5
16 075.37	13 227.73	6000	6.613 87	5.905 24	6
18 754.60	15 432.36	7000	7.716 18	6.889 44	7
21 433.83	17 636.98	8000	8.818 49	7.873 65	8
24 113.06	19 841.60	9000	9.920 80	8.857 86	9

TABLE 22.—Equivalents in Kilograms of 1 to 999 Avoirdupois Pounds

[Reduction factor: 1 avoirdupois pound=0.453592477 kilogram]

Pounds	Kilos	Pounds	Kilos	Pounds	Kilos	Pounds	Kilos	Pounds	Kilos	Pounds	Kilos	Pounds	Kilos	Pounds	Kilos	Pounds	Kilos	Pounds	Kilos		
0	0.45359	100	45.35924	200	90.71849	300	136.07773	400	181.43697	500	226.79621	600	272.15546	700	317.51470	800	362.87394	900	408.23318		
1	0.45359	1	45.81284	1	91.17208	1	136.53132	1	181.99056	1	227.44981	1	272.60905	1	317.96829	1	363.32753	1	408.68678	1	408.68678
2	0.90718	2	46.26643	2	91.62567	2	136.98491	2	182.44416	2	227.90340	2	273.06810	2	318.42188	2	363.78113	2	409.14037	2	409.14037
3	1.36078	3	46.72002	3	92.07926	3	137.43851	3	182.89775	3	228.35699	3	273.51623	3	318.87548	3	364.23472	3	409.59396	3	409.59396
4	1.81437	4	47.17361	4	92.53286	4	137.89210	4	183.35134	4	228.80583	4	273.96983	4	319.32307	4	364.68831	4	410.04755	4	410.04755
5	2.26796	5	47.62720	5	92.98645	5	138.34569	5	183.80493	5	229.25472	5	274.42342	5	319.78266	5	365.14190	5	410.50115	5	410.50115
6	2.72155	6	48.08080	6	93.44004	6	138.79928	6	184.25853	6	229.70817	6	274.87701	6	320.23625	6	365.59550	6	410.95474	6	410.95474
7	3.17515	7	48.53439	7	93.89363	7	139.25288	7	184.71212	7	230.16156	7	275.33060	7	320.68985	7	366.04909	7	411.40833	7	411.40833
8	3.62874	8	48.98798	8	94.34722	8	139.70647	8	185.16571	8	230.61525	8	275.78420	8	321.14344	8	366.50268	8	411.86192	8	411.86192
9	4.08233	9	49.44157	9	94.80082	9	140.16006	9	185.61930	9	231.06885	9	276.23779	9	321.59703	9	366.95627	9	412.31552	9	412.31552
10	4.53592	110	49.89517	210	95.25441	310	140.61365	410	186.07290	510	231.52214	610	276.69138	710	322.05062	810	367.40987	910	412.76911		
1	4.98952	1	50.34876	1	95.70800	1	141.06725	1	186.52649	1	231.97573	1	277.14497	1	322.50422	1	367.86346	1	413.22270	1	413.22270
2	5.44311	2	50.80235	2	96.16159	2	141.52084	2	186.98008	2	232.42932	2	277.59857	2	322.95781	2	368.31705	2	413.67629	2	413.67629
3	5.89670	3	51.25594	3	96.61519	3	141.97443	3	187.43367	3	232.88292	3	278.05216	3	323.41140	3	368.77064	3	414.12989	3	414.12989
4	6.35029	4	51.70954	4	97.06878	4	142.42802	4	187.88727	4	233.33651	4	278.50575	4	323.86499	4	369.22424	4	414.58348	4	414.58348
5	6.80389	5	52.16313	5	97.52237	5	142.88161	5	188.34086	5	233.79000	5	278.95934	5	324.31859	5	369.67783	5	415.03707	5	415.03707
6	7.25748	6	52.61672	6	97.97596	6	143.33521	6	188.79445	6	234.24369	6	279.41294	6	324.77218	6	370.13142	6	415.49066	6	415.49066
7	7.71107	7	53.07031	7	98.42956	7	143.78880	7	189.24804	7	234.69729	7	279.86653	7	325.22577	7	370.58501	7	415.94326	7	415.94326
8	8.16466	8	53.52391	8	98.88315	8	144.24239	8	189.70163	8	235.15088	8	280.32012	8	325.67936	8	371.03861	8	416.39785	8	416.39785
9	8.61826	9	53.97750	9	99.33674	9	144.69598	9	190.15523	9	235.60447	9	280.77371	9	326.13296	9	371.49220	9	416.85144	9	416.85144
20	9.07185	120	54.43109	220	99.79033	320	145.14958	420	190.50882	520	235.95806	620	281.22731	720	326.58655	820	371.94579	920	417.30503		
1	9.52544	1	54.88468	1	100.24393	1	145.60317	1	190.96241	1	236.41165	1	281.68090	1	327.04014	1	372.39938	1	417.75863	1	417.75863
2	9.97903	2	55.33828	2	100.69752	2	146.05676	2	191.41600	2	236.86525	2	282.13449	2	327.49373	2	372.85298	2	418.21222	2	418.21222
3	10.43262	3	55.79187	3	101.15111	3	146.51035	3	191.86960	3	237.31884	3	282.58808	3	327.94733	3	373.30657	3	418.66581	3	418.66581
4	10.88622	4	56.24546	4	101.60470	4	146.96395	4	192.32319	4	237.77243	4	283.04167	4	328.40092	4	373.76016	4	419.11940	4	419.11940
5	11.33981	5	56.69905	5	102.05830	5	147.41754	5	192.77678	5	238.22602	5	283.49527	5	328.85451	5	374.21375	5	419.57300	5	419.57300
6	11.79340	6	57.15265	6	102.51189	6	147.87113	6	193.23037	6	238.67962	6	283.94886	6	329.30810	6	374.66735	6	420.02659	6	420.02659
7	12.24700	7	57.60624	7	102.96548	7	148.32472	7	193.68397	7	239.13421	7	284.40245	7	329.76169	7	375.12094	7	420.48018	7	420.48018
8	12.70059	8	58.05983	8	103.41907	8	148.77832	8	194.13756	8	239.58780	8	284.85604	8	330.21529	8	375.57453	8	420.93377	8	420.93377
9	13.15418	9	58.51342	9	103.87267	9	149.23191	9	194.59115	9	239.95039	9	285.30964	9	330.66888	9	376.02812	9	421.38737	9	421.38737
30	13.60777	130	58.96702	230	104.32626	330	149.68550	430	195.04474	530	240.40399	630	285.76323	730	331.12247	830	376.48171	930	421.84096		
1	14.06137	1	59.42061	1	104.77985	1	150.13909	1	195.49834	1	240.85758	1	286.21682	1	331.57606	1	376.93531	1	422.29455	1	422.29455
2	14.51496	2	59.87420	2	105.23344	2	150.59269	2	195.95193	2	241.31117	2	286.67041	2	332.02966	2	377.38890	2	422.74814	2	422.74814
3	14.96855	3	60.32779	3	105.68704	3	151.04628	3	196.40552	3	241.76476	3	287.12401	3	332.48325	3	377.84249	3	423.20174	3	423.20174
4	15.42214	4	60.78139	4	106.14063	4	151.49987	4	196.85911	4	242.21836	4	287.57760	4	332.93684	4	378.29608	4	423.65533	4	423.65533
5	15.87573	5	61.23498	5	106.59422	5	151.95346	5	197.31271	5	242.67195	5	288.03119	5	333.39043	5	378.74968	5	424.10892	5	424.10892
6	16.32933	6	61.68857	6	107.04781	6	152.40700	6	197.76630	6	243.12554	6	288.48478	6	333.84403	6	379.20327	6	424.56251	6	424.56251
7	16.78292	7	62.14216	7	107.50141	7	152.86063	7	198.21989	7	243.57913	7	288.93838	7	334.29762	7	379.65686	7	425.01610	7	425.01610
8	17.23651	8	62.59576	8	107.95500	8	153.31424	8	198.67348	8	244.03273	8	289.39197	8	334.75121	8	380.11045	8	425.46970	8	425.46970
9	17.69010	9	63.04935	9	108.40859	9	153.76783	9	199.12708	9	244.48632	9	289.84556	9	335.20480	9	380.56405	9	425.92329	9	425.92329
40	18.14370	140	63.50294	240	108.86218	340	154.22143	440	199.58067	540	244.93991	640	290.29915	740	335.65840	840	381.01764	940	426.37688		
1	18.59729	1	63.95653	1	109.31578	1	154.67502	1	200.03426	1	245.39350	1	290.75275	1	336.11199	1	381.47123	1	426.83047	1	426.83047
2	19.05088	2	64.41012	2	109.76937	2	155.12861	2	200.48785	2	245.84709	2	291.20634	2	336.56558	2	381.92482	2	427.28407	2	427.28407
3	19.50447	3	64.86372	3	110.22296	3	155.58220	3	200.94145	3	246.30069	3	291.65993	3	337.01917	3	382.37842	3	427.73766	3	427.73766
4	19.95807	4	65.31731	4	110.67655	4	156.03580	4	201.39504	4	246.75428	4	292.11352	4	337.47277	4	382.83201	4	428.19125	4	428.19125
5	20.41166	5	65.77090	5	111.13014	5	156.48939	5	201.84863	5	247.20787	5	292.56712	5	337.92636	5	383.28560	5	428.64484	5	428.64484
6	20.86525	6	66.22449	6	111.58374	6	156.94298	6	202.30222	6	247.66146	6	293.02071	6	338.37995	6	383.73919	6	429.09844	6	429.09844
7	21.31884	7	66.67809	7	112.03733	7	157.39657	7	202.75582	7	248.11506	7	293.47430	7	338.83354	7	384.19279	7	429.55203	7	429.55203
8	21.77244	8	67.13168	8	112.49092	8	157.85016	8	203.20941	8	248.56865	8	293.92789	8	339.28714	8	384.64638	8	430.00562	8	430.00562
9	22.22603	9	67.58527	9	112.94451	9	158.30376	9	203.66300	9	249.02224	9	294.38149	9	339.74073	9	385.09997	9	430.45921	9	430.45921

Standards of Mass

50	22. 67962	150	68. 03886	250	113. 39811	350	158. 75735	450	204. 11659	550	249. 47584	650	294. 83508	750	340. 19432	850	385. 55356	950	430. 91281
1	23. 13321	1	68. 49246	1	113. 85170	1	159. 21094	1	204. 57018	1	249. 92943	1	295. 28867	1	340. 64791	1	386. 00716	1	431. 36640
2	23. 58661	2	68. 94605	2	114. 30529	2	159. 66453	2	205. 02378	2	250. 38302	2	295. 74226	2	341. 10151	2	386. 46075	2	431. 81999
3	24. 04040	3	69. 39964	3	114. 75888	3	160. 11813	3	205. 47737	3	250. 83661	3	296. 19586	3	341. 55510	3	386. 91434	3	432. 27358
4	24. 49399	4	69. 85323	4	115. 21248	4	160. 57172	4	205. 93096	4	251. 29020	4	296. 64945	4	342. 00869	4	387. 36793	4	432. 72718
5	24. 94758	5	70. 30683	5	115. 66607	5	161. 02531	5	206. 38455	5	251. 74380	5	297. 10304	5	342. 46228	5	387. 82153	5	433. 18077
6	25. 40118	6	70. 76042	6	116. 11966	6	161. 47890	6	206. 83815	6	252. 19739	6	297. 55663	6	342. 91588	6	388. 27512	6	433. 63436
7	25. 85477	7	71. 21401	7	116. 57325	7	161. 93250	7	207. 29174	7	252. 65098	7	298. 01022	7	343. 36947	7	388. 72871	7	434. 08795
8	26. 30836	8	71. 66760	8	117. 02685	8	162. 38609	8	207. 74553	8	253. 10457	8	298. 46382	8	343. 82306	8	389. 18230	8	434. 54155
9	26. 76195	9	72. 12120	9	117. 48044	9	162. 83968	9	208. 19892	9	253. 53817	9	298. 91741	9	344. 27665	9	389. 63590	9	434. 99514
60	27. 21555	160	72. 57479	260	117. 93403	360	163. 29327	460	208. 65252	560	254. 01176	660	299. 37100	760	344. 73025	860	390. 08949	960	435. 44873
1	27. 66914	1	73. 02838	1	118. 38762	1	163. 74687	1	209. 10611	1	254. 46535	1	299. 82459	1	345. 18384	1	390. 54308	1	435. 90232
2	28. 12273	2	73. 48197	2	118. 84122	2	164. 20446	2	209. 55970	2	254. 91894	2	300. 27819	2	345. 63743	2	390. 99667	2	436. 35592
3	28. 57632	3	73. 93557	3	119. 29481	3	164. 65405	3	210. 01329	3	255. 37254	3	300. 73178	3	346. 09102	3	391. 45027	3	436. 80951
4	29. 02992	4	74. 38916	4	119. 74840	4	165. 10764	4	210. 46689	4	255. 82613	4	301. 18537	4	346. 54461	4	391. 90386	4	437. 26310
5	29. 48351	5	74. 84275	5	120. 20199	5	165. 56124	5	210. 92048	5	256. 27972	5	301. 63896	5	346. 99821	5	392. 35745	5	437. 71669
6	29. 93710	6	75. 29634	6	120. 65559	6	166. 01483	6	211. 37407	6	256. 73331	6	302. 09256	6	347. 45180	6	392. 81104	6	438. 17029
7	30. 39069	7	75. 74994	7	121. 10918	7	166. 46882	7	211. 82766	7	257. 18691	7	302. 54615	7	347. 90539	7	393. 26463	7	438. 62388
8	30. 84429	8	76. 20353	8	121. 56277	8	166. 92201	8	212. 28126	8	257. 64050	8	302. 99974	8	348. 35898	8	393. 71823	8	439. 07747
9	31. 29788	9	76. 65712	9	122. 01636	9	167. 37561	9	212. 73485	9	258. 09409	9	303. 45333	9	348. 81258	9	394. 17182	9	439. 53106
70	31. 75147	170	77. 11071	270	122. 46996	370	167. 82920	470	213. 18884	570	258. 54768	670	303. 90693	770	349. 26617	870	394. 62541	970	439. 98465
1	32. 20506	1	77. 56431	1	122. 92355	1	168. 28279	1	213. 64203	1	259. 00128	1	304. 36052	1	349. 71976	1	395. 07900	1	440. 43825
2	32. 65865	2	78. 01790	2	123. 37714	2	168. 73638	2	214. 09563	2	259. 45487	2	304. 81411	2	350. 17335	2	395. 53260	2	440. 89184
3	33. 11225	3	78. 47149	3	123. 83073	3	169. 18998	3	214. 54922	3	259. 90846	3	305. 26770	3	350. 62695	3	395. 98619	3	441. 34543
4	33. 56584	4	78. 92509	4	124. 28433	4	169. 64357	4	215. 00281	4	260. 36205	4	305. 72130	4	351. 08054	4	396. 43978	4	441. 79902
5	34. 01943	5	79. 37867	5	124. 73792	5	170. 09716	5	215. 45640	5	260. 81565	5	306. 17489	5	351. 53415	5	396. 89337	5	442. 25262
6	34. 47302	6	79. 83227	6	125. 19151	6	170. 55075	6	215. 91000	6	261. 26924	6	306. 62848	6	351. 98772	6	397. 34697	6	442. 70621
7	34. 92662	7	80. 28586	7	125. 64510	7	171. 00435	7	216. 36359	7	261. 72283	7	307. 08207	7	352. 44132	7	397. 80056	7	443. 15980
8	35. 38021	8	80. 73945	8	126. 09869	8	171. 45794	8	216. 81718	8	262. 17642	8	307. 53567	8	352. 89491	8	398. 25415	8	443. 61339
9	35. 83380	9	81. 19304	9	126. 55229	9	171. 91153	9	217. 27077	9	262. 63002	9	307. 98926	9	353. 34850	9	398. 70774	9	444. 06599
80	36. 28739	180	81. 64664	280	127. 00588	380	172. 36512	480	217. 72437	580	263. 08361	680	308. 44285	780	353. 80209	880	399. 16134	980	444. 52058
1	36. 74099	1	82. 10023	1	127. 45947	1	172. 81871	1	218. 17796	1	263. 53720	1	308. 89644	1	354. 25569	1	399. 61493	1	444. 97417
2	37. 19458	2	82. 55382	2	127. 91306	2	173. 27231	2	218. 63155	2	263. 99079	2	309. 35004	2	354. 70928	2	400. 06852	2	445. 42776
3	37. 64817	3	83. 00741	3	128. 36666	3	173. 72590	3	219. 08514	3	264. 44439	3	309. 80363	3	355. 16287	3	400. 52211	3	445. 88136
4	38. 10176	4	83. 46101	4	128. 82025	4	174. 17949	4	219. 53874	4	264. 89798	4	310. 25722	4	355. 61646	4	400. 97571	4	446. 33495
5	38. 55536	5	83. 91460	5	129. 27384	5	174. 63308	5	219. 99233	5	265. 35157	5	310. 71081	5	356. 07006	5	401. 42930	5	446. 78854
6	39. 00895	6	84. 36819	6	129. 72743	6	175. 08668	6	220. 44592	6	265. 80516	6	311. 16441	6	356. 52365	6	401. 88289	6	447. 24213
7	39. 46254	7	84. 82178	7	130. 18103	7	175. 54027	7	220. 89951	7	266. 25876	7	311. 61800	7	356. 97724	7	402. 33648	7	447. 69573
8	39. 91613	8	85. 27538	8	130. 63462	8	175. 99386	8	221. 35310	8	266. 71235	8	312. 07159	8	357. 43083	8	402. 79008	8	448. 14932
9	40. 36973	9	85. 72897	9	131. 08821	9	176. 44745	9	221. 80670	9	267. 16594	9	312. 52518	9	357. 88443	9	403. 24367	9	448. 60291
90	40. 82332	190	86. 18256	290	131. 54180	390	176. 90105	490	222. 26029	590	267. 61953	690	312. 97878	790	358. 33802	890	403. 69726	990	449. 05650
1	41. 27691	1	86. 63615	1	131. 99540	1	177. 35464	1	222. 71388	1	268. 07312	1	313. 43237	1	358. 79161	1	404. 15085	1	449. 51010
2	41. 73050	2	87. 08975	2	132. 44809	2	177. 80823	2	223. 16747	2	268. 52672	2	313. 88506	2	359. 24520	2	404. 60945	2	449. 96369
3	42. 18410	3	87. 54334	3	132. 90258	3	178. 26182	3	223. 62107	3	268. 98051	3	314. 33955	3	359. 69880	3	405. 05804	3	450. 41728
4	42. 63769	4	87. 99693	4	133. 35617	4	178. 71572	4	224. 07466	4	269. 43390	4	314. 79314	4	360. 15239	4	405. 51163	4	450. 87087
5	43. 09128	5	88. 45052	5	133. 80977	5	179. 16901	5	224. 52825	5	269. 88749	5	315. 24674	5	360. 60598	5	405. 96522	5	451. 32447
6	43. 54487	6	88. 90412	6	134. 26336	6	179. 62260	6	224. 98184	6	270. 34109	6	315. 70033	6	361. 05957	6	406. 41882	6	451. 77806
7	43. 99847	7	89. 35771	7	134. 71695	7	180. 07619	7	225. 43544	7	270. 79468	7	316. 15392	7	361. 51316	7	406. 87241	7	452. 23165
8	44. 45206	8	89. 81130	8	135. 17054	8	180. 52979	8	225. 88903	8	271. 24827	8	316. 60751	8	361. 96676	8	407. 32600	8	452. 68524
9	44. 90565	9	90. 26189	9	135. 62414	9	180. 98338	9	226. 34262	9	271. 70186	9	317. 06111	9	362. 42035	9	407. 77959	9	453. 13884

Standards of Mass

80	110.2311 1 112.4357 2 114.6404 3 116.8450 4 119.0496 5 121.2542 6 123.4589 7 125.6635 8 127.8681 9 130.0727	150	330.6934 1 332.8980 2 335.1026 3 337.3072 4 339.5118 5 341.7165 6 343.9211 7 346.1257 8 348.3303 9 350.5350	250	551.1556 1 553.3602 2 555.5648 3 557.7695 4 559.9741 5 562.1787 6 564.3833 7 566.5879 8 568.7926 9 570.9972	350	771.6178 1 773.8224 2 776.0271 3 778.2317 4 780.4363 5 782.6409 6 784.8456 7 787.0502 8 789.2548 9 791.4594	450	992.0801 1 994.2847 2 996.4893 3 998.6939 4 1,000.8985 5 1,003.1032 6 1,005.3078 7 1,007.5124 8 1,009.7170 9 1,011.9217	550	1,212.5423 1 1,214.7469 2 1,216.9515 3 1,219.1562 4 1,221.3608 5 1,223.5654 6 1,225.7700 7 1,227.9746 8 1,230.1793 9 1,232.3839	650	1,433.0045 1 1,435.2091 2 1,437.4138 3 1,439.6184 4 1,441.8230 5 1,444.0276 6 1,446.2323 7 1,448.4369 8 1,450.6415 9 1,452.8461	750	1,653.4668 1 1,655.6714 2 1,657.8760 3 1,660.0806 4 1,662.2852 5 1,664.4899 6 1,666.6945 7 1,668.8991 8 1,671.1037 9 1,673.3084	850	1,873.2290 1 1,876.1336 2 1,879.0382 3 1,881.9429 4 1,884.8475 5 1,887.7521 6 1,890.6567 7 1,893.5613 8 1,896.4659 9 1,899.3706	950	2,094.3912 1 2,096.5958 2 2,098.8005 3 2,101.0051 4 2,103.2097 5 2,105.4143 6 2,107.6190 7 2,109.8236 8 2,112.0282 9 2,114.2328
60	132.2773 1 134.4820 2 136.6866 3 138.8912 4 141.0958 5 143.3005 6 145.5051 7 147.7097 8 149.9143 9 152.1189	160	352.7396 1 354.9442 2 357.1488 3 359.3534 4 361.5581 5 363.7627 6 365.9673 7 368.1719 8 370.3766 9 372.5812	260	573.2018 1 575.4064 2 577.6111 3 579.8157 4 582.0203 5 584.2249 6 586.4295 7 588.6342 8 590.8388 9 593.0434	360	793.6640 1 795.8687 2 798.0733 3 800.2779 4 802.4825 5 804.6872 6 806.8918 7 809.0964 8 811.3010 9 813.5056	460	1,014.1263 1 1,016.3309 2 1,018.5355 3 1,020.7401 4 1,022.9448 5 1,025.1494 6 1,027.3540 7 1,029.5586 8 1,031.7633 9 1,033.9679	560	1,234.5885 1 1,236.7931 2 1,238.9978 3 1,241.2024 4 1,243.4070 5 1,245.6116 6 1,247.8162 7 1,250.0209 8 1,252.2255 9 1,254.4301	660	1,455.0507 1 1,457.2554 2 1,459.4600 3 1,461.6646 4 1,463.8692 5 1,466.0739 6 1,468.2785 7 1,470.4831 8 1,472.6877 9 1,474.8923	760	1,675.5130 1 1,677.7176 2 1,679.9222 3 1,682.1268 4 1,684.3315 5 1,686.5361 6 1,688.7407 7 1,690.9453 8 1,693.1500 9 1,695.3546	860	1,895.9752 1 1,898.1798 2 1,900.3845 3 1,902.5891 4 1,904.7937 5 1,906.9983 6 1,909.2029 7 1,911.4076 8 1,913.6122 9 1,915.8168	960	2,116.4374 1 2,118.6421 2 2,120.8467 3 2,123.0513 4 2,125.2559 5 2,127.4606 6 2,129.6652 7 2,131.8698 8 2,134.0744 9 2,136.2790
70	154.3236 1 156.5282 2 158.7328 3 160.9374 4 163.1421 5 165.3467 6 167.5513 7 169.7559 8 171.9605 9 174.1652	170	374.7858 1 376.9904 2 379.1950 3 381.3997 4 383.6043 5 385.8089 6 388.0135 7 390.2182 8 392.4228 9 394.6274	270	595.2480 1 597.4527 2 599.6573 3 601.8619 4 604.0665 5 606.2711 6 608.4758 7 610.6804 8 612.8850 9 615.0896	370	815.7103 1 817.9149 2 820.1195 3 822.3241 4 824.5288 5 826.7334 6 828.9380 7 831.1426 8 833.3472 9 835.5519	470	1,036.1725 1 1,038.3771 2 1,040.5817 3 1,042.7864 4 1,044.9910 5 1,047.1956 6 1,049.4002 7 1,051.6049 8 1,053.8095 9 1,056.0141	570	1,256.6347 1 1,258.8394 2 1,261.0440 3 1,263.2486 4 1,265.4532 5 1,267.6578 6 1,269.8625 7 1,272.0671 8 1,274.2717 9 1,276.4763	670	1,477.0970 1 1,479.3016 2 1,481.5062 3 1,483.7108 4 1,485.9155 5 1,488.1201 6 1,490.3247 7 1,492.5293 8 1,494.7339 9 1,496.9386	770	1,697.5592 1 1,699.7638 2 1,701.9684 3 1,704.1731 4 1,706.3777 5 1,708.5823 6 1,710.7869 7 1,712.9916 8 1,715.1962 9 1,717.4008	870	1,918.0214 1 1,920.2261 2 1,922.4307 3 1,924.6353 4 1,926.8399 5 1,929.0445 6 1,931.2492 7 1,933.4538 8 1,935.6584 9 1,937.8630	970	2,138.4837 1 2,140.6883 2 2,142.8929 3 2,145.0975 4 2,147.3022 5 2,149.5068 6 2,151.7114 7 2,153.9160 8 2,156.1206 9 2,158.3253
80	176.3698 1 178.5744 2 180.7790 3 182.9837 4 185.1883 5 187.3929 6 189.5975 7 191.8021 8 194.0068 9 196.2114	180	396.8320 1 399.0366 2 401.2413 3 403.4459 4 405.6505 5 407.8551 6 410.0598 7 412.2644 8 414.4690 9 416.6736	280	617.2943 1 619.4989 2 621.7035 3 623.9081 4 626.1127 5 628.3174 6 630.5220 7 632.7266 8 634.9312 9 637.1359	380	837.7565 1 839.9611 2 842.1657 3 844.3704 4 846.5750 5 848.7796 6 850.9842 7 853.1888 8 855.3935 9 857.5981	480	1,058.2187 1 1,060.4233 2 1,062.6280 3 1,064.8326 4 1,067.0372 5 1,069.2418 6 1,071.4465 7 1,073.6511 8 1,075.8557 9 1,078.0603	580	1,278.6810 1 1,280.8856 2 1,283.0902 3 1,285.2948 4 1,287.4994 5 1,289.7041 6 1,291.9087 7 1,294.1133 8 1,296.3179 9 1,298.5226	680	1,499.1432 1 1,501.3478 2 1,503.5524 3 1,505.7571 4 1,507.9617 5 1,510.1663 6 1,512.3709 7 1,514.5755 8 1,516.7802 9 1,518.9848	780	1,719.6034 1 1,721.8100 2 1,724.0147 3 1,726.2193 4 1,728.4239 5 1,730.6285 6 1,732.8332 7 1,735.0378 8 1,737.2424 9 1,739.4470	880	1,940.0677 1 1,942.2723 2 1,944.4769 3 1,946.6815 4 1,948.8861 5 1,951.0908 6 1,953.2954 7 1,955.5000 8 1,957.7046 9 1,959.9093	980	2,160.5299 1 2,162.7345 2 2,164.9391 3 2,167.1438 4 2,169.3484 5 2,171.5530 6 2,173.7576 7 2,175.9623 8 2,178.1669 9 2,180.3715
90	198.4160 1 200.6206 2 202.8253 3 205.0299 4 207.2345 5 209.4391 6 211.6437 7 213.8484 8 216.0530 9 218.2576	190	418.8782 1 421.0829 2 423.2875 3 425.4921 4 427.6967 5 429.9014 6 432.1060 7 434.3106 8 436.5152 9 438.7198	290	639.3405 1 641.5451 2 643.7497 3 645.9543 4 648.1590 5 650.3636 6 652.5682 7 654.7728 8 656.9775 9 659.1821	390	859.8027 1 862.0073 2 864.2120 3 866.4166 4 868.6212 5 870.8258 6 873.0304 7 875.2351 8 877.4397 9 879.6443	490	1,082.2649 1 1,084.4696 2 1,086.6742 3 1,088.8788 4 1,089.0834 5 1,091.2881 6 1,093.4927 7 1,095.6973 8 1,097.9019 9 1,100.1065	590	1,300.7272 1 1,302.9318 2 1,305.1364 3 1,307.3410 4 1,309.5457 5 1,311.7503 6 1,313.9549 7 1,316.1595 8 1,318.3642 9 1,320.5688	690	1,521.1894 1 1,523.3940 2 1,525.5987 3 1,527.8033 4 1,530.0079 5 1,532.2125 6 1,534.4171 7 1,536.6218 8 1,538.8264 9 1,541.0310	790	1,741.6516 1 1,743.8563 2 1,746.0609 3 1,748.2655 4 1,750.4701 5 1,752.6748 6 1,754.8794 7 1,757.0840 8 1,759.2886 9 1,761.4933	890	1,962.1139 1 1,964.3185 2 1,966.5231 3 1,968.7278 4 1,970.9324 5 1,973.1370 6 1,975.3416 7 1,977.5462 8 1,979.7509 9 1,981.9555	990	2,182.5761 1 2,184.7807 2 2,186.9854 3 2,189.1900 4 2,191.3946 5 2,193.5992 6 2,195.8039 7 2,198.0085 8 2,200.2131 9 2,202.4177

UNITED STATES DEPARTMENT OF COMMERCE • Sinclair Weeks, *Secretary*
NATIONAL BUREAU OF STANDARDS • A. V. Astin, *Director*

Testing of Hydrometers

J. C. Hughes



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Testing of Hydrometers

J. C. Hughes

The design and construction of hydrometers, with particular reference to hydrometers that are to be submitted to the National Bureau of Standards for test are discussed. The various scales that are commonly used for hydrometers are defined and recommendations given for subdividing and marking them. These scales include density, specific gravity, degrees Baumé, degrees API, percentage by weight, percentage by volume, percentage proof, spirit, Brix, Balling, and some others. The relations between some of the arbitrary scales (for example, API) and specific gravity are stated.

The Circular outlines the procedure of testing hydrometers that are submitted to the Bureau and discusses the forms of certificates and reports issued as a result of these tests. Instructions and other helpful information about submitting hydrometers for test are given.

1. Introduction

1.1. Purpose of Circular

The object of this Circular is twofold: (1) to present to manufacturers and users information regarding the design, construction, and calibration of hydrometers in an effort to secure uniformity in these operations; and (2) to specify the requirements for hydrometers submitted to the Bureau for certification.

1.2. Classes of Hydrometers

There are two general types of hydrometers, namely, hydrometers proper and hydrometers combined with a thermometer, called thermo-hydrometers.

With reference to the indication or scale, hydrometers may be classified as follows:

(a) Density hydrometers, indicating density of a specified liquid, at a specified temperature, in specified units; for example, in grams per milliliter.

(b) Specific gravity hydrometers, indicating the specific gravity of a liquid, at a specified temperature, in terms of the density of water at a specified temperature as unity.

(c) Percentage hydrometers, indicating, at a specified temperature, the percentage of a substance dissolved in water; for example, the percentage of salts in a sample of sea water, or the percentage of sugar in an aqueous solution.

(d) Arbitrary scale hydrometers, indicating concentration or strength of a specified liquid, or its density, referred to an arbitrarily defined scale at a specified temperature (Baumé hydrometers and API hydrometers).

2. Recommendations on Construction

2.1. Materials and Form

Hydrometers should be made of smooth, transparent glass, free of bubbles, striae, or other imperfections. The glass should be of a kind that will adequately resist the action of chemical reagents in which it is normally used and also have suitable thermal qualities to permit its use over the range of temperatures to which it may be subjected. It should be thoroughly annealed before final adjustment and sealing.

The total length of any hydrometer should not exceed 45 cm, and, for convenience in use, a length of 35 cm or less is desirable.

All sections perpendicular to the axis should be circular, such that the outer surface is symmetrical about the vertical axis. There should be no uneven or unnecessary thickening of the walls and no abrupt changes nor constrictions that would hinder thorough cleaning.

The stem should be uniform in cross section with no perceptible irregularities. The top of the stem should be neatly rounded without unnecessary

thickening. It should extend above the top graduation line at least 1.5 cm and should continue cylindrical below the bottom graduation line for at least 3 mm.

Material used for ballast should be confined to the bottom of the instrument, and no loose material of any sort should be inside a hydrometer. The disposition of the weight should be such that the hydrometer will always float with its axis vertical. Bulbs containing shot may be flattened, but in all cases the shot must be confined in the bottom either by a glass partition or a cement that does not soften at temperatures to which it may normally be subjected.

Only the best quality of material should be used for scales and designating labels. Paper usually known as first-class ledger paper is suitable for this purpose. The scales and labels should be securely fastened in place by some material that does not soften at the highest temperature to which the instrument will be exposed in use, and which does not deteriorate with time. When reference marks are used on stem and scale to provide a means of

detecting slippage of the scale, they must coincide exactly. If a line other than the top numbered line is used as a reference, it must be clearly identified as such. The scale should be straight and without twist.

The graduation lines must be perpendicular to the axis of the hydrometer, that is, horizontal when the instrument is floating. They should extend at least one-fourth around the circumference of the stem, the minimum length being 2 mm, and join or intersect a line parallel to the axis indicating the front of the scale.

The lengths of division and subdivision lines should be so chosen as to facilitate readings. Sufficient lines should be numbered to clearly indicate the reading at any point. The numbers at the ends of the scale intervals should be complete, but those intermediate may be abbreviated.

The graduation lines and inscriptions should be clear and distinct in permanent ink, such as india ink.

The distance between any two adjacent subdivision lines of the scale should never be less than 0.8 mm nor more than 3.0 mm, and a distance of 1.5 to 2.0 mm is recommended.

If flat scales are used, the length and spacing of the graduation lines should conform to recommendations given above.

To facilitate readings near the end of the hydrometer scale, the graduations should be continued a few divisions beyond the ends of the principal interval.

The scale of a hydrometer for density indications should be divided into 0.001, 0.0005, 0.0002, or 0.0001 subdivisions. Similar values of the scale intervals may be used in specific gravity hydrometers. For percentage or degree indications, the hydrometer scale should be divided into whole, half, fifth, or tenth percents or degrees (never into fourths).

No hydrometer should have more than one scale, and there should be no secondary or auxiliary graduations.

In response to the demand from some users, a few hydrometers have been made with two scales, of the same units and range, and spaced about 180 deg apart in the stem. Although the use of such duplex scales does not conform to the recommendation of a single scale, such hydrometers will be calibrated, provided:

(a) That on one scale, and preferably both, the graduation lines extend one-fourth of the way around the circumference of the stem; and

(b) That where the numbered scale lines on one scale meet or overlap those of the second scale, there is no vertical displacement.

Furthermore, even when these provisions are fulfilled, hydrometers with duplex scales will receive reports only, since it is impracticable to check, adequately, the uniformity of the subdivisions of the two scales.

With thermohydrometers, the thermometer scale may be contained in either the body or stem of the hydrometer. In either case, the capillary stem of the thermometer should be parallel to the hydrometer axis. Other desirable features of the thermometer element are: the stem should contain an expansion chamber that will permit heating to 120° C; the space above the mercury should be either evacuated or filled with an inert gas; the thermometer scale should be divided into whole or half degrees; the thermometer scale should include the ice point (0° C); when within the stem of the hydrometer, the thermometer scale should be distinguished from the hydrometer scale by the use of a differently colored ink.

There should be no apparent irregularities in either the hydrometer scale or the thermometer scale.

The hydrometer should be thoroughly dry on the inside when sealed.

2.2. Inscriptions

The hydrometer scale or a suitable special label should bear an inscription that indicates unequivocally the purpose of the instrument. This inscription should denote: (a) the liquid for which the instrument is intended, if necessary; (b) the temperature at which it is to be used; and (c) the character of the indication, including definition of any arbitrary scale employed.

The designation of standard temperature and reference temperature may be abbreviated, as, for example, specific gravity 15°/15° C, meaning that the hydrometer indicates at 15° C the specific gravity of the liquid referred to water at 15° C as unity.

Hydrometers submitted to the Bureau for testing must show upon the scale or label, the maker's name or trade mark and an individual identifying number. This is important because it will be the only means by which the instrument may be associated with its certificate or report. Therefore, manufacturers must be particularly careful not to duplicate their identification numbers, although these numbers may be associated with the year.

3. Definition and Graduation of Various Scales

The tables used in the standardization of hydrometers by the Bureau are contained in NBS Circular 19 (6th edition), Standard density and volumetric tables, and are referred to below by the numbers used in that Circular.

3.1. Alcoholometers

Alcoholometers may be graduated to indicate the percentage of ethyl alcohol, either by weight or by volume, in mixtures of ethyl alcohol and water, or they may be graduated to indicate percentages

of "proof spirit" as defined by the Bureau of Internal Revenue, United States Treasury Department.¹

3.2. Saccharometers

Saccharometers should be graduated to indicate percentage of sugar by weight² or degrees Baumé at 20° C.

The basis for graduation of saccharometers, standard at 20° C, should be density at 20° C, as given in table 12 of NBS Circular 19.

Balling saccharometers should be graduated to indicate the percentage of sugar by weight at 60° F, and Brix saccharometers the percentage of sugar by weight at the temperature indicated.

Degrees Baumé for sugar is defined by the formula³

$$\text{degrees Baumé at } 20^{\circ}\text{C} = 145 - \frac{145}{\text{sp gr } 20^{\circ}/20^{\circ}\text{C}}$$

3.3. Hydrometers for Sulfuric Acid

The basis for the graduation of hydrometers indicating percentage of sulfuric acid by weight in mixtures of acid with water should be density at 20° C as given in table 13 of Circular 19.

3.4. Baumé Hydrometers

The basis for hydrometers indicating degrees Baumé of liquids lighter than water should be the relation to specific gravity at 60°/60° F = 15.56°/15.56° C given by the formula

$$\text{degrees Baumé} = \frac{140}{\text{sp gr } 60^{\circ}/60^{\circ}\text{F}} - 130,$$

as given in tables 22 and 23 of Circular 19.

For hydrometers indicating degrees Baumé of liquids heavier than water, the basis should be the relation to specific gravity at 60°/60° F = 15.56°/15.56° C given by the formula

$$\text{degrees Baumé} = 145 - \frac{145}{\text{sp gr } 60^{\circ}/60^{\circ}\text{F}}$$

as given in tables 20 and 21 of Circular 19.

The liquids for standardization of Baumé hydrometers should be, in general, mineral oils for liquids lighter than water, mixtures of sulfuric

acid and water for heavier liquids, the Thoulet solution for liquids heavier than sulfuric acid.

3.5. Hydrometers for Petroleum Oil

The hydrometers in general use in the petroleum oil industry in the United States are based on the modulus 141.5, which is the basis for the API scale (American Petroleum Institute Scale).

The formula for converting specific gravity to API is as follows:

$$\text{degrees API} = \frac{141.5}{\text{sp gr } 60^{\circ}/60^{\circ}\text{F}} - 131.5,$$

as given in tables 24 and 25 of Circular 19.⁴

3.6. Density Hydrometers

Hydrometers indicating density should be graduated to indicate, at the temperature marked on the scale, the density of liquids in grams per milliliter.

3.7. Specific Gravity Hydrometers

The scales of hydrometers for indicating specific gravity should be graduated to indicate the ratio of the density of the liquid at a specified temperature (usually 60° F or 20° C) to the density of water at a specified temperature (4° C, 60° F, or 20° C) as unity. Normally these scales will apply to a particular liquid and specific gravity range. The following are some of the common liquids and solutions and the approximate range of their specific gravities.

Liquids	Range
Mineral oil.....	0.62 to 1.00
Ammonia.....	.85 to 1.00
Common salt.....	1.00 to 1.23
Hydrochloric acid.....	1.00 to 1.25
Caustic soda.....	1.00 to 1.55
Nitric acid.....	1.00 to 1.55
Sulfuric acid.....	1.00 to 1.85
Thoulet solution (K ₂ HgI ₄).....	1.84 to 3.00

4. Manipulation of Hydrometers

Hydrometers are seldom used for the greatest accuracy, as the usual conditions under which they are used preclude such special manipulation and exact observation as are necessary to obtain high precision. It is, nevertheless, desirable that

they be accurately graduated to avoid, as far as possible, the use of instrumental corrections. To obtain this result, it is helpful to employ certain precautions and methods in standardizing these instruments.

The methods of manipulation described below are, in general, the ones employed at the Bureau in testing hydrometers, and it may be helpful for a maker or user to follow them to such extent as his needs for accuracy require.

¹ "Proof spirit" is that alcoholic liquor that contains one-half of its volume of pure ethyl alcohol of a specific gravity of 0.7939 at 60° F, referred to water at 60° F as unity. "Gauging Manual," p. 7, 1938, U. S. Treasury Dept., Bureau of Internal Revenue. (The percentage of proof spirit is in every case twice the percentage of ethyl alcohol by volume at 60° F.)

² Both of the terms, "Brix" and "Balling," are interpreted as meaning the percentage by weight of pure sucrose. The relation between percentage of sugar and density used by the Bureau is that determined by F. Plato, (Wiss. Abh. Kaiserlichen Normal-Eichungs-Kommission 2, 153, 1900.)

³ See NBS Tech. Pap. T115, New Baumé scale for sugar solutions; and NBS Circular 440, Polarimetry, saccharimetry, and the sugars.

⁴ More complete petroleum oil tables may be found in the ASTM-IP Petroleum Measurement Tables, published by the American Society for Testing Materials, 1916 Race Street, Philadelphia 3, Pa.

4.1. Observation

The hydrometer should be clean, dry, and at the temperature of the liquid before immersing to make a reading.

The liquid in which the observation is made should be contained in a clear, smooth glass vessel of suitable size and shape.

The liquid should be thoroughly mixed by means of a stirrer reaching to the bottom of the vessel. In the hydrometer comparator, (fig. 1), this is accomplished by the propeller, E, in figure 2.

The hydrometer should be slowly immersed in the liquid slightly beyond the point where it floats naturally and then allowed to float freely.

The scale reading should not be made until the liquid and hydrometer are free from bubbles and at rest.

In reading the hydrometer scale the eye is placed slightly below the plane of the surface of

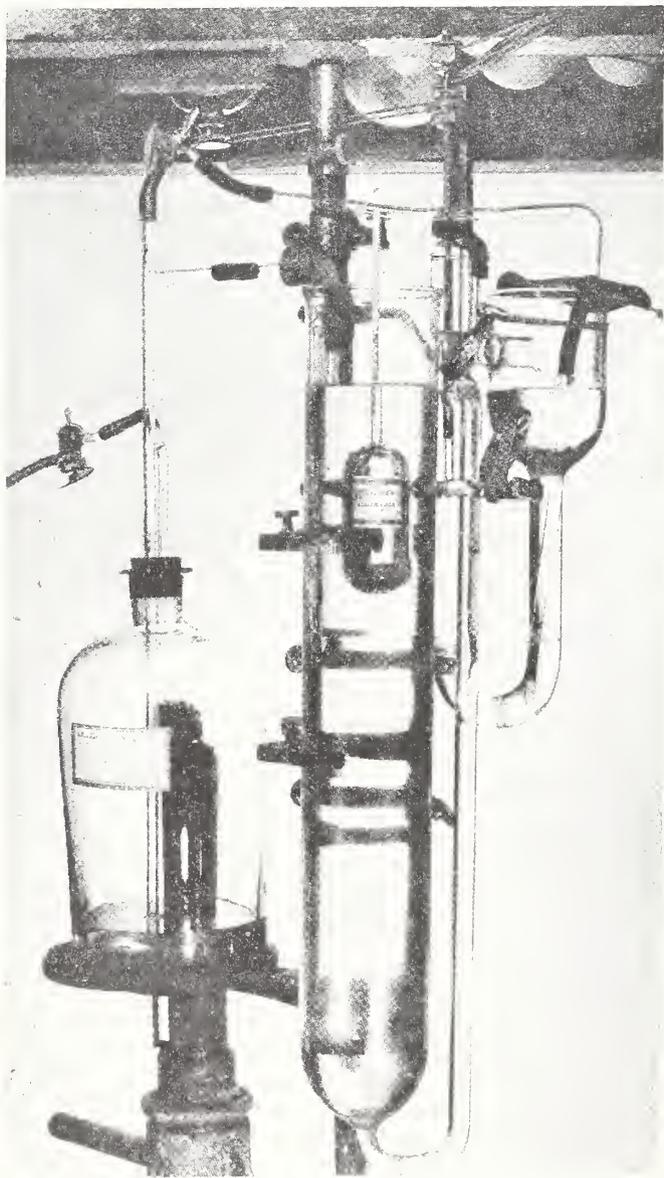


FIGURE 1. Hydrometer comparator.

the test liquid; it is raised slowly until the surface, seen as an ellipse, becomes a straight line. The point where this line intersects the hydrometer scale should be taken as the reading of the hydrometer.

4.2. Influence of Temperature

In order that a hydrometer may indicate correctly the density or strength of a specified liquid it is essential that the liquid be uniform throughout and at the temperature specified on the instrument. In comparing two hydrometers having the same standard temperature and made of the same type of glass, however, the temperature of the liquid need not be considered, since the correction required due to variation from the standard temperature is the same for both instruments.

To insure uniformity in the liquid, thorough mixing is required shortly before making the observation. This may be accomplished by a per-

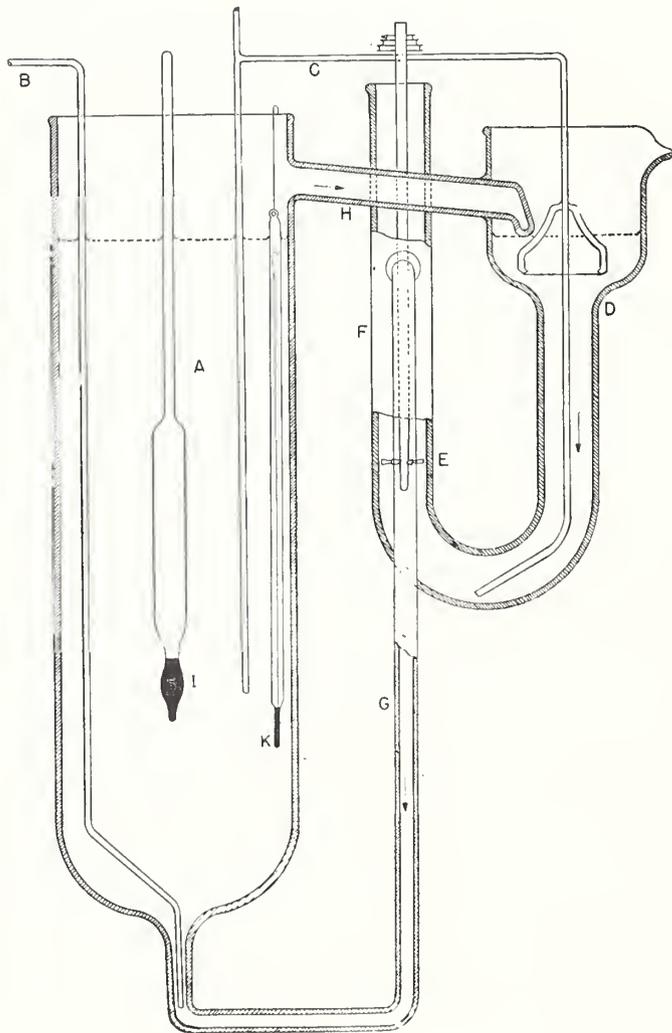


FIGURE 2. Section of hydrometer comparator.

A, Cylinder containing the test liquid; B, glass tube for filling and emptying A; C, siphon for removing the liquid from D into A; E, propeller that stirs the liquid and raises it in the tube, F, making it flow through G into A and through the cross tube, H, into D; I, hydrometer; K, thermometer.

forated disk or spiral at the end of a rod long enough to reach the bottom of the vessel. Motion of this stirrer from top to bottom serves to disperse layers of the liquid of different density. Or, if using a hydrometer comparator, the stirring can be accomplished by means of the propeller, E, in figure 2.

The temperatures of the liquid and of the surrounding atmosphere should be nearly equal during the observation; otherwise the temperature of the liquid will be changing, causing not only differences in density but also doubt as to the actual temperature. If an observation is made at some other temperature than that for which the hydrometer is designed, the reading will be in error. The magnitude of the error will depend upon the thermal expansion of the hydrometer and, in some cases, of the liquid used.

If the latter properties are known, tables of corrections for temperature may be prepared for use with hydrometers at various temperatures. Such tables should be used with caution, and only for approximate results when the temperature differs much from the standard temperature or from the temperature of the surrounding air. (See table 28, NBS Circular C19.)

Publications in which temperature correction tables may be found are listed below.

Liquid	Publication
Ethyl alcohol-----	NBS Circular 19. Gauging Manual, 1938, U. S. Treasury Department, Bureau of Internal Revenue.
Petroleum oils-----	
Sulfuric acid solutions.	NBS Circular 19. ASTM-IP Petroleum Measurement Tables.
Sugar solutions-----	
Vegetable tanning extracts-----	NBS Circular 19 and 440.
Turpentine-----	NBS Circular 449.
	NBS Technical Paper 9.

4.3. Influence of Surface Tension

When a hydrometer is floated in a liquid, a small quantity of the liquid rises about the stem to form a meniscus. This liquid adhering to the stem, above the general level of the liquid in which the instrument is floating, has the same effect as adding to the mass of the hydrometer, thus increasing the depth of immersion.

Because a hydrometer will indicate differently in two liquids having the same density but different surface tensions, and since surface tension is a specific property of liquids, it is necessary to specify the liquid for which a hydrometer is intended.

Although hydrometers of equivalent dimensions may be compared, without error, in a liquid differ-

ing in surface tension from the specified liquid, the results of comparisons of dissimilar instruments in such a liquid must be corrected for the effect of the surface tension.

In many liquids spontaneous changes in surface tension occur due to the formation of surface films of impurities, which may come from the apparatus, the liquid, or the air.

Errors from this cause may be avoided by the use of liquids not subject to such changes; however, if the liquid used is different in surface tension from the specified liquid, a correction is required when dissimilar instruments are compared, as mentioned above. A second method of avoiding these errors is to purify the surface of the test liquid before making an observation, by causing an overflow of the liquid. This method is employed at the Bureau for testing hydrometers in sulfuric acid, sodium chloride, and alcohol solutions, and is accomplished by causing the liquid to overflow from the part of the apparatus in which the hydrometer is immersed by a small rapidly rotating propeller, which serves also to stir the liquid.

The necessity for such special manipulation is confined to the reading of hydrometers in liquids that are subject to surface contamination. Such, in general, are aqueous solutions or mixtures of acids, alkalies, salts, sugar, and weak alcoholic mixtures. Oils, alcoholic mixtures of strength above 40 percent by volume, and other liquids of relatively low surface tension are not, in general, liable to surface contamination sufficient to cause appreciable changes in hydrometer readings.

4.4. Cleanliness

The accuracy of hydrometer observations depends, in many cases, upon the cleanliness of the instruments and of the liquids in which the observations are made.

In order that readings shall be uniform and reproducible, the surface of the hydrometers, and especially of the stem, must be clean, so that the liquid will rise uniformly and merge into an imperceptible film on the stem.

The readiness with which this condition is fulfilled depends somewhat upon the character of the liquid. Certain liquids, such as mineral oils and strong alcoholic mixtures, adhere to the stem very readily. On the other hand, with weak aqueous solutions of sugar, salts, acids, and alcohol, scrupulous cleaning of the stem is required in order to secure the normal condition.

Before being tested, hydrometers are thoroughly cleaned by dipping in a mixture of one part concentrated sulfuric acid and two parts fuming sulfuric acid, rinsed with water, and dried by wiping with a clean, lint-free cloth.

If they are to be used in aqueous solutions that do not adhere readily to the glass, the stems are wiped with alcohol and dried immediately before each reading.

5. Tolerances

5.1. General

The maximum error of the hydrometer scale should not exceed the following:

Smallest subdivision of hydrometer scale	Tolerance
Fifth, tenth, or twentieth percents or degrees or any density subdivision.	One smallest division.
Whole or half percents of proof spirit.	One smallest division.
Whole or half percents or degrees (other than above).	One-half smallest division.

The maximum error of thermometer scales in thermohydrometers should not exceed the following:

Smallest division of thermometer scale	Tolerance
2.0° F	1.0° F
1.0° C	0.5° C
0.5° C	0.3° C

5.2. Special

When the smallest subdivision of the hydrometer scale is less than 0.0002 specific gravity or its equivalent in other units, the following tolerances shall apply:

Scale	Tolerance (scale divisions)
Specific gravity in 0.0001.....	2.0
Percentage of alcohol by volume in 0.1:	
0.0 to 9.9.....	1.5
10.0 to 34.9.....	2.0
35.0 to 44.9.....	1.5
45.0 to 100.0.....	1.0
Percentage of proof spirit in 0.2:	
0.0 to 19.8.....	1.5
20.0 to 69.8.....	2.0
70.0 to 89.8.....	1.5
90.0 to 200.0.....	1.0

Scale—Continued	Tolerance (scale divisions)—Continued
Percentage of proof spirit in 0.1:	
0.0 to 19.9.....	3.0
20.0 to 69.9.....	4.0
70.0 to 89.9.....	3.0
90.0 to 149.9.....	2.0
150.0 to 189.9.....	1.5
190.0 to 200.0.....	1.0
Degrees API in 0.05:	
10.00 to 79.95.....	1.0
80.00 to 100.00.....	.. 5
Baumé for heavy liquids in 0.02:	
0.00 to 19.98.....	1.5
20.00 to 70.00.....	1.0
Baumé for heavy liquids in 0.01:	
0.00 to 9.99.....	3.0
10.00 to 19.99.....	2.5
20.00 to 39.99.....	2.0
40.00 to 59.99.....	1.5
60.00 to 70.00.....	1.0
Quevenne scale for milk in 0.1.....	2.0
N. Y. State Board of Health lactometer in 0.5.....	1.5
N. Y. State Board of Health lactometer in 0.2.....	3.0

Other scales not specifically mentioned here will be governed by the same principle; i. e., when the general requirements above would result in a tolerance of less than the equivalent of 0.0002 specific gravity, the limit of error shall be 0.0002 specific gravity expressed to the nearest one-half scale division.

5.3. Accuracy of Corrections Reported

Corrections will be expressed to one-tenth of a scale division, as has been the practice heretofore. The accuracy of these corrections will vary with the instrument design and no general statement of the accuracy can be made. However, even in those cases where the inaccuracies exceed one-tenth of a scale division it is preferable to give the corrections as found, since the value actually observed is the best estimate that can be obtained from a given test, and any rounding introduces an additional error. In general, the precision of the measurement process is such that only in rare instances (and depending on the quality of the instrument tested) will the reported correction differ from the true correction by more than a third of the listed tolerance.

6. Tests Performed by the Bureau

6.1. Instruments Admitted for Test

At present the Bureau is prepared to test the following kinds of hydrometers and thermohydrometers: Alcoholometers, saccharometers, salinometers, lactometers,⁵ API hydrometers, Baumé hydrometers, hydrometers indicating either density or specific gravity of liquids lighter than water or heavier than water, hydrometers indicating percentage of sulfuric acid.

Instruments other than the above should not be submitted for test without previous arrangement.

6.2. Description of Tests

a. General Inspection

All hydrometers submitted for test will be given a preliminary examination to insure conformity with the recommendations on construction contained in section 2.

b. Points Tested

The hydrometer will be tested at three points on the scale: one point in the upper 25 percent of the scale length, one point near the middle of the scale, and one point in the lower 25 percent of the scale length.

Tests will be made at more than three points if requested. An additional fee is charged for each point over three tested.

c. Precision Stamp and Certificate

If the preliminary inspection of any hydrometer or thermohydrometer indicates a satisfactory conformity with the recommendations on construction, and at all points tested the results are within the limits given in the section on tolerances, the official precision stamp, consisting of the letters NBS and the year, as shown below, is etched on the instrument.



In order that this stamp may be applied to a thermohydrometer, the thermometer element also must meet the requirements set forth above.

The calibration of thermohydrometers containing "temperature correction" tables does not include a check of the accuracy of these tables, as they can, in general, be correct for only a very limited portion of the hydrometer scale.

⁵The corrections to lactometer readings as reported by the Bureau apply to readings taken at the top of the milk meniscus.

If request is made when the hydrometer is submitted, a certificate of corrections will be furnished for any instrument qualified to receive the precision stamp. An additional fee is charged for this service. Hydrometers for which certificates are issued will be marked as shown below.



d. Report of Corrections

When the results of the test show that at one or more of the points tested the corrections exceed the tolerance for precision grade but are not more than three times the precision tolerance, a report giving the corrections will be furnished if requested. When a hydrometer does not conform to the specifications as to construction, but is found suited to the testing facilities of the Bureau and adapted to the intended purpose, a report may be furnished giving the corrections and a statement explaining in what respect the hydrometer does not meet the requirements for a certificate. The fee for reports is the same as for certificates. Hydrometers on which reports are issued will receive the "reported" stamp, such as shown below, to show that they have been tested by the Bureau and, while not meeting the precision requirements, are of serviceable quality.



Certificates or reports may be issued for thermohydrometers based on test of the hydrometer element alone. The absence of any entry in the space provided for data on the test of the thermometer element will be evidence that the thermometer element was not tested.

If the correction at one or more test points exceeds three times the precision tolerance, or if for some other reason the hydrometer is found to be unsuited for the grade of use intended, neither certificate nor report is issued. Also, there is no NBS stamp applied to the instrument. The results of the test and reason for the action taken may be given in a letter.

e. Weight in Air

In addition to making the tests outlined above, the weight in milligrams of the hydrometer against brass weights in air at 760 mm of mercury pressure and 20° C, will be determined and reported if the sender so requests. (There is an additional charge for this.)

7. Directions for Submitting Apparatus for Test

7.1. Application for Test

The request for test should be made in writing and should include a complete list of the apparatus and a statement of the nature of the test desired as explained in section 6.2.

It is important that the request specify if a certificate is desired, also whether a report of corrections would be acceptable in the event that any instrument is found not qualified for a certificate.

For thermohydrometers, the request should further indicate whether or not a test of the thermometer element is desired (see section 6.2, c and d).

The sender should always examine apparatus carefully before submitting it for test to determine whether or not it complies with the recommendations and is qualified for the test desired.

7.2. Identification Marks

Instruments and the packages in which they are shipped should both be plainly marked to facilitate identification, preferably with the name of the manufacturer or slipper, and a special reference number should be given, which should be referred to in the correspondence concerning the test. After the Bureau has acknowledged the request for the test and assigned to it a test number, this test number should be given on any correspondence about the test.

7.3. Shipping Directions

Instruments should be securely packed in cases or packages, which may be used in returning them to the owner. In all cases, transportation charges are payable by the party desiring the test and

must be prepaid. Return shipment is made by express, collect unless other arrangements are made in advance. Apparatus must be accompanied by an itemized shipping invoice.

7.4. Breakage

About 3.0 percent of the hydrometers shipped to the Bureau for test are received broken. In most cases this is due to inadequate protective packing, although rough handling in shipment is undoubtedly a contributing factor. About 0.5 percent of the hydrometers received are broken during the testing operations in the laboratory. There is insufficient information at hand on which to estimate the breakage in return shipments. While the Bureau endeavors to keep the laboratory and return shipment breakage to a minimum, such breakage must be viewed as a part of the cost of submitting hydrometers for test.

7.5. Address

Articles should be addressed, "National Bureau of Standards, Washington 25, D. C." Articles delivered in person or by messenger may be left at the Shipping Room of the Bureau, or at the laboratory if the submitter wishes to discuss the tests in which he is interested. In either case, there should be a written request for the tests desired.

7.6. Remittances

Payment of test fee should be made promptly upon receipt of bill. Copies of current Test Fee Schedules may be obtained from the Bureau upon request.

8. Appendix. Relations Between Principal Scale Interval and the Dimensions of Hydrometers

Referring to figure 3, let

V = total volume (cm^3) of hydrometer from top scale mark to bottom of bulb

v = volume (cm^3) of stem per unit length

m = total mass (g) of hydrometer

D = diameter (cm) of the bulb

d = diameter (cm) of the stem

H = length (cm) of bulb

L = length (cm) of principal interval of scale in the stem (that is, distance between the two extreme marked graduation lines)

h = distance (cm) between top of bulb and bottom of scale

s_1 and s_2 = distances (cm) from the top (zero) scale line to any two readings.

If the hydrometer is placed in a liquid of density ρ_0 , it will sink to the zero (top) scale mark, hence

$$m = V\rho_0. \quad (1)$$

If placed in a second liquid having a density ρ_1 , ($\rho_1 > \rho_0$) and sinks only to position s_1 , then

$$m = (V - s_1v)\rho_1 \quad (2)$$

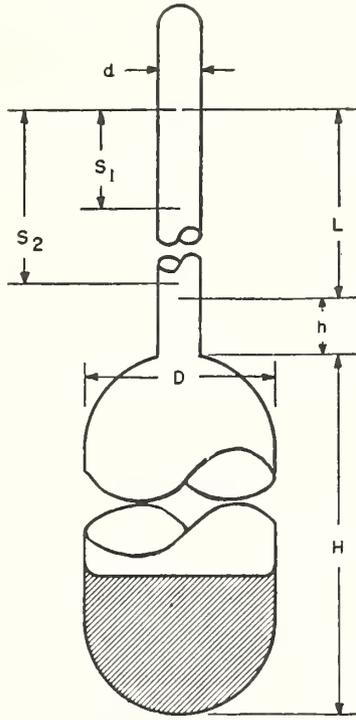


FIGURE 3. Principal dimensions of a hydrometer.

If a third liquid is used having a density ρ_2 ($\rho_2 > \rho_1$) the reading position will be s_2 , and

$$m = (V - s_2 v) \rho_2. \quad (3)$$

From eq (1) and (2),

$$V \rho_0 = (V - s_1 v) \rho_1$$

and

$$s_1 = \frac{V}{v} \left(\frac{\rho_1 - \rho_0}{\rho_1} \right); \quad (4)$$

likewise, from eq (1) and (3),

$$s_2 = \frac{V}{v} \left(\frac{\rho_2 - \rho_0}{\rho_2} \right), \quad (5)$$

and by eq (4) and (5),

$$s_1 - s_2 = \frac{V}{v} \left[\left(\frac{\rho_1 - \rho_0}{\rho_1} \right) - \left(\frac{\rho_2 - \rho_0}{\rho_2} \right) \right]. \quad (6)$$

Now assume that the densities ρ_1 and ρ_2 are going to be so chosen that the positions determined by s_1 and s_2 will coincide with the highest and lowest principal graduation lines of the scale. This

means that $\rho_1 = \rho_0$, $s_1 = 0$ and $s_2 = L$, and, therefore eq (6) reduces to

$$s_1 - s_2 = 0 - s_2 = -L = \frac{V}{v} \left[\left(\frac{\rho_1 - \rho_1}{\rho_1} \right) - \left(\frac{\rho_2 - \rho_1}{\rho_2} \right) \right],$$

or

$$L = \frac{V}{v} \left(\frac{\rho_2 - \rho_1}{\rho_2} \right). \quad (7)$$

If the ends of the bulb are assumed to be spherical segments approximately $D/3$ cm in height, the volume, V , of the hydrometer is

$$V = \frac{\pi}{4} D^2 (H - 0.3D) + \frac{\pi}{4} d^2 h + \frac{\pi}{4} d^2 L. \quad (8)$$

The 0.3 factor in $(H - 0.3D)$ is a rounded value but is sufficiently exact for most purposes. Also, since by definition, the length factor of v is unity,

$$v = \frac{\pi}{4} d^2. \quad (9)$$

Therefore,

$$\frac{V}{v} = \frac{D^2}{d^2} (H - 0.3D) + h + L \quad (10)$$

and with eq (7)

$$L \left(\frac{\rho_2}{\rho_2 - \rho_1} \right) = \frac{D^2}{d^2} (H - 0.3D) + h + L. \quad (11)$$

In many cases it may be more convenient to use specific gravity, g , in place of density. This will not change the numerical value of the density term; hence, in place of eq (11) we may write

$$L \left(\frac{g_2}{g_2 - g_1} \right) = \frac{D^2}{d^2} (H - 0.3D) + h + L. \quad (12)$$

Returning to eq (6), if ρ_2 is the density corresponding to the lowest scale mark, then $s_2 = L$ and eq (6) may be written

$$L - s_1 = \frac{V}{v} \rho_0 \left(\frac{\rho_2 - \rho_1}{\rho_1 \rho_2} \right). \quad (13)$$

Having determined the dimensions of the bulb and stem, we have established the values of V and v , so that V/v is a constant. Also, for any given range, ρ_0 is a constant. Therefore, the density term $(\rho_2 - \rho_1)/\rho_1 \rho_2$, governs the distance between the graduation lines on the scale. Furthermore, as ρ_1 is increased by equal increments from ρ_0 to ρ_2 , the value of the density term changes by de-

creasing increments. This means that for equal changes in density the distance between graduation marks on the scale will be smaller near the bottom of the scale than at the top of the scale.

In order to use either eq (11) or (12) to determine the proportions of a hydrometer, it will be necessary to assign values to enough of the variables, or to use additional relations between some of them, so as to make it possible to solve for one variable. One additional relation that may be used is

$$\text{total length} = H + h + L. \quad (14)$$

Thus, we may assign values to total length, D , d , and h . Then for any range of specific gravities that it is desired to have the hydrometer cover,

we may solve for H and L .

Example:

$$\begin{aligned} \text{Total length} &= 21.0 \text{ cm} \\ D &= 3.0 \text{ cm} \\ d &= 0.6 \text{ cm} \\ h &= 1.0 \text{ cm} \\ g_1 &= 0.95178 \\ g_2 &= 1.0000 \end{aligned}$$

Then by eq (14)

$$H = 20 - L$$

and by eq (12)

$$19.738L = 25(19.10 - L) + 1$$

or

$$L = 10.70 \text{ cm}$$

and

$$H = 9.30 \text{ cm.}$$

WASHINGTON, April 28, 1954.

Simplification of Calculations in Routine Density and Volumetric Determinations

Charles T. Collett

In density and volumetric determinations, certain methods of weighing have encouraged the use of approximations. These can be avoided by minor changes in procedure that simplify calculations. The use of counterpoises is shown to eliminate some buoyancy corrections.

1. Introduction

In the course of performing numerous density and volumetric determinations several methods were compared with regard to the calculations associated with them.

Some methods involved equations that were found adequate and correct but too complicated to be desirable for routine work. For example, Gould and Stott¹ represent the density of a liquid in a glass pycnometer by an equation of 26 terms and some 37 brackets, parentheses, shillings, etc.

Other systems were observed to lead the worker into dilemmas, from which he escaped by the use of approximations. These did not reduce the accuracy of his computed values, but were time-consuming and difficult to record in concise form. Often the steps of the calculations were worked out on scratch paper and then discarded, only the terminal values of the series being permanently recorded.

The methods and equations finally selected for everyday use were derived from inspection of the component forces acting on the arms of a balance in equilibrium, i. e., forces resulting from the masses pushing downward and the buoyant effects pushing upward. Because these equations are based on considerations that are elementary to the problem, it would seem highly probable that they have been used before, and perhaps published. A search of the literature has not revealed them, at least not in the forms shown here. Certainly, they have been neglected in modern practice, and their simplicity and usefulness suggest that they should be presented for consideration.

2. Hydrostatic Weighing

Consider that it is desired to find the mass, M_s , of a glass object of irregular shape by the conventional substitution weighing. Assume that at all times a constant load, c , remains on the right pan of the laboratory balance, a condition that will be unchanged during all weighings subsequently discussed, and the designation as right or left being an arbitrary identification without other significance.

¹ F. A. Gould and Verney Stott, *Glazebrook's dictionary of applied physics*, III, 127 (Peter Smith, New York, N. Y., 1950).

Equilibrium is established by placing on the left pan brass weights e and f , whose masses are M_e and M_f and which are subject to air-buoyancy effects, B_e and B_f . M_c is the mass of c , and B_c is the air buoyancy on c . Then

$$M_e - B_e + M_f - B_f = M_c - B_c. \quad (1)$$

If the glass specimen, s , is then placed on the left pan, and equilibrium restored by removing weight f ,

$$M_c - B_c + M_s - B_s = M_c - B_c, \quad (2)$$

where M_s represents the mass of the specimen and B_s the air buoyancy acting upon it. The second weighing must be accomplished within a reasonably short time, so that the air density has not changed. Subtracting eq (2) from eq (1), and rearranging,

$$\begin{aligned} M_s &= M_f - B_f + B_s \\ &= M_f - \frac{M_f}{\rho_f} \rho_a + V_s \rho_a \\ &= M_f \left(1 - \frac{\rho_a}{\rho_f} \right) + V_s \rho_a, \end{aligned} \quad (3)$$

where ρ_a is the density of the air, ρ_f is the density of the weights, and V_s is the volume of the specimen.

ρ_a can be obtained by well-known methods,² and ρ_f is usually known. However, V_s is unknown due to the shape of the specimen and is customarily found by means of hydrostatic weighing.

A suspension is fitted below the left pan to support a basket immersed in a liquid, usually distilled water, and equilibrium is secured by the addition of weights g and h on the pan. Let M_k equal the mass of the suspension system and B_k and B'_k the respective air and water buoyancy effects.

$$M_g - B_g + M_h - B_h + M_k - B_k - B'_k = M_c - B_c. \quad (4)$$

The next operation must be performed within a short period of time, that is, before the density of the air or the water has changed appreciably. The speci-

² N. Bauer, *Technique of organic chemistry*, vol. 1, part 1, Physical Methods, p. 273 (Interscience Publishers, Inc., New York, N. Y., 1949). N. S. Osborne, E. C. McKelvey, and H. W. Bearce, Density and thermal expansion of ethyl alcohol and of its mixtures with water, *Bull. BS* 9, 327 (1913) S197 (see p. 378).

men is immersed within the basket and is subject to buoyancy effect B'_s . Weight h is removed to restore equilibrium. Then

$$M_g - B_g + M_k - B_k - B'_k + M_s - B'_s = M_c - B_c. \quad (5)$$

Subtracting eq (5) from eq (4)

$$\begin{aligned} B'_s &= M_s - M_h + B_h \\ &= M_s - M_h + \frac{M_h}{\rho_h} \rho'_a \\ &= M_s - M_h \left(1 - \frac{\rho'_a}{\rho_h}\right). \end{aligned} \quad (6)$$

Again an unknown, M_s , appears on the right side of the equation, making it impossible to use directly the relationship

$$\begin{aligned} V_s &= \frac{B'_s}{\rho_w} \\ &= \frac{M_s - M_h \left(1 - \frac{\rho'_a}{\rho_h}\right)}{\rho_w}, \end{aligned} \quad (7)$$

where ρ_w is the density of the water.³

The unknown quantities may be eliminated from the right-hand sides of eq (3) and (7) by substitution. The resulting equations can be solved directly but contain more terms, thus:

$$M_s = \frac{M_f \left(1 - \frac{\rho_a}{\rho_f}\right) - M_h \left(\frac{\rho_a}{\rho_w}\right) \left(1 - \frac{\rho'_a}{\rho_f}\right)}{1 - \frac{\rho_a}{\rho_w}} \quad (8)$$

$$V_s = \frac{M_f \left(1 - \frac{\rho_a}{\rho_f}\right) - M_h \left(1 - \frac{\rho'_a}{\rho_f}\right)}{\rho_w - \rho_a} \quad (9)$$

However, if eq (9) is solved first, the value of V_s may be used in eq (3), which is much shorter.

Equations (3) and (7) may be solved by the use of approximations, a method that has been found in considerable use.⁴ In a typical case it was found necessary to remove weights equal to 11.6954 g after the glass sample was placed on the pan. Air density was determined to be 0.001170 g/ml, and the weights were known to have a density of 8.4 g/cm³. From eq (3):

$$\begin{aligned} M_s &= 11.6954 \left(1 - \frac{0.001170}{8.4}\right) + V_s(0.001170) \\ &= 11.6938 + V_s(0.001170), \text{ in grams.} \end{aligned} \quad (10)$$

For the hydrostatic weighing, equilibrium was maintained by removing 6.4711 g after the sample was placed in the immersed basket. Densities of

the water and air were 0.996953 g/ml and 0.001176 g/ml. From eq (7)

$$\begin{aligned} V_s &= \frac{M_s - 6.4711 \left(1 - \frac{0.001176}{8.4}\right)}{0.996953} \\ &= \frac{M_s - 6.4702}{0.996953} \text{ ml.} \end{aligned} \quad (11)$$

The first approximation is secured by using the apparent weight of the sample in place of M_s in eq (11), giving

$$\text{approx.}_1 V_s = \frac{11.6954 - 6.4702}{0.996953} = 5.2412 \text{ cm}^3. \quad (12)$$

This approximate value for V_s is used in eq (10), and we have

$$\text{approx.}_1 M_s = 11.6938 + 5.2412(0.001170) = 11.6999 \text{ g.} \quad (13)$$

The second approximations are

$$\text{approx.}_2 V_s = \frac{11.6999 - 6.4702}{0.996953} = 5.2457 \text{ cm}^3 \quad (14)$$

$$\text{approx.}_2 M_s = 11.6938 + 5.2457(0.001170) = 11.6999 \text{ g.} \quad (15)$$

These last approximate values are accepted for the real values, as obviously no further change will occur.

A minor alteration in this procedure will simplify the equations and entirely eliminate the use of approximations. The suspended, immersed basket is retained, but the specimen rests on the left pan during the first weighing, and equilibrium is produced by the presence of weight g . Equation (4) then becomes

$$M_g - B_g + M_s - B_s + M_k - B_k - B'_k = M_c - B_c. \quad (16)$$

For the second weighing the specimen is transferred to the basket to be immersed as before, but to produce equilibrium it is necessary to add weight h on the pan.⁵ Equation (5) then is changed to:

$$\begin{aligned} M_g - B_g + M_h - B_h + M_k - B_k - \\ B'_k + M_s - B'_s = M_c - B_c. \end{aligned} \quad (17)$$

Subtracting eq (17) from eq (16)

$$\begin{aligned} B_s - B'_s &= M_h - B_h \\ V_s(\rho_w - \rho_a) &= M_h - \frac{M_h}{\rho_h} \rho_a \\ V_s &= \frac{M_h \left(1 - \frac{\rho_a}{\rho_h}\right)}{\rho_w - \rho_a}. \end{aligned} \quad (18)$$

³ A similar equation appears in *Bul. BS 9*, 327 (1913) S197, see p. 384, where the density of a liquid is found by immersing a sinker of known mass and volume.

⁴ NBS Circular 487, p. 2 (1950).

⁵ The transfer of sample from pan to basket is described by W. Watson (*A textbook of physics*, p. 175, Longmans, Green & Co., London, 1902), but the calculations are not mentioned.

Because the densities of weight h , air, and water can be secured easily, it is apparent that eq (18) contains no unknowns on the right-hand side, and the exact value of V_s may be calculated at once. After its volume is thus determined, the object is weighed in air in the conventional manner, after which eq (3) can be solved.

The same glass piece previously considered was also weighed by this latter method. After the sample was moved from the pan to the immersed basket, it was necessary to place 5.2243 g on the pan. The densities of the air and water were respectively 0.001170 and 0.996953 g/ml, and of the weights, 8.4 g/cm.³

From eq (18)

$$V_s = \frac{5.2243 \left(1 - \frac{0.001170}{8.4}\right)}{0.996953 - 0.001170} = 5.2457 \text{ cm}^3. \quad (19)$$

The sample was then weighed in air in the conventional substitution method, 11.6954 g being removed after the specimen was placed on the pan. Air density was 0.001170 g/ml.

From eq (3):

$$\begin{aligned} M_s &= 11.6954 \left(1 - \frac{0.001170}{8.4}\right) + 5.2457(0.001170) \\ &= 11.6999 \text{ g}. \end{aligned} \quad (20)$$

3. Weighing a Picnometer by the Counterpoise Method

Weighing a picnometer is a special case of determining the mass of a glass object having irregular shape and unknown volume, and is therefore subject to the same general principles. If an attempt is made to use conventional substitution weighing, the same dilemma will of course arise, that is, neither the mass nor the volume is known, and neither can be determined without knowledge of the other. The volume here referred to is the volume of material (glass) that will displace air during the weighing, and does not refer to the capacity of the container, or so-called "internal volume."

Furthermore, the interior of the picnometer for liquids is of necessity closed to the outside except for limited entrances, usually capillary tubes of small diameter. For this reason, filling, emptying, cleaning, and drying are not quickly accomplished, and the observer will wish to hold the number of such operations to a minimum. For example, a series of trials may be performed while the same liquid remains in the instrument, only minor adjustments of level being made between succeeding weighings.

Thus the weighing of the empty picnometer and the weighing of the filled picnometer are often separated by considerable periods of time, and the air density, and hence the air buoyancy effects, will vary.

However, the user of the picnometer is not primarily interested in the mass of the instrument;

this value is never required except as a tare, and its consistent treatment as such throughout all operations will permit the use of simple calculations with no approximations. By the use of a counterpoise the picnometer's mass and its buoyancy in air may be entirely eliminated from all calculations, and in fact need never be known at any time.⁶

The counterpoise is a second picnometer similar in size and shape to the first, made of glass with the same density, and the two instruments must be approximately equal in mass. A small protuberance is provided on each, such as the handle on the cap, and by grinding and polishing it is not difficult to adjust the masses within a milligram. However, the two instruments may be allowed to differ by as much as 0.1 g because the difference in buoyant effects when they are suspended from opposite balance pans with equilibrium maintained by weights is $(\Delta/\rho_g - \Delta/\rho_f)\rho_a$, where Δ is the difference between the masses of the two picnometers, and ρ_g , ρ_f , and ρ_s are the densities of the glass, weights, and air. If Δ is 0.1 g and the respective densities are about 2.5, 8.4, and 0.0012 g/ml, the difference in buoyant effect will be negligible.

In conventional laboratory practice the mass, volume of glass, and internal volume of a picnometer are not usually determined each time the instrument is used, but only at the time it is calibrated. With occasional checking, these values are considered true as long as there is no reason to suspect a change. In the method to be considered, only the internal volume at various temperatures is determined.

The counterpoise is placed upon the right pan with a constant load composed of weights e and f , and the active picnometer is placed on the left pan with weights E and F sufficient to secure a state of balance, from which the equation may be written

$$\begin{aligned} M_p - B_p + M_e - B_e + M_f - B_f = \\ M_p - B_p + M_e - B_e + M_f - B_f, \end{aligned} \quad (21)$$

where M is the mass, and B is the air buoyancy effect of the object indicated by the subscript letter; P and p being the active picnometer and the counterpoise picnometer.

The picnometer is next filled with a sample, S , which will be distilled water during the calibration and later the particular liquid being tested for density. Balance is restored by removing weight F . Then

$$\begin{aligned} M_p - B_p'' + M_s - B_s'' + M_e - B_e'' = \\ M_p - B_p'' + M_e - B_e'' + M_f - B_f''. \end{aligned} \quad (22)$$

It has been observed that the two weighings may be performed at different times, thus a change in air density is to be expected. The buoyant effects in eq (22) will not be equal to those in eq (21) except by coincidence and are thus designated as B'' .

⁶ N. Bauer, *Technique of organic chemistry*, I, part I, Physical methods, p. 272 (Interscience Publishers, Inc., New York, N. Y., 1949).

Subtracting eq (22) from eq (21), we have

$$M_F - B_F - B_P - B_E + B_P'' + B_E'' - M_S + B_S'' = B_P'' + B_E'' + B_F'' - B_P - B_E - B_F. \quad (23)$$

Assume that the small difference in mass between P and p in eq (21) and (22) has been compensated by a corresponding difference in E and e , so that

$$M_P + M_E = M_p + M_e. \quad (24)$$

It has been shown that the buoyancy effects may be considered equal, that is,

$$B_P + B_E = B_p + B_e. \quad (25)$$

Also

$$B_P'' + B_E'' = B_p'' + B_e''. \quad (26)$$

It follows that M_F must be equal to M_f , and as the weights have the same density, B_F and B_f are equal.

We then have

$$M_S - B_S'' = M_F - B_F'', \quad (27)$$

which is essentially the same as eq (3).

$$M_S - \frac{M_S}{\rho_S} \rho_a'' = M_F - \frac{M_F}{\rho_F} \rho_a'', \quad (28)$$

where ρ_a'' is the air density corresponding to eq (22).

$$M_S = M_F \frac{1 - \frac{\rho_a''}{\rho_F}}{1 - \frac{\rho_a''}{\rho_S}}. \quad (29)$$

As the sample in the picnometer during calibration is water at a known temperature, the density ρ_S is read from standard tables, and the volume then is M_S divided by that density. This process is repeated at different temperatures until a calibration curve has been developed for the picnometer over the desired range.

When a liquid of unknown density is to be tested the weighing procedure is exactly the same. However, eq (27), as shown in eq (3), can be rewritten to obtain

$$M_S = M_F \left(1 - \frac{\rho_a''}{\rho_F} \right) + V_S \rho_a''. \quad (30)$$

The picnometer having been previously calibrated, V_S is readily determined and

$$\rho_S = \frac{M_S}{V_S} = \frac{M_F \left(1 - \frac{\rho_a''}{\rho_F} \right) + V_S \rho_a''}{V_S}. \quad (31)$$

For example, when a picnometer was calibrated according to the method described, a weight of 120.9878 g was on the left pan with the empty instrument, and 21.1467 g when it was filled with distilled water at a temperature of 24.288° C. The densities of the weights, air, and water were determined to be 8.4; 0.001170, and 0.997257 g/ml, respectively. From eq (29) the mass of the water was

$$M_S = 99.8411 \frac{1 - \frac{0.001170}{8.4}}{1 - \frac{0.001170}{0.997257}} = 99.9445 \text{ g.} \quad (32)$$

The volume of the instrument was

$$V_S = \frac{99.9445}{0.997257} = 100.2194 \text{ ml.} \quad (33)$$

This was used as one point on a calibration curve.

When the instrument was used to test a sample of heptane a weight of 116.5413 g was required on the left pan with the empty picnometer. It was then filled with the sample at a temperature of 24.992° C, and balance was restored by reducing the weight on the pan to 48.5356 g. The densities of the weights and the air were 8.4 and 0.001173 g/ml, respectively. From the previously drawn calibration curve the volume of the instrument at that temperature was known to be 100.2201 ml.

Using eq (31), the density of the heptane was

$$\rho_S = \frac{68.0057 \left(1 - \frac{0.001173}{8.4} \right) + (100.2201)(0.001173)}{100.2201} = 0.67964 \text{ g/ml.} \quad (34)$$

WASHINGTON, October 19, 1953.

Testing of Glass Volumetric Apparatus

J. C. Hughes



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Issued April 1, 1959

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Foreword

This Circular is a revision of NBS Circular 434, by Elmer L. Peffer (deceased) and Grace C. Mulligan. Its purpose is to bring up to date the specifications for glass volumetric apparatus of precision grade, the description of the test methods used, and the directions for submitting apparatus for test.

Specifications for newly developed items have been added, while others have been changed to improve the usefulness of items or to take into consideration new manufacturing methods.

A. V. ASTIN, *Director*.

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Testing of Glass Volumetric Apparatus

J. C. Hughes

This Circular contains specifications and tolerances for glass volumetric apparatus of precision grade. Detailed information is given as to dimensions, graduations, inscriptions, and tolerances for burets, pipets, flasks, cylindrical graduates, and certain kinds of special apparatus.

A description of test methods used and the reports furnished and directions for submitting apparatus are included.

1. Introduction—Purpose and Scope

This Circular presents, to manufacturers and users, information on the design, construction, and verification of glass volumetric apparatus and indicates the facilities available at the Bureau for this verification.

The specifications and tolerances given herein

apply to burets, flasks, pipets, and other glass volumetric apparatus of precision grade.

Information is given on the method of reading, the test liquid used, and the methods of test. A description of the tests performed and the reports furnished and directions for submitting apparatus are included.

2. Standard Specifications for Glass Volumetric Apparatus

The primary purpose of these specifications is to define the requisite qualifications for precision apparatus.

The Bureau aims to encourage excellence in quality by cooperating with makers and users of apparatus, and to this end endeavors to assist manufacturers in establishing standards and perfecting their methods. In order that users of standardized apparatus may fully benefit by the facilities of the Bureau, it is necessary for them when purchasing apparatus to be submitted for test to require that the apparatus shall comply with the specifications of the Bureau. By admitting for test only apparatus conforming to these standards, the work of testing is confined to apparatus whose utility is sufficient to justify the labor expended in its accurate calibration. Certain of the specifications, such as those regarding quality of glass and process of annealing before calibration, are largely dependent on the integrity of the maker for their fulfillment. Only by supporting conscientious makers, in giving consideration first to quality and second to cost, can users of standardized apparatus secure a high degree of excellence.

2.1. Types of Apparatus That Will Be Regularly Admitted for Test

Precision grade apparatus of the following types will be accepted for test on written request: transfer pipets and capacity pipets, without subdivisions; burets, measuring pipets, and dilution pipets, with partial or complete subdivisions; volumetric flasks, cylindrical graduates, and specific gravity flasks.

Other types of apparatus should not be submitted without prior arrangement.

2.2. General Specifications¹

a. Units of Capacity

A *liter* is equal to the volume occupied by the mass of 1 kg of pure water at its maximum density (at a temperature of 4° C, practically).

A *milliliter* (ml),² is the one-thousandth part of the liter.

b. Standard Temperature

Although 20° C has been almost universally adopted as the standard temperature for glass volumetric apparatus, many chemists are of the opinion that 25° C more nearly approximates the average laboratory temperature in the United States. The Bureau therefore will calibrate glass volumetric apparatus marked either 20° or 25° C.

c. Material and Annealing

The material should be of best quality glass, transparent and free from striae, surface irregularities, and other defects which may distort the appearance of the liquid surface or the portion of the graduation line seen through the glass. All apparatus should be thoroughly annealed before being graduated.

d. Design and Workmanship

The cross section must be circular and the shape must permit complete emptying and draining and thorough cleaning.

Instruments having a base or foot must stand solidly on a level surface. For all instruments

¹ The Bureau reserves the right to reject any apparatus on points affecting its accuracy or utility not covered by these specifications.

² The terms "milliliter" and "cubic centimeter" are sometimes used interchangeably. While they are not exactly equal (1 ml=1.000028 cm³), the difference is seldom of consequence in volumetric analysis.

except small flasks (below 25 ml) the base shall be of such size that the instrument, empty and without stopper, will stand on a plane inclined 15° to the horizontal. Flasks smaller than 25 ml shall stand on a 10° incline.

Stoppers and stopcocks must be ground or fitted so as to work easily and prevent leakage.

Apparatus which is manifestly fragile or otherwise defective in construction will not be accepted.

The parts on which graduations are placed must be cylindrical for at least 1 cm on each side of every mark (except on certain small flasks and special apparatus), but elsewhere may be enlarged to provide the desired capacities in convenient lengths.

Two scales are not permitted on the same piece of apparatus. For example, apparatus should not be graduated in both fluid ounces and milliliters. In the case of two units, one of which is an exact multiple of the other, such, for example, as drams and fluid ounces, there is no objection to having the 8-dr line, 16-dr line, etc., marked respectively, 1 fl oz, 2 fl oz, etc., provided that the two series of numbers are placed on opposite sides of the apparatus and the value of each subdivision is suitably indicated.

e. Graduation Lines

Graduation lines shall be fine, clean, permanent, continuous, and of uniform width, perpendicular to the axis and parallel to the base of the apparatus. Line width should not exceed 0.3 mm for subdivided apparatus and 0.4 mm for single-line apparatus.

All graduations must extend at least halfway around; and on subdivided apparatus at least every 10th mark, and on undivided apparatus, all marks must extend completely around the circumference. Subdivided apparatus must be provided with a sufficient number of lines of suitable length to facilitate reading.

The clear space between two adjacent marks must be not less than 1 mm wide. The spacing of marks on subdivided apparatus must show no apparent irregularities, and sufficient divisions must be numbered to readily indicate the intended capacity of any interval.

f. Inscriptions

Every instrument must bear in permanent legible characters the capacity, the temperature at which it is to be used, the method of use—that is, whether to contain or to deliver—and on instruments which deliver through an outflow nozzle, the time required to empty the total nominal capacity with unrestricted outflow. Etching is preferred for inscriptions, although engraved or fused marking is acceptable provided such marking is neat and clear. Grit-blasted serial numbers will be permitted with the same provision.

Every instrument must bear the name or trademark of the maker. Every instrument must bear a permanent identification number, and detach-

able parts, such as stoppers, stopcocks, etc., belonging thereto, if not interchangeably ground, must bear the same number. Interchangeable ground-glass parts shall be marked on both members with the proper standard taper symbol and the size designation, in accordance with Commercial Standard CS 21-57.

Figure 1 illustrates several arrangements of designating marks that are considered suitable. Marks may be placed elsewhere on apparatus if they are easily readable and do not interfere with the proper use of the apparatus.

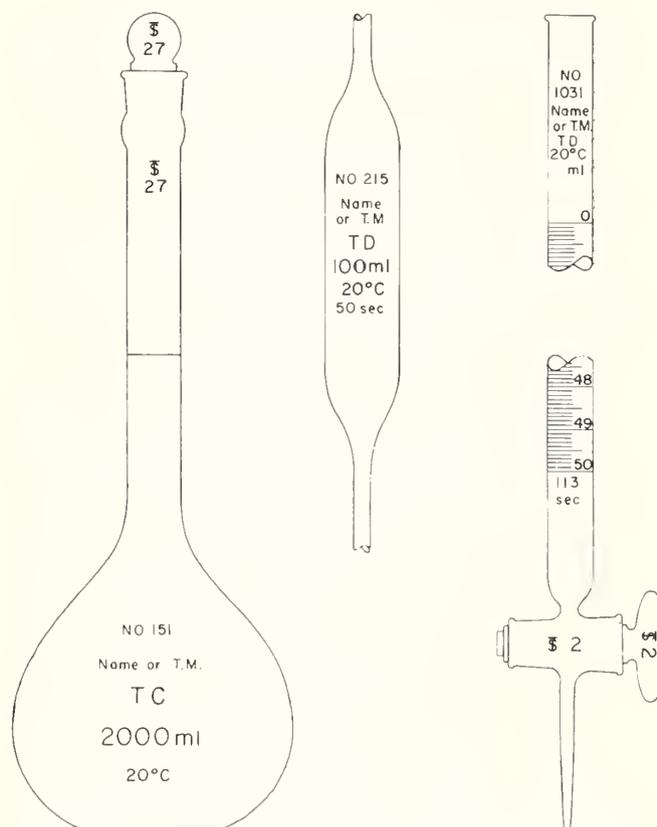


FIGURE 1. Examples of inscriptions suitable for volumetric apparatus.

2.3. Special Requirements

a. Flasks

The limiting dimensions for precision-grade volumetric flasks are given in table 1. For flasks smaller than 5-ml capacity, the limits on location of the graduation line will be the same as shown for a 5-ml flask.

Stopper sizes should be so chosen that the smallest diameter of the ground zone in the flask neck is approximately equal to the inside diameter at the graduation line. The inside diameter of the neck at the graduation line should never exceed the smallest diameter of the ground zone by more than 1.5 mm. The shoulder at the base of the ground zone should be smoothly rounded to permit complete drainage.

TABLE 1. *Limiting dimensions for volumetric flasks*
(In millimeters)

Capacity	Inside diameter of neck at graduation line		Minimum distance between graduation line and:		
	Minimum	Maximum	Top of flask		Bulb ^a
			Stoppered type	Unstoppered type	
<i>ml</i>					
5.....	6	7	22	22	5
10.....	6	8	28	28	7
25.....	6	8	35	30	10
50.....	6	10	40	30	10
100.....	8	12	40	30	10
200.....	9	14	55	45	10
250.....	10	15	55	45	10
500.....	12	18	60	60	20
1,000.....	14	20	70	60	20
2,000.....	18	25	70	60	20

^a The distance between the graduation line and the bulb is measured to the point where the neck begins to expand into the bulb (on outside).

A flask may be graduated both *to contain* and *to deliver*, provided the intention of the different marks is clearly indicated and provided the distance between the two marks is not less than 1 mm.

b. Cylindrical Graduates

Cylinders may be graduated either to contain or to deliver, but a scale numbered both up and down the length of the graduate is not permitted, as it is obvious that the same graduate cannot be correct both to contain and to deliver.

The relation of the height to the diameter must be such that the graduation marks are not less than 1 mm apart, and also that the graduated height is at least five times the inside diameter. In the case of the 10-ml cylinder subdivided to 0.1 ml, and the 25-ml cylinder subdivided to 0.2 ml, it will be found necessary to make the graduated height considerably more than five times the inside diameter in order to give a separation of 1 mm to the graduation marks. To avoid excessive height, the subdivisions on 10-ml, 25-ml, and 50-ml cylinders may be 0.2 ml, 0.5 ml, and 1.0 ml, respectively.

Subdivision lines shall be omitted between the base and the first numbered line. This will eliminate readings near the base which are difficult and not always accurate. (See fig. 2.)

The numbers indicating the capacity of the graduate at its different points should be placed immediately above the marks to which they refer.

Method of use—Graduates that are to be used dry to receive and measure liquids should be calibrated to contain.

Graduates that are to be used to pour water into other measures, and those which are to be used wet to contain water from other measures, should be calibrated to deliver. For example, a graduate that is to be used in testing milk bottles, either by pouring water from the graduate into the bottles or from the bottles into the graduate without drying the graduate between bottles, should be cali-

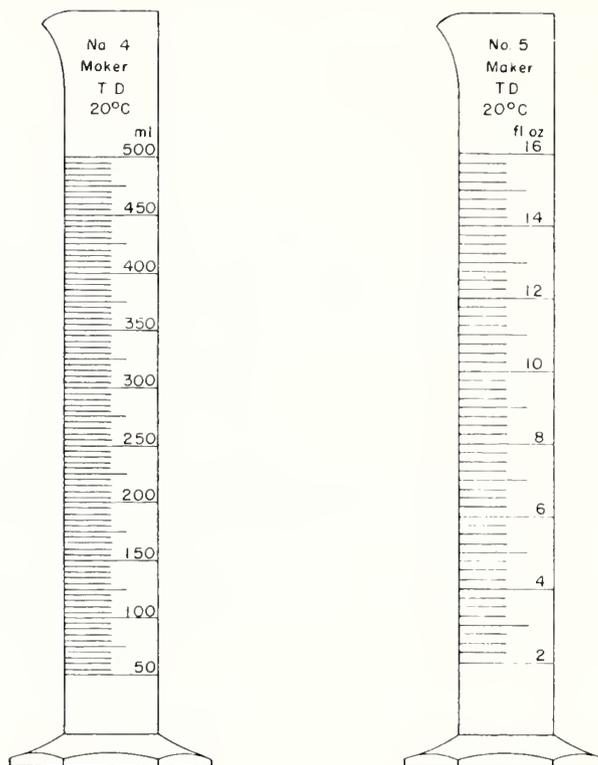


FIGURE 2. *Examples of cylindrical graduates.*

brated to deliver. After having been wet the graduate will, on successive fillings and emptyings, deliver the same quantity that is poured into it.

In ordering graduates, the purchaser should consider the use to which they are to be put and should specify accordingly whether they should be calibrated to contain or to deliver.

Basis of graduation—Graduates in customary units should be graduated in accordance with the following relations:

$$\begin{aligned}
 60 \text{ minims} &= 1 \text{ fl dr} \\
 8 \text{ fl dr} &= 1 \text{ fl oz} \\
 32 \text{ fl oz} &= 1 \text{ liq qt} \\
 4 \text{ liq qt} &= 1 \text{ U. S. gal} \\
 1 \text{ U. S. gal} &= 231 \text{ in.}^3
 \end{aligned}$$

For conversion to the metric system, the relation is:

$$1 \text{ fl oz} = 29.5729 \text{ ml}$$

TABLE 2. *Limits of distance from highest graduation to top of graduate*

Total nominal capacity (customary units)	Distance of highest graduation from top		Total nominal capacity (metric units)
	Minimum	Maximum	
<i>Minims</i>	<i>cm</i>	<i>cm</i>	<i>ml</i>
60.....	2	4	5
120.....	2	4	10
<i>fl oz</i>			
1.....	2	5	25
2.....	3	5	50
4.....	3	6	100
8.....	4	8	250
16.....	5	10	500
32.....	5	10	1,000

TABLE 3. Minimum neck diameter of cylinders with stoppers

Capacity-----ml	5	10	25	50	100	250	500	1,000
Minimum diameter of neck-----mm	6	7	8	9	10	12	15	18

c. Transfer Pipets

Pipets for delivering a single volume are designated "transfer" pipets.

The suction tube of each transfer pipet must be at least 16 cm long, and the delivery tube must be not less than 3 cm nor more than 25 cm long. The top of the suction tube must be finished with a smooth plane surface, at right angles to the axis.

The outside diameter of the suction and delivery tubes of transfer pipets, exclusive of the tip, must be not less than 5 mm. Limits of inside diameter at the capacity mark are given in table 4.

The capacity mark on transfer pipets must be not more than 6 cm from the bulb.

The outlet of any transfer pipet must be of such size that the free outflow shall last not more than 1 min and not less than the times shown in table 5 for the respective sizes.

TABLE 4. Inside diameter of transfer pipets at capacity mark

Capacity of pipets (in milliliters) up to and including-----	25	50	200
Diameter (in millimeters): Max-----	4	5	6
Min-----	2	2	2

TABLE 5. Minimum delivery times for transfer pipets

Capacity (in milliliters) up to and including-----	5	10	50	100	200
Outflow time (in seconds)-----	15	20	30	40	50

d. Burets and Measuring Pipets

Only those burets emptying through a nozzle permanently attached at the bottom are accepted for test.

Side tubes, unless provided with stopcocks, are not permitted on burets.

So-called Schellbach burets—that is, those having a milk-glass background with a colored center line—will not be accepted for test on account of possible errors resulting from parallax.

The distance between the extreme graduations must not exceed 70 cm on burets nor 35 cm on measuring pipets.

The rate of outflow of burets and measuring pipets must be restricted by the size of the tip, and for the graduated length the time of free outflow must be not more than 3 min nor less than the times shown in table 6.

The upper end of any measuring pipet must be not less than 10 cm from the uppermost mark and the lower end not less than 4 cm from the lowest mark.

On 50- and 100-ml burets, the highest graduation mark should be not less than 4 cm nor more than 10 cm from the upper end of the buret.

TABLE 6. Minimum delivery times for burets and measuring pipets

Length graduated	Time of outflow not less than—	Length graduated	Time of outflow not less than—
<i>cm</i>	<i>sec</i>	<i>cm</i>	<i>sec</i>
15	30	45	80
20	35	50	90
25	40	55	105
30	50	60	120
35	60	65	140
40	70	70	160

On burets having a capacity of 25 ml or less this distance should be not less than 3 cm nor more than 6 cm. (These requirements do not apply to burets where the "zero" is at the end of an overflow tip.)

e. Buret and Pipet Tips

Buret and pipet tips should be made with a gradual taper of from 2 to 3 cm, the taper at the extreme end being slight.

A sudden contraction at the orifice is not permitted, and the end of the tip must be ground perpendicular to the axis of the tube. The outside edge should be beveled slightly and all ground surfaces polished.

In order to facilitate the removal of drops and to avoid splashing, the tip of a buret may be bent slightly.

Approved forms of tips for burets, measuring pipets, and transfer pipets are shown in figure 3.

2.4. Special Apparatus

a. Giles Flasks

The permissible error in the volume at the first mark shall be the tolerance allowed for a flask of that capacity. The permissible error in the volume at the second mark shall be the tolerance at the first mark plus the tolerance for a flask of capacity equal to the difference between the two marks, provided, however, that the error in the volume between the two marks shall not exceed the permissible error in the volume indicated by the first mark.

At the capacity mark the inside diameter of the neck shall be within the limits given in table 7.

The neck shall be cylindrical on each side of every graduation mark for at least the distances given in table 8.

On account of the bulb in the neck of a Giles flask it is more unstable or topheavy than a flask of the ordinary type. For that reason it has been thought advisable to allow a somewhat shorter minimum length for the cylindrical portion of the neck, on each side of the graduation mark, than is allowed on ordinary flasks. In other respects the same general specifications apply to the Giles flasks as to other volumetric apparatus.

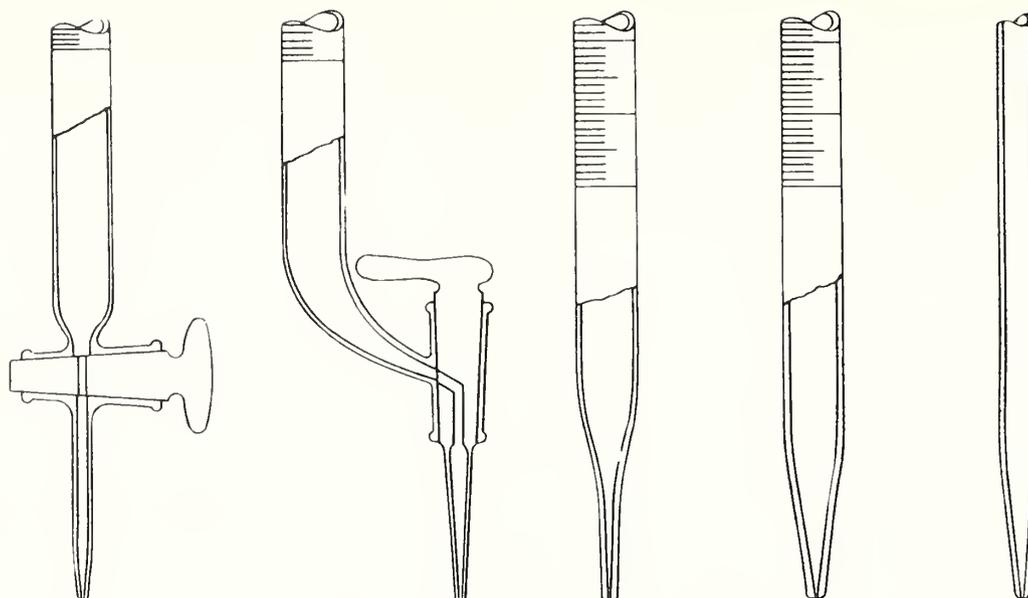


FIGURE 3. Types of buret and pipet tips.

TABLE 7. Neck diameters of Giles flasks

Capacity	Inside diameter of neck	
	Minimum	Maximum
<i>ml</i>	<i>mm</i>	<i>mm</i>
25 to 27.5.....	6	9
50 to 55.....	6	11
100 to 110.....	8	13
200 to 220.....	9	15
500 to 550.....	12	18
1,000 to 1,100.....	14	20
2,000 to 2,200.....	18	25

TABLE 8. Minimum length of cylindrical portions of neck on Giles flask

Capacity	Above upper graduation mark	Between upper graduation mark and bulb	Between lower graduation mark and bulb	Below lower graduation mark
<i>ml</i>	<i>cm</i>	<i>cm</i>	<i>cm</i>	<i>cm</i>
25 to 27.5.....	3	0.8	1	1
50 to 55.....	3	.8	1	1
100 to 110.....	3	.8	1	1
200 to 220.....	4	.8	1.5	2
500 to 550.....	4	1.0	1.5	2
1,000 to 1,100.....	5	1.0	1.5	2
2,000 to 2,200.....	5	1.0	1.5	2

b. Specific Gravity Flasks

Material and annealing—The material from which the flasks are made shall be glass of the best quality, transparent, and free from striae. It shall adequately resist chemical action and have small thermal hysteresis. The flasks shall be thoroughly annealed before being graduated. They shall be of sufficient thickness to insure reasonable resistance to breakage.

Design—The cross section of the flask shall be circular, and the shape and dimensions shall conform to the diagram shown in figure 4. This design is intended to insure complete drainage of the flask on emptying and stability of standing on a level surface, as well as accuracy and precision of reading. There shall be a space of at least 1 cm between the highest graduation mark and the lowest point of the grinding for the glass stopper.

Capacity—The flask should contain approximately 250 ml when filled to the zero graduation mark.

Graduations—The neck shall be graduated from 0 to 1 ml and from 18 to 24 ml into 0.1-ml divisions. There shall be two 0.1-ml graduations below the 0 and two above the 1-ml graduation.

Standard temperature—The flasks shall be standard at 20° C. The indicated specific gravities will then be at 20° referred to water at 4° as unity—that is, density at 20° in grams per milliliter.

Inscriptions—Each flask shall bear a permanent identification number and the stopper, if not interchangeably ground, shall bear the same number. The standard temperature shall be indicated and the unit of capacity shall be shown by the letters "ml" placed above the highest graduation mark.

Tolerance—The error of any indicated capacity shall be not greater than 0.05 ml.

Interpretation of the specification—The foregoing specification is intended to represent the most desirable form of specific-gravity flask for use in testing cements. Variations of a few millimeters in such dimensions as total height of flask, diameter of base, etc., are to be expected and will not be considered sufficient cause for rejection. The requirements in regard to tolerance, inscriptions, and the length, spacing, and uniformity of graduations will, however, be rigidly observed.

d. Babcock Milk Test Apparatus

Because most of the States now require that all Babcock glassware used in the State be approved by officials of that State, the National Bureau of Standards no longer accepts Babcock bottles and pipets for calibration, except when specifically requested by a State to settle a dispute. Specifications for Babcock glassware may be found in official procedures of the Association of Official Agricultural Chemists and certain dairy associations.

e. Dilution (Hemocytometer) Pipets

The pipets should be of good quality glass, free from bubbles and other defects, and sufficiently strong to withstand normal usage. Tubing with milk-glass backing is permissible. The capillary should be of uniform diameter, except that the tip of a white cell pipet may be tapered slightly. The inside diameter at the tip should be between 0.3 and 0.5 mm. On all pipets the tip should be ground smooth and at right angles to the axis of the pipet, and beveled so that the external diameter at the end does not exceed 2.0 mm. All ground surfaces should be polished to restore transparency.

The bulb or mixing chamber should contain a small, nonspherical glass bead to aid in mixing the blood and diluting fluid. Pipets for red blood cells may be designated by a red bead, by a red stripe running parallel to the axis, or both. White cell pipets should have white or clear beads.

The bulb should be so shaped that it can be filled without entrapping air bubbles. The capacity of the bulb is usually about 0.8 to 1.2 ml for the red cell pipet, and 0.2 to 0.4 ml for the white cell pipet.

The pipet should be so graduated as to give dilution ratios of 1 to 10, or 1 to 100, and may be further graduated to give ratios of 0.1, 0.2, 0.5, etc., to 10, and to 100; that is, the interval 0 to 1 may be subdivided to 0.1 or 0.5. The subdivisions, when present, should be uniformly spaced along the capillary tube. The graduations at 0.5, 1.0, 11, and 101 should be numbered.

All numbered graduation lines should extend at least halfway around the pipet, diametrically opposite to the milk-glass backing. Etched lines are preferred, but engraved or fused lines are acceptable provided they meet the requirements of paragraph 2.2.e. The "1" line shall be located not less than 2 mm nor more than 5 mm below the bulb, and the "11" (or "101") line shall be not less than 2 mm nor more than 4 mm above the bulb. Measurement is from the points where the capillaries begin to expand into the bulb.

Dilution pipet tolerances—If V represents the volume between the marks immediately above and below the bulb, v_1 represents the volume of the capillary between the tip and the line marked "1", and v_2 represents the volume of the capillary between the tip and the line marked "0.5", the tolerances may be expressed as follows:

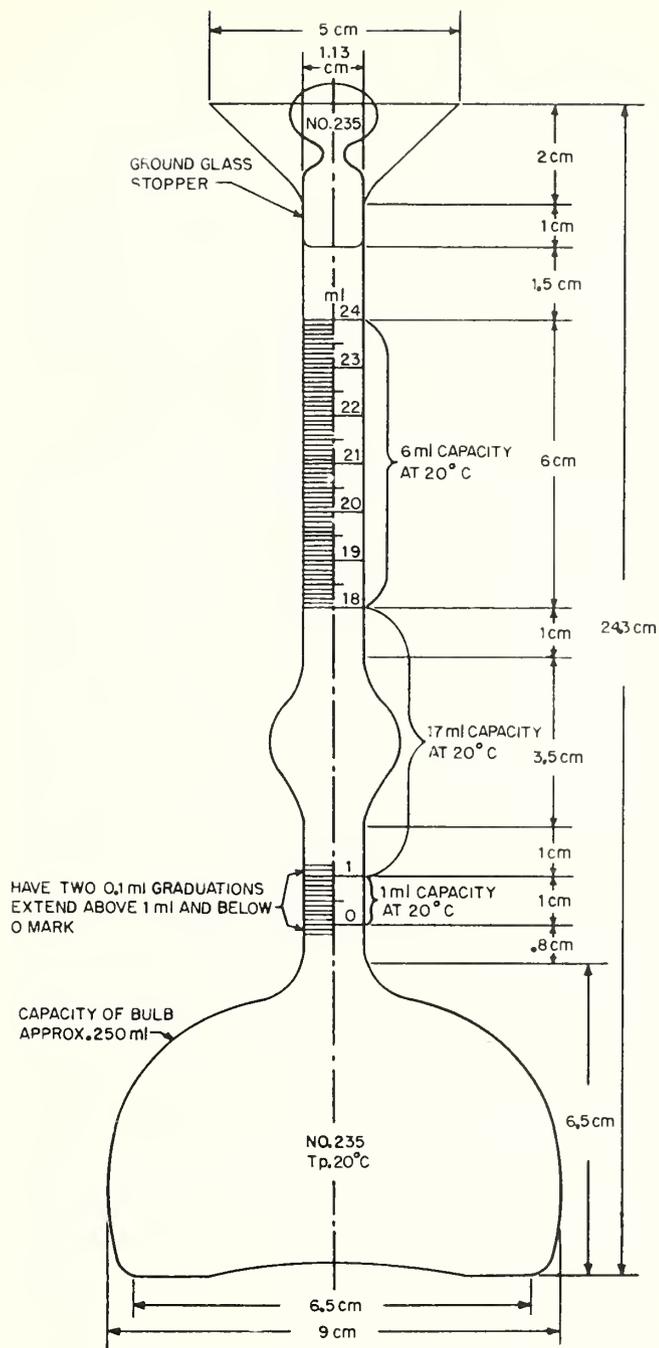


FIGURE 4. Specific-gravity flask.

c. Sugar-Testing Flasks (Bates)

The flask shall have a height of 130 mm, and the neck shall be 70 mm in length; a tolerance of ± 5 mm is allowed for each. The internal diameter shall be not less than 11.5 mm and not more than 12.5 mm. The upper end of the neck shall be flared, and the graduation mark shall be not less than 30 mm from the upper end and 15 mm from the lower end of the neck. The flask shall be standardized to contain 100 ml at 20°C with a tolerance of 0.08 ml.

- Red cell pipets—nominal ratio 100:1,
 V/v_1 must be not less than 95 and not more than 105
 V/v_2 must be not less than 190 and not more than 210.
- White cell pipets—nominal ratio 10:1,
 V/v_1 must be not less than 9.65 and not more than 10.35
 V/v_2 must be not less than 19.30 and not more than 20.70.

In an effort to give more exact information as to accuracy, certain manufacturers have marked dilution pipets with a so-called "correction factor" This is usually written simply "+2", "-1", etc. Because of the possibility that such a correction will not be applied as the manufacturer intended, and because mass-production test methods cannot always be relied upon to fix the corrections within 1 percent, the Bureau does not recommend this practice. In those cases where greater accuracy is required than is provided by the application of the prescribed tolerances ($\pm 5\%$ for red cell pipets and $\pm 3.5\%$ for white cell pipets), it is suggested that the purchaser specify the desired limits of error. This would provide the needed accuracy without the application of corrections.

The Bureau does not accept for test pipets bearing a "correction factor" except in very special cases and only in small quantities.

f. Sahli Hemoglobin Pipets

The capacity tolerance for Sahli pipets is ± 2 percent of the nominal volume.

g. Immunity Unit Cylinders and Pipets

These instruments were designed by the Hygienic Laboratory of the U. S. Public Health Service (USPHS) for use in the measurement of toxin and serum dilutions. Specifications may be found in USPHS Hygienic Laboratory Bulletin 21. The instruments are made to conform in general with the specifications contained in this Circular, but with certain modifications necessary to adapt them to the specific application.

The pipets are of three types: (1) Capacity or "wash-out" pipets, calibrated to contain the nominal volume; (2) delivery pipets, calibrated to deliver the nominal volume when the residue in the tip after free outflow has ceased is blown out and added to the initial delivery; (3) graduated delivery pipets, called Ehrlich pipets, calibrated to deliver the contents in the same manner as transfer pipets.

Tolerances for the first two types of pipets named are the same for ordinary transfer pipets; for the Ehrlich delivery pipets, the tolerance is 0.01 ml. For the cylinders, the tolerances are: For capacities less than and including 50 ml—0.05 ml; for capacities over 50 ml and including 100 ml—0.08 ml.

h. Special-Purpose Volumetric Flasks

Certain types of special-purpose volumetric flasks such as the Engler viscosity flask and the Kohlrausch and Stift sugar flasks, while not in conformity with all the special requirements for volumetric flasks listed in section 2.3.a because of their specific applications, will be calibrated and marked with the precision stamp if they are in satisfactory conformity with the general specifications in section 2.2 and the errors in capacity do not exceed the tolerances for volumetric flasks given in section 2.5.

i. Other Special Apparatus

Other types of special-purpose instruments may be calibrated and marked with the precision stamp if they conform with the general specifications (section 2.2) and the errors in capacity do not exceed the applicable tolerances in section 2.5.

Permanent identification numbers are required on all glass volumetric apparatus submitted for certification.

Instruments obviously not capable of precision measurements, as for example, centrifuge tubes, are not accepted because the time expended in their calibration is not warranted by the degree of accuracy required in their use.

2.5. Tolerances

a. Flasks

The capacity tolerances for flasks are given in table 9.

TABLE 9. Capacity tolerances for volumetric flasks

Capacity (in milliliters) less than and including—	Limit of error	
	If to contain—	If to deliver—
1.....	ml 0.01	ml
3.....	.015
5.....	.02
10.....	.02	0.04
25.....	.03	.05
50.....	.05	.10
100.....	.08	.15
200.....	.10	.20
300.....	.12	.25
500.....	.15	.30
1,000.....	.30	.50
2,000.....	.50	1.00
Above 2,000.....	1 part in 4,000	1 part in 2,000

b. Transfer Pipets

The capacity tolerances for transfer pipets are given in table 10. These tolerances are applicable to pipets of similar design made to contain the nominal volumes.

c. Burets and Measuring Pipets

The capacity tolerances for burets and measuring pipets are given in table 11.

TABLE 10. Capacity tolerances for transfer pipets

Capacity (in milliliters) less than and including—	Limit of error
	<i>ml</i>
2.....	0.006
5.....	.01
10.....	.02
30.....	.03
50.....	.05
100.....	.08
200.....	.10

TABLE 11. Capacity tolerances for burets and measuring pipets

Capacity (in milliliters) of total graduated portion less than and including—	Limit of error of total or partial capacity	
	Burets	Measuring pipets
	<i>ml</i>	<i>ml</i>
2.....	0.01	0.01
5.....	.02	.02
10.....	.03	.03
30.....	.05	.05
50.....	.08	.08
100.....	.10	.15

d. Cylindrical Graduates

The capacity tolerances for cylindrical graduates are given in table 12.

e. Delivery Time

The actual delivery time of any instrument must be within the limits prescribed in section 2.3, and the error in the marked delivery time must not exceed the limits given in table 13.

3. Special Rules for Manipulation

These rules indicate the essential points in the manipulation of volumetric apparatus which must be observed in order that the conditions necessary to obtain accurate measurements may be reproduced.

3.1. Test Liquid

Apparatus will ordinarily be calibrated with distilled water, and the capacity determined will therefore be the volume of water contained or delivered by an instrument at its standard temperature.

Certain special types of apparatus, such as pipets having a capacity of 0.01 ml or less, are more accurately calibrated with mercury. Instruments designed to deliver their contents should always be calibrated with water.

3.2. Method of Reading

a. Using Water or Other Wetting Liquid

In all apparatus where the volume is limited by a meniscus, the reading or setting is made on the

TABLE 12. Capacity tolerances for cylindrical graduates

Metric units				Customary units			
Capacity of total graduated portion less than and including—	Maximum diameter	Limit of error of total or partial capacity		Capacity of total graduated portion less than and including—	Maximum diameter	Limit of error of total or partial capacity	
		If to contain—	If to deliver—			If to contain—	If to deliver—
<i>ml</i>	<i>mm</i>	<i>ml</i>	<i>ml</i>	<i>Minims</i>	<i>in.</i>	<i>Minims</i>	<i>Minims</i>
5.....	11	0.05	0.06	60.....	3/8	0.6	0.7
10.....	14	.08	.10	120.....	1/2	1.0	1.3
25.....	19	.14	.18				
50.....	23	.20	.26	1 fl oz	in.	fl oz	fl oz
100.....	29	.35	.40	1 (8 fl dr)	3/4	0.005	0.006
250.....	40	.65	.80	2.....	1	.008	.010
500.....	50	1.1	1.3	4.....	1 1/4	.014	.017
1,000.....	63	2.0	2.5	8.....	1 1/2	.021	.027
2,000.....	80	3.5	5.0	16.....	1 3/4	.035	.045
				32.....	2 1/8	.060	.080

TABLE 13. Limits of error in marked delivery time

Delivery time (in seconds) less than and including—	Limit of error in marked delivery time
	<i>sec</i>
15.....	3
20.....	4
30.....	6
50.....	8
100.....	15
200.....	20

f. Special Apparatus

Capacity tolerances for the special purpose instruments described in section 2.4 are included in the appropriate parts of that section.

lowest point of the meniscus. In order that the lowest point may be observed, it is necessary to place a shade of some dark material immediately below the meniscus, which renders the profile of the meniscus dark and clearly visible against a light background. A convenient device for this purpose is a collar-shaped section of thick black rubber tubing, cut open at one side and of such size as to clasp the tube firmly.

The position of the lowest point of the meniscus with reference to the graduation line is such that it is in the plane of the middle of the graduation line. This position of the meniscus is obtained by making the setting in the center of the ellipse formed by the graduation line on the front and the back of the tube as observed by having the eye slightly below the plane of the graduation line. This is illustrated in figure 5. The setting is accurate if, as the eye is raised and the ellipse narrows, the lowest point of the meniscus remains midway between the front and rear portions of the graduation line. By this method it is possible to observe the approach of the meniscus from either above or below the line to its proper setting.

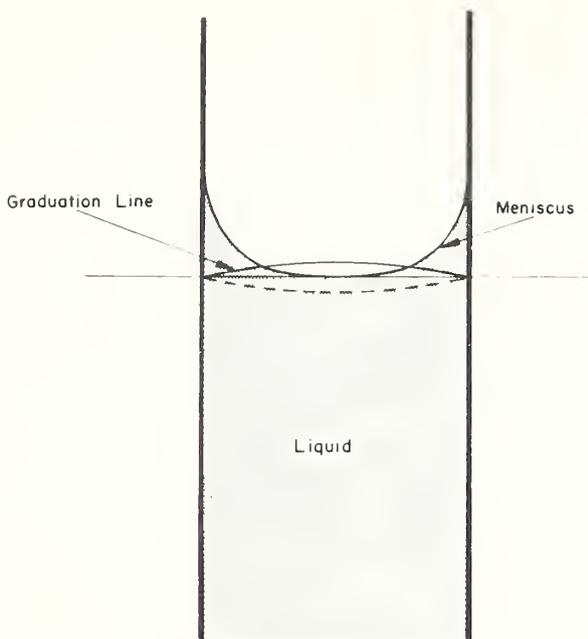


FIGURE 5. Method of setting water meniscus.

b. Using Mercury

In setting a mercury meniscus on a graduation line, the instrument must be tapped sharply in order that the meniscus may assume a normal shape. The *highest* point of the meniscus is set on the middle of the graduation line by employing the principles outlined for water, but observing from *above* the line.

3.3. Cleanliness of Apparatus

Apparatus must be sufficiently clean to permit uniform wetting of the surface and to minimize contamination of the liquid surface.

Of the various cleaning agents in common use, the Bureau prefers fuming sulfuric acid or a chromic-sulfuric acid mixture. For removal of oil and grease, an organic solvent is used and the cleaning completed with one of the above agents.

When drying is required before calibration, it is done with unheated compressed air which is cleaned and dried by passing through concentrated sulfuric acid and a drying compound such as calcium chloride.

3.4. Flasks and Cylinders

In filling flasks, the entire interior of the flask below the stopper will be wetted; in the case of cylinders, the liquid is allowed to flow down one side only. After filling to a point slightly below the graduation line, these instruments are allowed to drain for about 2 min. They are then placed below a buret having a long delivery tube and a bent tip, and the filling is completed by discharging water from the buret against the wall of the flask or cylinder about 1 cm above the graduation line, and rotating the receiving vessel to re-wet the wall uniformly.

Flasks and cylinders which are to be used to

deliver are filled approximately to the test point, then emptied by gradually inclining them, avoiding as much as possible agitation of the contents and re-wetting of the walls. Allow half a minute for emptying. When the continuous outflow has ceased, the vessel should be nearly vertical and should be held in this position for another half-minute. The adhering drop is removed by contact with the wetted wall of the receiving vessel. The rest of the calibration is performed in the same manner as in calibrating to contain.

In calibrating a flask or cylinder to contain it is only necessary, after cleaning and drying the apparatus, to weigh it empty, fill it accurately to the graduation mark, and again weigh. Table 16 or table 17 may be used to determine the capacity at 20° C from the weight of the water contained at a known temperature.

In case flasks are to be tested in large numbers, a volumetric method may be used to advantage. The Bureau uses a series of volumetric standard pipets (illustrated in fig. 6) each having a capacity slightly less than that of the flask it is intended to test. The water delivered from the appropriate standard pipet fills the flask nearly to the graduation mark. After the 2-min drainage period, the filling is completed from an accurately calibrated buret. The capacity of the flask is then found from the known volume delivered by the standard pipet and the additional volume delivered by the buret. The standard pipets and burets are themselves calibrated by weighing the water delivered.

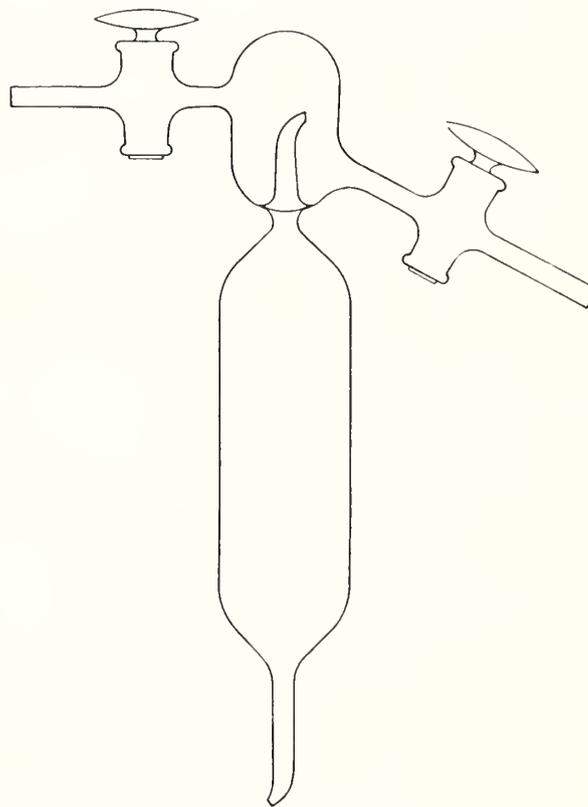


FIGURE 6. Standard pipet for flask calibrations.

a. Conical Graduates

The conical graduate is not classed as a precision volumetric measure, and is not recommended for use in checking the accuracy of other volumetric apparatus. The Bureau does not accept these graduates for calibration except when requested by a State weights and measures official.

Conical graduates are calibrated in the same manner as cylindrical graduates, except that in calibrating "to deliver", the emptying time is reduced from 30 sec to 10 sec and the drainage time (after emptying) is also reduced to 10 sec, following customary methods of use.

3.5. Burets

Burets are calibrated in a vertical position. After cleaning, they are filled with distilled water to about 1 cm above the zero line. They are allowed to stand for about 30 min to check for leakage, not only at the tip but also around the stopcock plug. The water is then allowed to discharge with unrestricted outflow and the delivery time is noted and recorded. This flushing serves to equalize the temperatures of apparatus and water. The water temperature is taken on the next filling by inserting a thermometer in the buret or, if it is not large enough to receive a thermometer, in a plain glass tube of larger diameter mounted near the buret.

If the temperature was taken in the buret, the thermometer is then removed and enough water added to raise the level to about 1 cm above the zero line. Any water on the outside of the tip is removed with absorbent paper. The setting of the meniscus on the zero line is made by allowing the water to run out slowly. After the setting is made, any excess water on the tip is removed by touching the tip with the wetted wall of a beaker or other vessel. The very small quantity of water which remains on the extreme end, or ground portion, of the tip is not removed, as the same quantity of water will remain after delivery into the weighing flask.

Delivery of the water into the weighing flask is made with the buret tip in contact with the inside of the flask neck, the flask being inclined at an angle of about 20°. When delivery has been completed, the weighing flask is immediately removed horizontally from contact with the instrument being calibrated. The flask is then stoppered and weighed. The buret and thermometer tube (if used) are refilled preparatory to measuring the next interval.

Buret stopcocks should be completely open during delivery until the water level is only a few millimeters above the test point, when the discharge must be slowed in order to make an accurate setting.

In making the weighings it is both convenient and accurate to use the method of substitution. By this method a constant tare is kept on one pan of the balance, while on the other pan is placed the object to be weighed and with it suffi-

cient weights to secure equilibrium. In testing volumetric apparatus, the receiving flask is first weighed empty and then again after having delivered into it the water from the interval under test. The difference between the two weights is the weight of the water delivered.

Suppose, for example, that with a certain tare on the right-hand pan and the empty flask on the left a load of 151.276 g is required in addition to the flask to restore equilibrium, and that after the water from the 10-ml interval of the buret under test is delivered into the flask, only 141.310 g are required, and that the water is at a temperature of 23.4°C. The weight of the water added is then $151.276 - 141.310 = 9.966$ g. From table 16 (appendix), it is seen that to determine the capacity of a 10-ml soft glass vessel at 20°C from the weight of water delivered at 23.4°C, 0.035 must be added to the weight; that is, the capacity of the 10-ml interval of the buret under test at 20°C is $9.966 + 0.035 = 10.001$ ml.

3.6. Measuring and Transfer Pipets

Measuring and transfer pipets are calibrated in much the same manner as burets, except that the tip must be in contact with the wet wall of a beaker or other vessel when the setting is made on the zero line of a measuring pipet or the capacity mark of a transfer pipet. Also, the delivery time of a transfer pipet is measured with the tip in contact with the vessel.

In calibrating transfer pipets, the water surface must be observed closely as it approaches the tip to make certain that outflow has ceased before the weighing flask is removed. On the other hand, if the flask removal is unduly delayed, after-drainage will affect the result.

The water remaining in the tip of a pipet is not blown out unless, as in the case of certain types of special-purpose pipets, there is a wide band or two narrower bands permanently marked near the top of the instrument.

The water temperature is measured in the beaker from which the pipet is filled (by suction).

3.7. Special Apparatus

The flasks and cylinders mentioned in section 2.4 are calibrated in the manner described in section 3.4, except that the inside of the neck is wetted for a distance of only about 1 cm or less above the test point.

A specific gravity flask is calibrated at the 1, 18, and 24 ml graduations.

Capacity pipets made *to contain* the nominal volume are calibrated by weighing before and after filling.

In calibrating "blow-out" pipets, the water remaining in the tip after free outflow has ceased is blown out, without waiting for drainage, and added to the quantity delivered. The blow-out is accomplished by a single strong puff, with the pipet tip in contact with the neck of the weighing flask.

4. Tests Performed by the Bureau

4.1. Nature of Tests

Apparatus submitted for test is first examined as to its conformity with the specifications concerning quality and workmanship, design, markings, outflow time, etc.

If the apparatus meets these requirements, it is calibrated either to ascertain whether the capacity is correct within the prescribed limits of error or to determine the correction for use in precise measurements.

4.2. Precision Stamp

If the results of the preliminary examination indicate a satisfactory conformity with the specifications, and the error in capacity is within the prescribed tolerance, the official precision stamp, consisting of the letters NBS and the year date, surrounded by a circle, is etched on the instrument as shown below:



4.3. Certificates and Reports of Capacity

Burets will be calibrated for at least five intervals, and if found to conform with the specified requirements will, in addition to the precision stamp, be marked with an NBS identification number as shown below:

NBS No. 1234
1958

A certificate will be issued giving the volumes delivered from the intervals tested.

5. Directions for Submitting Apparatus for Test

5.1. Application for Test

The request for test should be made in writing and should include a complete list of the apparatus and a statement of the nature of the test desired. Representatives of state institutions entitled to tests free of charge must make application in writing for each test in order to avail themselves of the privilege.

Unless otherwise requested, burets will be examined and the capacity of five intervals tested. If in satisfactory conformity with the requirements, the results of the test are certified. If more than five intervals are to be tested, the request must so state.

Patrons should always examine apparatus carefully before submitting it for test to ascertain if it complies with the construction specifications. Delay and cost of transportation on apparatus not entitled to verification will thus be avoided.

Only in special cases will certificates of capacity be issued for apparatus other than burets. When the measurements depending upon the use of the volumetric glassware are required to be so precise that a statement to the effect that the apparatus is within the tolerances prescribed in this Circular does not give sufficiently exact information, certificates may be issued. The request for certificates, when required, should be made at the time the apparatus is submitted for test.

Reports of capacity may be issued under the following circumstances:

(a) For burets when the error in capacity is greater than, but not more than twice, the tolerance.

(b) When certificates are requested for other apparatus, and the error in capacity exceeds, but is not more than twice, the prescribed tolerance.

(c) For special types of volumetric apparatus not covered by the specifications in this Circular.

When a report of capacity is issued, a number is applied as shown above, but the precision stamp is omitted.

4.4. Special Tests

The Bureau will gladly cooperate with scientific investigators, manufacturers of apparatus, and others in the calibration of precision volumetric apparatus not covered by the specifications in this Circular, as far as the regular work of the Bureau will permit. Tests of instruments not included in the current fee schedules should be arranged for by correspondence before shipment of the apparatus; the application should state fully the purpose for which the apparatus is to be used, the need for the test, and the precision desired.

The Bureau does not sell volumetric apparatus. It may be purchased from manufacturers, importers, or jobbers, and submitted to the Bureau for test.

Purchasers of apparatus to be submitted to the Bureau for test should so specify to the dealer in order to avoid unnecessary delays and misunderstandings.

5.2. Shipping Directions

The apparatus should be securely packed in cases or packages which will not be broken in transportation and which may be used in returning the tested material to the owner. Great care should be taken in packing. Clean, dry excelsior is a suitable packing material in most cases. Each instrument should also be wrapped in strong paper or other covering to prevent dust and excelsior from getting into it.

Transportation charges are payable by the party requesting the test. The charges for shipment to the Bureau must be prepaid; and, unless otherwise arranged, articles will be returned or forwarded by express "collect".

After the material is received at the Bureau, the shipper will be notified of the test number assigned. This number should be mentioned in any correspondence pertaining to the test.

5.3. Breakage

A considerable number of pieces of glassware are received broken, either on account of improper packing or rough treatment in transportation, or both. Some are broken in return shipment. A small percentage is broken in the testing laboratory.

There is no legal way in which the Bureau can make reimbursement for breakages of this kind. It is therefore a matter of necessity, and not of choice, that the Bureau makes those who send apparatus for test assume all the risks involved.

Under the circumstances, all that the Bureau can do is to make every effort to reduce such breakage to the absolute minimum.

5.4. Address

Articles and communications should be addressed, "National Bureau of Standards, Washington 25, D.C."; delays incident to other forms of addresses will thus be avoided. Articles delivered personally or by messenger should be left at the receiving office of the Bureau, and should be accompanied by a written request for test.

5.5. Remittances

Payment of test fee should be made promptly upon receipt of bill. Remittances may be made by money order or check drawn to the order of the "National Bureau of Standards".

Copies of the current Test Fee Schedules may be obtained from the Bureau upon request.

6. Appendix

Tables 14 and 16 are reprints of tables 38 and 43 from NBS Circular 19, 6th edition, Standard Density and Volumetric Tables. They are included in this Circular for the convenience of users of volumetric glassware. Tables 15 and 17 have been added because of the increasing popularity of borosilicate glass apparatus.

6.1. Temperature Corrections for Volumetric Solutions

Table 14 gives the correction to various observed volumes of water, measured at the designated temperatures, to give the volume at the standard

temperature, 20° C. Conversely, by subtracting the corrections from the volume desired at 20° C, the volume that must be measured at the designated temperatures in order to give the desired volume at 20° C will be obtained. It is assumed that the volumes are measured in glass apparatus having a coefficient of cubical expansion of 0.000-025/° C. The table is applicable to dilute aqueous solutions having the same coefficient of expansion as water.

Table 15 gives temperature corrections for water, when measured in borosilicate glass apparatus having a coefficient of cubical expansion of 0.000010/° C.

TABLE 14. *Temperature corrections for water*

Measured in soft glass apparatus having a coefficient of cubical expansion of 0.000025/°C

Temperature of measurement	Capacity of apparatus in milliliters at 20° C						
	2,000	1,000	500	400	300	250	150
	Correction in milliliters to give volume of water at 20° C						
° C							
15.....	+1.54	+0.77	+0.38	+0.31	+0.23	+0.19	+0.12
16.....	+1.28	+0.64	+0.32	+0.26	+0.19	+0.16	+0.10
17.....	+0.99	+0.50	+0.25	+0.20	+0.15	+0.12	+0.07
18.....	+0.68	+0.34	+0.17	+0.14	+0.10	+0.08	+0.05
19.....	+0.35	+0.18	+0.09	+0.07	+0.05	+0.04	+0.03
21.....	-0.37	-0.18	-0.09	-0.07	-0.06	-0.05	-0.03
22.....	-0.77	-0.38	-0.19	-0.15	-0.12	-0.10	-0.06
23.....	-1.18	-0.59	-0.30	-0.24	-0.18	-0.15	-0.09
24.....	-1.61	-0.81	-0.40	-0.32	-0.24	-0.20	-0.12
25.....	-2.07	-1.03	-0.52	-0.41	-0.31	-0.26	-0.15
26.....	-2.54	-1.27	-0.64	-0.51	-0.38	-0.32	-0.19
27.....	-3.03	-1.52	-0.76	-0.61	-0.46	-0.38	-0.23
28.....	-3.55	-1.77	-0.89	-0.71	-0.53	-0.44	-0.27
29.....	-4.08	-2.04	-1.02	-0.82	-0.61	-0.51	-0.31
30.....	-4.62	-2.31	-1.16	-0.92	-0.69	-0.58	-0.35

TABLE 15. *Temperature corrections for water*

Measured in borosilicate glass apparatus having a coefficient of cubical expansion of 0.000010/°C

Temperature of measurement	Capacity of apparatus in milliliters at 20° C						
	2,000	1,000	500	400	300	250	150
	Correction in milliliters to give volume of water at 20° C						
° C							
15.....	+1.69	+0.84	+0.42	+0.34	+0.25	+0.21	+0.13
16.....	+1.39	+0.70	+0.35	+0.28	+0.21	+0.17	+0.10
17.....	+1.08	+0.54	+0.27	+0.22	+0.16	+0.13	+0.08
18.....	+0.74	+0.37	+0.19	+0.15	+0.11	+0.09	+0.06
19.....	+0.38	+0.19	+0.10	+0.08	+0.06	+0.05	+0.03
21.....	-0.40	-0.20	-0.10	-0.08	-0.06	-0.05	-0.03
22.....	-0.83	-0.41	-0.21	-0.17	-0.12	-0.10	-0.06
23.....	-1.27	-0.64	-0.32	-0.25	-0.19	-0.16	-0.10
24.....	-1.73	-0.87	-0.43	-0.35	-0.26	-0.22	-0.13
25.....	-2.22	-1.11	-0.55	-0.44	-0.33	-0.28	-0.17
26.....	-2.72	-1.36	-0.68	-0.54	-0.41	-0.34	-0.20
27.....	-3.24	-1.62	-0.81	-0.65	-0.49	-0.41	-0.24
28.....	-3.79	-1.89	-0.95	-0.76	-0.57	-0.47	-0.28
29.....	-4.34	-2.17	-1.09	-0.87	-0.65	-0.54	-0.33
30.....	-4.92	-2.46	-1.23	-0.98	-0.74	-0.62	-0.37

In using the above tables to correct the volume of certain standard solutions to 20° C, more accurate results will be obtained if the numerical values of the corrections are increased by the percentages given below:

Solution	Normality		
	N	N/2	N/10
HNO ₃	50	25	6
H ₂ SO ₄	45	25	5
NaOH.....	40	25	5
KOH.....	40	20	4

6.2. Tables of Corrections for Determining the True Capacities of Glass Vessels From the Weight of Water in Air

Table 16 gives, for a nominal capacity of 100 ml and observed temperatures from 15° C to 32.9° C, the amounts to be added to the apparent weight in grams (in air against brass weights) of the water contained in or delivered by a glass vessel to give the capacity in milliliters at 20° C. It is calculated on the following data assumed as approximating ordinary conditions: Observed barometric pressure—760 mm; relative humidity—50 percent; coefficient of expansion of glass—0.000025/° C.

Example of use of table. Determination of capacity of glass volumetric flask marked "To contain 100 ml at 20° C".

Apparent weight of water at the observed temperature, 24.5° C.....	99.615 g
From table 16, correction.....	+0.372
Actual capacity at 20° C.....	99.987 ml

For capacities other than 100 ml, the corrections given must be multiplied by the appropriate

factor. For example, the correction for 500 ml would be five times the correction for 100 ml.

For borosilicate glass having a coefficient of cubical expansion of approximately 0.000010/° C, Table 17 should be used.

6.3. Change in Capacity Caused by Change of Temperature

Having determined the capacity at 20° C, if it is desired to know what the capacity of the same vessel will be at another temperature, the following formula may be used:

$$V_t = V_{20}[1 + \alpha(t - 20)],$$

where

V_t = capacity at t ° C,
 V_{20} = capacity at 20° C,
 α = coefficient of cubical expansion of material of which the instrument is made. (For soft glass 0.000025/° C)

This formula is applicable to instruments or measures made of any material of which the coefficient of cubical expansion is known. A few materials commonly used in volumetric apparatus and their approximate coefficients are:

	Coefficient of cubical expansion ⁴ per deg C
Soft glass.....	0.000025
Pyrex or KG-33.....	.000010
Copper.....	.000050
Brass.....	.000054
Steel.....	.000035
Stainless steel.....	.000031

⁴ Coefficients shown are averages, but are sufficiently accurate for volumetric determinations.

TABLE 16. Table of corrections for determining the true capacities of glass vessels from the weight of water in air (Soft glass, coefficient of cubical expansion 0.000025/° C)

Temperature	Tenths of degrees									
	0	1	2	3	4	5	6	7	8	9
15.....	0.207	0.208	0.210	0.211	0.212	0.213	0.215	0.216	0.217	0.219
16.....	.220	.221	.223	.224	.225	.227	.228	.230	.231	.232
17.....	.234	.235	.237	.238	.240	.241	.243	.244	.246	.247
18.....	.249	.250	.252	.253	.255	.257	.258	.260	.261	.263
19.....	.265	.266	.268	.270	.272	.273	.275	.277	.278	.280
20.....	.282	.284	.285	.287	.289	.291	.293	.294	.296	.298
21.....	.300	.302	.304	.306	.308	.310	.312	.314	.315	.317
22.....	.319	.321	.323	.325	.327	.329	.331	.333	.336	.338
23.....	.340	.342	.344	.346	.348	.350	.352	.354	.357	.359
24.....	.361	.363	.365	.368	.370	.372	.374	.376	.379	.381
25.....	.383	.386	.388	.390	.392	.395	.397	.399	.402	.404
26.....	.406	.409	.411	.414	.416	.418	.421	.423	.426	.428
27.....	.431	.433	.436	.438	.440	.443	.446	.448	.451	.453
28.....	.456	.458	.461	.463	.466	.469	.471	.474	.476	.479
29.....	.482	.484	.487	.490	.492	.495	.498	.501	.503	.506
30.....	.509	.511	.514	.517	.520	.522	.525	.528	.531	.534
31.....	.536	.539	.542	.545	.548	.551	.554	.556	.559	.562
32.....	.565	.568	.571	.574	.577	.580	.583	.586	.589	.592

TABLE 17. *Table of corrections for determining the true capacities of glass vessels from the weight of water in air*

(Borosilicate glass, coefficient of cubical expansion 0.00010/° C)

Indicated capacity 100 ml

Temperature ° C	Tenths of degrees									
	0	1	2	3	4	5	6	7	8	9
15 -----	0.200	0.201	0.202	0.204	0.205	0.207	0.208	0.210	0.211	0.212
16 -----	.214	.215	.217	.218	.220	.222	.223	.225	.226	.228
17 -----	.229	.231	.232	.234	.236	.237	.239	.241	.242	.244
18 -----	.246	.247	.249	.251	.253	.254	.256	.258	.260	.261
19 -----	.263	.265	.267	.269	.271	.272	.274	.276	.278	.280
20 -----	.282	.284	.286	.288	.290	.292	.294	.296	.298	.300
21 -----	.302	.304	.306	.308	.310	.312	.314	.316	.318	.320
22 -----	.322	.324	.327	.329	.331	.333	.335	.338	.340	.342
23 -----	.344	.346	.349	.351	.353	.355	.358	.360	.362	.365
24 -----	.367	.369	.372	.374	.376	.379	.381	.383	.386	.388
25 -----	.391	.393	.396	.398	.400	.403	.405	.408	.410	.413
26 -----	.415	.418	.420	.423	.426	.428	.431	.433	.436	.438
27 -----	.441	.444	.446	.449	.452	.454	.457	.460	.462	.465
28 -----	.468	.470	.473	.476	.479	.481	.484	.487	.490	.492
29 -----	.495	.498	.501	.504	.506	.509	.512	.515	.518	.521
30 -----	.524	.526	.529	.532	.535	.538	.541	.544	.547	.550
31 -----	.553	.556	.559	.562	.565	.568	.571	.574	.577	.580
32 -----	.583	.586	.589	.592	.595	.598	.602	.605	.608	.611

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Testing a Quick-Weighing Balance

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The increasing use of quick-weighing balances necessitated the development of test procedures suited to such balances. Two tests have been developed, one for determining the linearity of the direct-reading scale, the other for calibrating the dial-operated weights. Estimates of the performance of the balance are obtained from each test. The computation form for the second test provides for the rapid and systematic least squares adjustment of the observed corrections of the weights. The test procedure, which is presented through the use of a numerical example and the discussion of statistical tests and estimates, provides for the calibration of the dial-operated weights and the estimation of the performance of a quick-weighing balance.

HOW good are modern quick-weighing balances? How much of the accuracy is sacrificed for the convenience and speed of a quick-weighing attachment? Such a device as a rider, chain, projected direct-reading scale, or set of built-in weights may contribute not only to the convenience or speed of operation of the balance but also to the variability of the balance. The test of a balance with projected direct-reading scale (capacity 100 mg.) and a set of built-in weights (capacity 99.9 grams) for a total capacity of 100 grams is considered in this paper. The rider, chain, and other quick-weighing devices will be considered in another paper. The tests described are designed specifically for the 100-gram Mettler Gram-atic balance, but could be adapted to other quick-weighing balances of similar design by taking into account the particular arrangement of the built-in weights. These special tests for the Mettler balance were devised because the built-in weights of this balance cannot be easily removed for testing and hence must be tested along with the balance.

DESCRIPTION OF BALANCE

Only those features of the balance related to the tests of the quick-weighing devices are described here. Built into the Gram-atic balance are three sets of dial-controlled weights arranged in decades, each set controlled by a separate dial. The "tenths" dial controls four weights: one each of nominal values 0.5 and 0.2

gram, and two of nominal value 0.1 gram. The weights used for each dial setting are shown in the following table, where the two 0.1-gram weights are distinguished by subscripts.

Dial Setting	Combination of Weights Used
0.1	(0.1) ₁
0.2	(0.2)
0.3	(0.2) + (0.1) ₁
0.4	(0.2) + (0.1) ₁ + (0.1) ₂
0.5	(0.5)
0.6	(0.5) + (0.1) ₁
0.7	(0.5) + (0.2)
0.8	(0.5) + (0.2) + (0.1) ₁
0.9	(0.5) + (0.2) + (0.1) ₁ + (0.1) ₂

Similarly, the "units" dial controls the four weights: 5 grams, 2 grams, 1 gram₁, and 1 gram₂. The "tens" dial is also similar, except that the weights come in pairs: (25 + 25 grams), (10 + 10 grams), (5 + 5 grams)₁, and (5 + 5 grams)₂. This is because it is necessary to balance the larger weights on the weight hanger. Thus the 50-gram dial setting removes two 25-gram weights symmetrically located on the hanger. Since, however, the two weights of a pair are always used together, they may be considered as one weight, and therefore the tens dial may be treated in the same manner as the units and tenths dials.

A fourth dial controls a 50-mg. weight used in differential weighing which, briefly, is a weighing performed within the range of the optical scale—i.e., without changing the dial settings. When the method of differential weighing is used, errors in the values of the dial-controlled weights do not enter into the results of a weighing. Only the variability of the balance and any error in the adjustment of the sensitivity will affect the results.

The balance has only one pan and stirrup and two knife-edges. A counterpoise on the beam replaces the second pan and stirrup and the third knife-edge. This feature eliminates the variability which would arise from the third knife-edge.

The dial-controlled weights normally hang from the weight hanger which is attached to the stirrup. All weighing is by substitution—that is, with the unknown on the pan of the balance the dial-controlled weights are removed by the dial controls until equilibrium is achieved. The total load on the stirrup is always 99.9 grams (plus the weight of pan and weight hanger and the amount indicated by the projection scale and the 50-mg. weight).

Because of the high magnification of the optical system, the balance is particularly sensitive to tilt. The balance should therefore be mounted on a sturdy support.

Table I. Determination of Correction for 0.1-Gram Dial Weight and Determination of Sensibility Reciprocal

Step	Operation	Observation, Mg.	Corr. for Std. ^a , Mg.	Computations	
				For 0.1-g. wt.	For 100 divisions
1	Adjust zero and read	a_1
2	Place a 100-mg. standard on pan, set dial at 0.1 g.; read	a_2	s_1	$s_1 + a_1 - a_2 = x_1$...
3	With standard still on pan, set dial at 0.0 g.; read	a_3	s_1	...	$s_1 + a_3 - a_2 = y_1$
4	Remove standard from pan and make zero reading (but do not readjust zero)	a_4
5	Arrest balance; reset zero (arbitrarily, need not read exactly zero); read	a_5
6	Place a different 100-mg. standard on pan; set dial at 0.1 g.; read	a_6	s_2	$s_2 + a_5 - a_6 = x_2$...
7	With the second standard still on pan, set dial at 0.0 g.; read	a_7	s_2	...	$s_2 + a_7 - a_6 = y_2$
8	Remove standard from pan and make zero reading (do not readjust zero)	a_8
9	Repeat steps 1 through 8, using same standards, until a total of five sets of observations are obtained	a_9
		b_1
		b_2	s_1	$s_1 + b_1 - b_2 = x_3$...
		b_3	s_1	...	$s_1 + b_4 - b_3 = y_3$
		b_4
		b_5
		b_6	s_2	$s_2 + b_5 - b_6 = x_4$...
		b_7	s_2	...	$s_2 + b_8 - b_7 = y_4$
		b_8
		etc.
Average value of correction for 0.1-g. weight, $= \bar{x}$				\bar{x}	\bar{y}
Average value of correction for 100 divisions, $= \bar{y}$					

^a Correction for standard is quantity which must be added to nominal value to give actual mass of the standard.

SENSITIVITY

The manufacturer recommends that the 0.1-gram dial weight be used as the standard for adjusting the sensitivity of the balance. If this weight is in error, then the sensitivity will be incorrectly adjusted and the optical scale will not be direct reading in milligrams. The first step in testing the balance after leveling it and making sure that it is otherwise in working order is to adjust the sensitivity by comparing full scale deflection against standard weights.

After the sensitivity is adjusted as closely as possible, the sensibility reciprocal, SR , may be accurately determined by repeated weighings against standards. The sensibility reciprocal is a measure of the sensitivity of the balance and is defined as the number of milligrams which must be added to the load on the balance pan to cause a one-division change in the rest point. At the same time that the sensibility reciprocal is determined it is convenient to determine the value of the 0.1-gram dial weight. The sequence of weighings shown in Table I may be used, in which all readings are estimated to 0.01 mg.

The correction for the 0.1-gram dial weight is given by \bar{x} . The sensibility reciprocal can be computed from the average value of the correction for 100 divisions, \bar{y} , by the following equation:

$$SR = \frac{100 \text{ mg.} + \bar{y} \text{ mg.}}{100 \text{ divisions}} = \left(1 + \frac{\bar{y}}{100}\right) \frac{\text{mg.}}{\text{division}}$$

If it is assumed that the values of the standards are known very much better than the balance reproduces its readings, then the standard deviation from their average of the ten values for the 0.1-gram dial weight may be used as a measure of the variability of the balance. (Weights having a National Bureau of Standards Class S certificate will meet this condition with most projection-reading balances. If better accuracy is required, a Class M set of weights may be used.) If $x_i = x_1, x_2, \dots, x_{10}$ are the ten values and \bar{x} is their average Table (I), then the standard deviation is computed by the formula:

$$\text{Standard deviation } (S_1) = \sqrt{\frac{\sum(x_i - \bar{x})^2}{9}} = \frac{1}{3} \sqrt{\sum(x_i - \bar{x})^2}$$

The standard deviation from their average of the ten values of the correction for 100 divisions may also be used as a measure of the variability of the balance at readings near full scale of the projected scale.

$$\text{Standard deviation } (S_2) = \sqrt{\frac{\sum(y_i - \bar{y})^2}{9}} = \frac{1}{3} \sqrt{\sum(y_i - \bar{y})^2}$$

If the variability of the balance when the balance is used near the zero end of the projected scale and when it is used near the other end is the same, then the data may be combined to form a

Table II. Computation Form for Balance Test and Calibration of Dial-Controlled Weights

Dial Setting	Obsvd. ^a	Computed	Residuals, Obs.-Comp. (Residuals) ²
1 = (1) ₁	$a =$		
2 = (2)	$b =$		
3 = (2) + (1) ₁	$c =$		
4 = (2) + (1) ₁ + (1) ₁	$d =$		
5 = (5)	$e =$		
6 = (5) + (1) ₁	$f =$		
7 = (5) + (2)	$g =$		
8 = (5) + (2) + (1) ₁	$h =$		
9 = (5) + (2) + (1) ₁ + (1) ₂	$i =$		
Sum of Squares =			
	A	B	C
	+ -	+ -	+ -
	-2 a	0 a	+2 a
	-2 b	+2 b	0 b
	-4 c	+2 c	+2 c
	-3 d	+1 d	+1 d
	+6 e	-2 e	-2 e
	+4 f	-2 f	0 f
	+4 g	0 g	-2 g
	+2 h	0 h	0 h
	+3 i	-1 i	-1 i
Check on Computations of A, B, C, and D	(5)	(2)	(1) ₁ (1) ₂
	+ $\frac{1}{19} A$	+10(5)	+10(5) -100(5)
	-3 a	+2B	+2C +4D
	-3 b	Sum	Sum
	-6 c	$\frac{1}{8}$ Sum	$\frac{1}{8}$ Sum
	-4 d		
	+10 e		
	+7 f		
	+7 g		
	+4 h		
	+6 i		
	=		
Checks on Computations (Add residuals as follows)			
$r_4 + r_9$			Sum
$r_2 + r_3 + r_7 + r_8$			$\frac{1}{15}$ Sum
$r_5 + r_6 + r_7 + r_6 + r_9$			$\frac{1}{15}$ Sum
$r_1 + r_2 + r_6 + r_8$			Standard deviation = $\sqrt{\frac{1}{15} \text{ Sum}}$
			Summary of Tests of Three Dials
			Dial Tenth Units Tens
			Sum of Squares

^a To the observed difference, zero reading minus standard reading, add correction for standard weight

single measure of the variability. The test to determine whether the two estimates of the variability, S_1 and S_2 , are estimates of the same thing (4) is as follows:

$$\left(\frac{S_1}{S_2}\right)^2 < 3.18 \quad \text{or} \quad \left(\frac{S_2}{S_1}\right)^2 < 3.18 \quad 9 \text{ times in } 10$$

where the larger value is used in the numerator. If the above test is satisfied, the data may be combined to form the single measure of variability:

$$\text{Standard deviation } (S_3) = \sqrt{\frac{\Sigma(x_i - \bar{x})^2 + \Sigma(y_i - \bar{y})^2}{9 + 9}}$$

DIAL-CONTROLLED WEIGHTS

The corrections for the dial-controlled weights may be determined by comparing each dial setting against suitable standards. This introduces errors which are due to the variability of the balance. It would be better if the dial weights could be removed from the balance and calibrated against standards on a more precise equal-arm balance. However, the weights cannot be removed from the Mettler balance with ease or without danger of putting the dial mechanism out of alignment. Hence, the following scheme has been devised.

The following weighings are made, in which all scale readings are estimated to 0.01 mg:

Load on Pan, G.	Observations		Corr. for Std., Mg.	Corrected Observations for Dial Wt., Mg.
	Dials, g.	Scale, mg.		
Set 0	00.0	a_0
	0.1	a_1	s_1	$a = s_1 + a_0 - a_1$
	0.2	b_1	s_2	$b = s_2 + b_0 - b_1$
	0	b_0
Reset 0	00.0	c_0
	0.3	c_1	s_3	$c = s_3 + c_0 - c_1$
	0.4	d_1	s_4	$d = s_4 + d_0 - d_1$
	0	d_0
Reset 0	00.0	e_0
	0.5	e_1	s_5	$e = s_5 + e_0 - e_1$
	0.6	f_1	s_6	$f = s_6 + f_0 - f_1$
	0	f_0
Reset 0	00.0	g_0
	0.7	g_1	s_7	$g = s_7 + g_0 - g_1$
	0.8	h_1	s_8	$h = s_8 + h_0 - h_1$
	0	h_0
Reset 0	00.0	i_0
	0.9	i_1	s_9	$i = s_9 + i_0 - i_1$
1	01.0	etc.	etc.	etc.
0	00.0			
Reset 0	00.0			
	2	02.0		
	3	03.0		
	0	00.0		
	etc.	etc.		

And so forth through the remainder of the units dial and through the tens dial. If the weighings are made in the order shown, and the corrected observations (a through i for each dial) are computed from adjacent zeros as shown, most of the effect of a uniform zero shift will be cancelled out of the final values for the weights. A zero shift will, however, increase the residuals as discussed below.

Nine values (a through i) are now known for as many combinations of the four weights 0.5, 0.2, 0.1₁, and 0.1₂ gram. Thus the observation equations are:

$$\begin{aligned} & (0.1)_1 & - 0.1 & = a \\ & (0.2) & - 0.2 & = b \\ & (0.2) + (0.1)_1 & - 0.3 & = c \\ & (0.2) + (0.1)_1 + (0.1)_2 & - 0.4 & = d \\ (0.5) & & - 0.5 & = e \\ (0.5) & + (0.1)_1 & - 0.6 & = f \\ (0.5) + (0.2) & & - 0.7 & = g \\ (0.5) + (0.2) + (0.1)_1 & & - 0.8 & = h \\ (0.5) + (0.2) + (0.1)_1 + (0.1)_2 & - 0.9 & = i \end{aligned}$$

It will be noted that there are more observation equations than unknowns. The least squares method of solution is appropriate (3). If the observation equations are given equal weight, the normal equations are:

$$\begin{aligned} 5(0.5) + 3(0.2) + 3(0.1)_1 + (0.1)_2 & = +e + f + g + h + i \\ 3(0.5) + 6(0.2) + 4(0.1)_1 + 2(0.1)_2 & = +b + c + d + e + f + g + h + i \\ 3(0.5) + 4(0.2) + 6(0.1)_1 + 2(0.1)_2 & = +a + b + c + d + e + f + g + h + i \\ (0.5) + 2(0.2) + 2(0.1)_1 + 2(0.1)_2 & = +a + b + c + d + e + f + g + h + i \end{aligned}$$

In the above equations, the constants 0.1 through 0.9 have been eliminated by assuming that the weight symbols (0.5), (0.2), (0.1)₁, and (0.1)₂ now stand for the corrections for the weights rather than their actual values. For example, correction (0.5) = actual value (0.5) - 0.5 gram.

The solution of the normal equations is

$$\begin{aligned} (0.5) & = \frac{1}{19} (-2a - 2b - 4c - 3d + 6e + 4f + 4g + 2h + 3i) \\ (0.2) & = \frac{1}{38} (-5a + 14b + 9c + 2d - 4e - 9f + 10g + 5h - 2i) \\ (0.1)_1 & = \frac{1}{38} (+14a - 5b + 9c + 2d - 4e + 10f - 9g + 5h - 2i) \\ (0.1)_2 & = \frac{1}{38} (-7a - 7b - 14c + 18d + 2e - 5f - 5g - 12h + 20i) \end{aligned}$$

The necessary computations may be simplified by arranging them according to the computation form illustrated in Table II. First the corrected observations (a through i) are recorded. These are then multiplied by the appropriate factors and added to obtain the quantities A , B , C , and D . The computations of A , B , C , and D are now checked for accuracy as shown on the form.

As indicated, the correction for the weight (5), in this case (0.5), is $\frac{1}{19} A$. The corrections for the weights (0.2), (0.1)₁, and (0.1)₂ are then obtained from this value for (0.5) and the quantities B , C , and D , respectively. Thus

$$\begin{aligned} (0.5) & = + \frac{1}{19} A \\ (0.2) & = \frac{1}{8} [+10(5) + 2B] \\ (0.1)_1 & = \frac{1}{8} [+10(5) + 2C] \\ (0.1)_2 & = \frac{1}{8} [-100(5) + 4D] \end{aligned}$$

By using the computation form shown only one division by 19 need be made longhand (or using a machine). All of the remaining calculations may be made mentally.

The same computation form may also be used for the units and tens weights.

After the values of the corrections for the four weights controlled by each dial have been computed in the manner described, the corrections for the individual dial settings may be computed from them and recorded on the computation form in the column headed "computed." These values which have been computed by the method of least squares are the best values to use in allowing for the errors in the dial-controlled weights.

Agreement with the value previously determined for the 0.1-gram dial weight may be used as a check on the computations involved in the least squares adjustment of the values for the corrections of the tenths dial weights. The computations may also be checked at each step, as described below.

VARIABILITY

A measure of the variability of the balance may be computed from the residuals obtained when computing the corrections for the dial-controlled weights. The residuals (Table II) are the differences between the corrected observed values for each dial setting and the least-squares adjusted values. If the residuals are squared and the sum of the squares found for the tenths, units, and tens dials, and if the sum of these three sums is divided by 15 and the square root found, the result is the standard deviation.

$$\text{Standard deviation } (S_4) = \sqrt{\frac{\Sigma r_{0.1}^2 + \Sigma r_1^2 + \Sigma r_{10}^2}{15}}$$

CHECKS ON OBSERVATIONS
AND COMPUTATIONS

Table III. Balance Test and Calibration of Dial-Controlled Weights for Tenths Dial

Dial Setting	Obsvd. ^a	Computed	Residuals, Obs.-Comp.	(Residuals) ²
0.1 = (1) ₁	<i>a</i> = -0.025 mg.	-0.026 mg.	+0.001 <i>r</i> ₁	0.000001
0.2 = (2)	<i>b</i> = +0.038	+0.044	-0.006 <i>r</i> ₂	0.000036
0.3 = (2) + (1) ₁	<i>c</i> = +0.026	+0.018	-0.008 <i>r</i> ₃	0.000064
0.4 = (2) + (1) ₁ + (1) ₂	<i>d</i> = +0.039	+0.037	+0.002 <i>r</i> ₄	0.000004
0.5 = (5)	<i>e</i> = +0.028	+0.011	-0.017 <i>r</i> ₅	0.000289
0.6 = (5) + (1) ₁	<i>f</i> = -0.027	-0.015	-0.012 <i>r</i> ₆	0.000144
0.7 = (5) + (2)	<i>g</i> = +0.049	+0.055	-0.006 <i>r</i> ₇	0.000036
0.8 = (5) + (2) + (1) ₁	<i>h</i> = +0.033	+0.029	+0.004 <i>r</i> ₈	0.000016
0.9 = (5) + (2) + (1) ₁ + (1) ₂	<i>i</i> = +0.047	+0.048	-0.001 <i>r</i> ₉	0.000001
Sum of squares = 0.000591				

A		B		C		D	
+	-	+	-	+	-	+	-
-2 <i>a</i>	0.050	0 <i>a</i>		+2 <i>a</i>	0.050	-3 <i>a</i>	0.075
-2 <i>b</i>		+2 <i>b</i>	0.076	0 <i>b</i>		-3 <i>b</i>	
-4 <i>c</i>	0.104	+2 <i>c</i>	0.052	+2 <i>c</i>	0.052	-6 <i>c</i>	0.156
-3 <i>d</i>	0.117	+1 <i>d</i>	0.039	+1 <i>d</i>	0.039	-3 <i>d</i>	0.117
+6 <i>e</i>	0.168	-2 <i>e</i>		0 056		+8 <i>e</i>	0.224
+4 <i>f</i>		-2 <i>f</i>	0.054	0 <i>f</i>		+5 <i>f</i>	0.135
+4 <i>g</i>	0.196	0 <i>g</i>		-2 <i>g</i>	0.098	+5 <i>g</i>	0.245
+2 <i>h</i>	0.066	0 <i>h</i>		0 <i>h</i>		+2 <i>h</i>	0.066
+3 <i>i</i>	0.141	-1 <i>i</i>		0.047	0.047	+5 <i>i</i>	0.235
+0.621 -0.405		+0.221 -0.103		+0.091 -0.251		+0.845 -0.522	
-0.405		-0.103		+0.091		-0.522	
+0.216		+0.118		-0.160		+0.323	

Check on Computations of A, B, C, and D		(0.5)	(0.2)	(0.1) ₁	(0.1) ₂
+	-	+ 1/19 A	+10(5) + 2 B	+10(5) + 2 C	-100(5) + 4 D
-3 <i>a</i>	0.075		+0.114	+0.114	-1.137
-3 <i>b</i>			+0.236	-0.320	+1.292
-6 <i>c</i>	0.114		+0.350	-0.206	+0.155
-4 <i>d</i>	0.156		+0.044	-0.026	+0.019
+10 <i>e</i>	0.280				
+7 <i>f</i>	0.189				
+7 <i>g</i>	0.343				
+4 <i>h</i>	0.132				
+6 <i>i</i>	0.282				
+1.112 -0.615		+0.657 -0.160		Note that sums vanish	
-0.615		-0.160			
+0.497		+0.497 ^b			

^a Note. To the observed difference, zero reading minus standard reading, add the correction for the standard weights.

^b Note agreement.

The reason for dividing by 15 is that although 27 observations are made (9 observations for each dial) 12 unknowns are found (4 weights for each dial), leaving only 15 degrees of freedom (4).

This value of the standard deviation may be compared with that previously computed, S_3 , and should be in good agreement with it (see the section on checks). When in good agreement, the two values may be combined according to the formula:

Standard deviation (S_5) =

$$\sqrt{\frac{\sum(x_i - \bar{x})^2 + \sum(y_i - \bar{y})^2 + \sum r_{0.1}^2 + \sum r_1^2 + \sum r_{10}^2}{9 + 9 + 15}}$$

S_5 or some multiple of S_5 may be used as a measure of the variability of the balance and therefore of the suitability of the balance for a particular application. S_1 , S_2 , or S_4 and therefore S_3 or finally S_5 is the standard deviation not of a single observation but of the difference between a pair of observations. It takes a pair of observations to make a weighing: The mass of an object is determined from the difference between the balance readings with the object on the pan and with the pan empty. The mass of a sample is determined from the difference between the balance reading for the container and the balance reading for the container plus sample. (The latter is called a differential weighing whenever the two balance readings are made without changing the setting of the dials.) Since a weighing involves the determination of the difference between two observations, S_5 is the standard deviation of a weighing.

The computation sheet illustrated in Table II contains various checks on observations and computations.

An intermediate check on the computations is provided by computing the sum of A , B , C , and D :

$$A + B + C + D = -3a - 3b - 6c - 4d + 10e + 7f + 7g + 4h + 6i$$

These sums should be in exact numerical agreement.

The calculations involved in the least squares adjustment may be checked by observing whether or not the following sums of residuals add to zero within the error of rounding off.

$$\begin{aligned} r_4 + r_9 &= 0 \\ r_2 + r_3 + r_7 + r_8 &= 0 \\ r_5 + r_6 + r_7 + r_8 + r_9 &= 0 \\ r_1 + r_3 + r_6 + r_8 &= 0 \end{aligned}$$

The uniformity of the residuals serves as a check against gross errors in the observations.

The following additional statistical checks may be made:

There is only one chance in twenty according to statistical theory that the ratio of the largest of the three sums $\sum r_{0.1}^2$, $\sum r_1^2$, or $\sum r_{10}^2$ to the sum of the three will be greater than 0.7071 (Cochran's test) (1).

$$\frac{(\sum r^2) \text{ max.}}{\sum r_{0.1}^2 + \sum r_1^2 + \sum r_{10}^2} < 0.7071$$

19 times in 20

This statement is true only if the three sums are measures of the same thing—i.e., only if there is reason to expect the same variability when using the different dials. As the balance is always operated at the same total load, the variability of the balance should probably be independent of the dial used. Hence, the inequality may be considered a rough check on the observations.

In comparing the standard deviations, S_3 and S_4 , it is necessary to divide the larger of these two values by the smaller. This ratio will be greater than the values given below only one time in ten, provided that the two standard deviations are measures of the same thing (2, 5).

$$\left(\frac{S_3}{S_4}\right)^2 < 2.36 \quad \text{or} \quad \left(\frac{S_4}{S_3}\right)^2 < 2.27 \quad 9 \text{ times in } 10$$

NUMERICAL EXAMPLE

The following is a sample of the observations from which the sensibility reciprocal and the correction for the 0.1-gram dial weight are computed:

Load on Pan, Mg.	Observations		Corr. for Std., Mg.	Computations	
	Dials, g.	Scale, mg.		0.1-G. wt., mg.	100 divisions, mg.
Set	0	00.0	0.00
	L 100 ₁	00.1	+0.04	-0.015	...
	L 100 ₁	00.0	99.96	-0.015	+0.005
	0	00.0	-0.02
Reset	0	00.0	0.00
	L 100 ₂	00.1	+0.03	-0.007	-0.037
	L 100 ₂	00.0	99.99	-0.007	+0.003
	0	00.0	0.00
Reset	0	etc.
Average values				\bar{x} = -0.036 mg.	\bar{y} = +0.009 mg.
$\sum(x_i - \bar{x})^2$ or $\sum(y_i - \bar{y})^2$				0.003650	0.001970
Standard deviation				s_1 = 0.020 mg.	s_2 = 0.015 mg.

Table IV. Balance Test and Calibration of Dial-Controlled Weights for Units Dial

Dial Setting	Obsvd. ^a	Computed	Residuals, Obs.-Comp.	(Residuals) ²
1 = (1) ₁	a = -0.015 mg.	-0.018 mg.	+0.003 r ₁	0.000009
2 = (2)	b = +0.084	+0.064	+0.020 r ₂	0.000400
3 = (2) + (1) ₁	c = +0.029	+0.046	-0.017 r ₂	0.000289
4 = (2) + (1) ₁ + (1) ₂	d = -0.042	-0.032	-0.010 r ₂	0.000100
5 = (5)	e = +0.043	+0.065	-0.022 r ₅	0.000484
6 = (5)	f = +0.063	+0.047	+0.016 r ₅	0.000256
7 = (5) + (2)	g = +0.128	+0.129	-0.001 r ₇	0.000001
8 = (5) + (2) + (1) ₁	h = +0.107	+0.111	-0.004 r ₇	0.000016
9 = (5) + (2) + (1) ₁ + (1) ₂	i = +0.042	+0.033	+0.009 r ₉	0.000081
Sum of Squares = 0.001636				

A		B		C		D	
+	-	+	-	+	-	+	-
-2 a	0.030	0 a		+2 a	0.030	-3 a	0.045
-2 b	0.168	+2 b	0.168	0 b		-3 b	0.252
-4 c	0.116	+2 c	0.058	+2 c	0.058	-6 c	0.174
-3 d	0.126	+1 d	0.042	+1 d	0.042	-3 d	0.126
+6 e	0.258	-2 e	0.086	-2 e	0.086	+8 e	0.344
+4 f	0.252	-2 f	0.126	0 f		+5 f	0.315
+4 g	0.512	0 g		-2 g	0.256	+5 g	0.640
+2 h	0.214	0 h	0.042	0 h	0.042	+2 h	0.214
+3 i	0.126	-1 i		-1 i		+5 i	0.210
+1.518 -0.284		+0.226 -0.296		+0.058 -0.456		+1.894 -0.426	
-0.284		+0.226		+0.058		-0.426	
+1.234		-0.070		-0.398		+1.468	

Check on Computations of A, B, C, and D		(5)	(2)	(1) ₁	(1) ₂
+	-	+1/19 A	+10(5) +2 B	+10(5) +2 C	-100(5) +4 D
-3 a	0.045	+0.06495	+0.649	+0.649	-6.495
-3 b	0.252		-0.140	-0.796	+5.872
-6 c	0.174	+0.065	Sum	Sum	Sum
-4 d	0.168		+0.064	-0.018	-0.078
+10 e	0.430		1/8 Sum	1/8 Sum	1/8 Sum
+7 f	0.441	A	Checks on Computations (Add residuals as follows)		
+7 g	0.896	B	r ₄ + r ₉	-0.001	
+4 h	0.428	C	r ₂ + r ₂ + r ₇ + r ₈	-0.002	
+6 i	0.252	D	r ₅ + r ₅ + r ₇ + r ₈ + r ₉	-0.002	
			r ₁ + r ₃ + r ₆ + r ₈	-0.002	
+2.660 -0.426		+2.702 -0.468	Note that sums vanish		
-0.426		-0.468			
+2.234		+2.234 ^b			

^a Note. To the observed difference, zero reading minus standard reading, add the correction for the standard weights.
^b Note agreement.

The L100₁ and L100₂ are two 100-mg. standards of the L set, and are marked with one and two dots, respectively. Although the weighings are estimated to only 0.01 mg., the computations are made to 0.001 mg. in order not to lose any precision by rounding off the known values of the standards.

From this it is seen that

$$SR = \frac{100 + 0.009}{100} = 1.00009 \text{ mg./division}$$

$$S_1 = \sqrt{\frac{0.003650}{9}} = 0.020 \text{ mg.}$$

$$S_2 = \sqrt{\frac{0.001970}{9}} = 0.015 \text{ mg.} \quad \left(\frac{S_1}{S_2}\right)^2 = \frac{3650}{1970} = 1.85 < 3.18$$

Hence, these two estimates of the variability of the balance may be combined.

$$S_3 = \sqrt{\frac{0.003650 + 0.001970}{9 + 9}} = 0.018 \text{ mg.}$$

To show how the corrected observations are obtained, the first few observations for the tenths dial are shown:

Load on Pan, Mg.	Observations Dials, g.	Scale, mg.	Adjacent Zero Minus Observation, Mg.	Corr. for Std., Mg.	Corrected Observations, Mg.
0	00.0	+0.04
L100 ₁	00.1	+0.05	-0.01	-0.015	-0.025
L100 ₁ + 100 ₂	00.2	0.00	+0.06	-0.022	+0.038
0	00.0	+0.06
0	00.0	+0.04
L200 + 100 ₁	00.3	+0.01	+0.03	-0.004	+0.026
L200 + 100 ₁ + 100 ₂	00.4	0.00	+0.05	-0.011	+0.039
0	00.0	+0.05
0	00.0	+0.01
L500	00.5	+0.01	0.00	+0.028	+0.028
etc.					

The computations for the dial-controlled weights are given in Tables III, IV, and V.

From the summary at the end of Table V, it is seen that

$$\frac{(\sum r^2) \text{ max.}}{\sum r_{0.1}^2 + \sum r_{1}^2 + \sum r_{10}^2} = \frac{0.001636}{0.002736} = 0.60 < 0.7071$$

The ratio 0.60 is large, but not so large as to create doubt that the standard deviations for the three dials may be considered to be measures of the same thing. Hence, the standard deviations for the separate dials may be combined to form a single estimate of the variability of the balance.

$$S_4 = \sqrt{\frac{0.002736}{15}} = 0.014 \text{ mg.}$$

If the appropriate inequality is applied, it is seen that S₃ and S₄ are measures of the same thing, and hence a single measure of the variability of the balance (S₅) may be computed:

$$\left(\frac{S_3}{S_4}\right)^2 = \left(\frac{0.018}{0.014}\right)^2 = 1.7 < 2.36$$

$$S_5 = \sqrt{\frac{0.003650 + 0.001970 + 0.002736}{9 + 9 + 15}} = \sqrt{\frac{0.008356}{33}} = 0.016 \text{ mg.}$$

This is the standard deviation to be expected in the weighing of a sample on this balance.

CONCLUSIONS

Four 100-gram Mettler Gram-atic balances were tested in the Mass Laboratory of the National Bureau of Standards. The standard deviations to be expected in the differential weighing of samples on these balances were found to be 0.016, 0.017, 0.016, and 0.034 mg., respectively.

The first three of these are the standard deviation S₅—that is, all of the other standard deviations satisfied the various tests for combination into the single standard deviation S₅. The last value shown is the value of S₄ for the fourth balance. The value of S₃ for the fourth balance was found to be 0.015 mg. Thus

$$\left(\frac{S_4}{S_3}\right)^2 = \left(\frac{0.034}{0.015}\right)^2 = 5.1 > 2.27$$

Since this ratio is greater than 2.27, the two standard deviations are not likely to be measures of the same thing. The zero shift during the course of the test was considerably larger for this fourth balance than for the other three. Recomputing S₄ for this balance using interpolated zeros rather than adjacent zeros, however, resulted in only a slight decrease in S₄ (to 0.032 mg.). Without further investigation, the best that can be said is that the large zero shift and the large value for S₄ are possibly results of the same constructional defects.

An accuracy of 0.02 mg. is claimed by the manufacturer for samples measured by differential weigh-

Table V. Balance Test and Calibration of Dial-Controlled Weights for Tens Dial

Dial Setting	Obsvd. ^a	Computed	Residuals Obs.-Comp.	(Residuals) ²
10 = (1) ₁	a = +0.042 mg	+0.047 mg.	-0.005 r ₁	0.000025
20 = (2)	b = +0.014	+0.024	-0.010 r ₂	0.000100
30 = (2) + (1) ₁	c = +0.066	+0.071	-0.005 r ₃	0.000025
40 = (2) + (1) ₁ + (1) ₂	d = -0.104	-0.102	-0.002 r ₄	0.000004
50 = (5)	e = -0.112	-0.096	-0.016 r ₅	0.000256
60 = (5) + (1) ₁	f = -0.050	-0.049	-0.001 r ₆	0.000001
70 = (5) + (2)	g = -0.068	-0.072	+0.004 r ₇	0.000016
80 = (5) + (2) + (1) ₁	h = -0.016	-0.025	+0.009 r ₈	0.000081
90 = (5) + (2) + (1) ₁ + (1) ₂	i = -0.197	-0.198	+0.001 r ₉	0.000001
Sum of squares = 0.000509				

A		B		C		D	
+	-	+	-	+	-	+	-
-2 a	0.084	0 a		+2 a	0.084	-3 a	0.126
-2 b	0.028	+2 b	0.028	0 b		-3 b	0.042
-4 c	0.264	+2 c	0.132	+2 c	0.132	-6 c	0.396
-3 d	0.312	+1 d	0.104	+1 d	0.104	-3 d	0.312
+6 e	0.672	-2 e	0.224	-2 e	0.224	+8 e	0.896
+4 f	0.200	-2 f	0.100	0 f		+5 f	0.250
+4 g	0.272	0 g		-2 g	0.136	+5 g	0.340
+2 h	0.032	0 h		0 h		+2 h	0.032
+3 i	0.591	-1 i	0.197	-1 i	0.197	+5 i	0.985
+0.312	-2.143	+0.681	-0.104	+0.773	-0.104	+0.312	-3.067
	+0.312		-0.104		-0.104		+0.312
	-1.831		+0.577		+0.669		-2.755

Check on Computations A, B, C, and D		(50)	(20)	(10) ₁	(10) ₂
+	-	+ 1/19 A	-0.09636	+10(5) -0.964	+10(5) -0.964
-3 a	0.126			+2 B +1.154	+2 C +1.338
-3 b	0.042			Sum +0.190	Sum +0.374
-6 c	0.396	+ 1/19 A	-0.096	1/8 Sum +0.024	1/8 Sum +0.047
-1 d	0.416			1/8 Sum	1/8 Sum
+10 e	1.120				
+7 f	0.350	A	1.831	Checks on Computations (Add residuals as follows)	
+7 g	0.476	B	0.577	r ₄ + r ₉	-0.001
+4 h	0.064	C	0.669	r ₂ + r ₃ + r ₇ + r ₈	-0.002
+6 i	1.182	D	2.755	r ₅ + r ₆ + r ₇ + r ₈ + r ₉	-0.003
				r ₁ + r ₄ + r ₆ - r ₈	-0.002
				Summary of Tests of Three Dials	
				Tens	Squares
				Units	0.000591
				Sum	0.001636
				Tens	0.000509
				Sum	0.002736
				1/15 Sum	0.0001825
				1/15 sum	0.0135 mg.

^a Note. To the observed difference, zero reading minus standard reading, add the correction for the standard weights.
^b Note agreement.

ing with balances of this model. The differential method of weighing eliminates the errors of the dial weights from the result of a weighing. If it is assumed that a differential weighing is also free from other systematic errors, it follows that the uncertainty present in a differential weighing will be determined entirely by the precision with which the balance repeats its indications. Therefore, the standard deviation or the probable error could be used as the measure of the accuracy of a differential weighing; however, the concept accuracy usually carries with it the connotation that an actual error greater than the stated accuracy will rarely occur. There is no generally accepted definition of a rare occurrence—one time in ten or hundred or otherwise is used depending on the purpose. One time in twenty has been chosen as being fairly rare and yet not too unfavorable to the manufacturer.

If therefore by the accuracy of a differential weighing is meant that 95% of the time any two differential weighings of the same sample will agree with each other to within a given value, it may be stated that the values of accuracy for the balances tested are 0.044, 0.047, 0.044, and 0.09 mg., respectively. (The quantities given in the text are for agreement between two weighings. Agreement between two weighings is the same as the error of an analysis involving two weighings. The standard deviation for the difference between two quantities each of standard deviation S is $S' = \sqrt{2}S$. Ninety-five per cent of the area under the normal curve lies between limits of $\pm 1.960 S'$.) In order to have an accuracy of 0.02 mg. in this sense it is necessary that the standard deviation be 0.007 mg. Alternatively, it could be said that each of the first three of the foregoing balances has an accuracy of 0.02 mg. if instead of requiring this agreement in 95% of the cases it is required in two thirds of the cases only.

It will be observed from the data given in the numerical example that, if other than differential weighing is used and if the corrections for the dial-controlled weights are neglected, a maximum positive error of +0.255 mg. is obtained at a dial setting of 37.7 grams, and a maximum negative error of -0.256 mg. is obtained at 94.1 grams. Corresponding figures for the other balances tested are +0.210 and -0.221 mg.; +0.22 and -0.06 mg.; and +0.095 and -0.258 mg. The maximum possible error in the difference between two dial settings is approximately 0.5 mg.

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CALIBRATION OF DIAL CONTROLLED WEIGHTS

DATE _____

METTLER ARRANGEMENT

DIAL _____

BALANCE _____ OBSERVER AND COMPUTER _____

DIAL SETTING	Note: To the observed difference—zero reading minus standard reading—add the correction for the standard weights.	OBSERVED (Zero—standard + Cr. standard)	COMPUTED	RESIDUALS OBS.—COMP.	(RESIDUALS) ²			
1	(1) ₁	= a =						
2	(2)	= b =						
3	(2) + (1) ₁	= c =						
4	(2) + (1) ₁ + (1) ₂	= d =						
5	(5)	= e =						
6	(5) + (1) ₁	= f =						
7	(5) + (2)	= g =						
8	(5) + (2) + (1) ₁	= h =						
9	(5) + (2) + (1) ₁ + (1) ₂	= i =						
SUM OF SQUARES =								
A		B		C		D		
	+	-		+	-	+	-	
-2 a			0 a	+2 a		-3 a		
-2 b			+2 b	0 b		-3 b		
-4 c			+2 c	+2 c		-6 c		
-3 d			+1 d	+1 d		-3 d		
+6 e			-2 e	-2 e		+8 e		
+4 f			-2 f	0 f		+5 f		
+4 g			0 g	-2 g		+5 g		
+2 h			0 h	0 h		+2 h		
+3 i			-1 i	-1 i		+5 i		
CHECKS ON COMPUTATIONS OF A, B, C, & D			(5)	(2)	(1) ₁	(1) ₂		
-3 a	+	-	+ $\frac{1}{19}$ A	+10(5)	+10(5)	-10(5)		
-3 b				+2 B	+2 C	+4 D		
-6 c				SUM	SUM	SUM		
-4 d			+ $\frac{1}{19}$ A	$\frac{1}{8}$ SUM	$\frac{1}{8}$ SUM	$\frac{1}{8}$ SUM		
+10 e				CHECK ON COMPUTATION Add residuals as follows		SUMMARY OF TESTS OF THREE DIALS		
+7 f			A	r ₄ + r ₉		DIAL	SUM OF SQUARES	
+7 g			B	r ₂ + r ₃ + r ₇ + r ₈		TENTHS		
+4 h			C	r ₅ + r ₆ + r ₇ + r ₈ + r ₉		UNITS		
+6 i			D	r ₁ + r ₃ + r ₆ + r ₈		TENS		
				Note that sums vanish		SUM		
			=			$\frac{1}{15}$ SUM		
Note agreement			STANDARD DEVIATION = $\sqrt{\frac{1}{15} \text{SUM}}$					

Sources of Error in Various Methods of Airplane Camera Calibration*†

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ABSTRACT: *Sources of error inherent in several methods used in the calibration of airplane-mapping cameras are discussed. The investigation is confined to errors affecting the measured values of focal length and distortion. Three photographic methods used in camera calibration are considered. Principal sources of error are errors in length measurements on the calibration negative, errors in angle separating targets in the object space, and errors arising out of departure from flatness in the photographic plates used in the calibration.*

1. INTRODUCTION,

THE calibration of airplane mapping cameras (1, 2, 3) has occupied the attention of a number of organizations both here and abroad since the first development of aerial photography. As the airplanes and cameras have improved, the requirements placed on the cameras and the lenses with which they are equipped have become more stringent (4, 5, 6). To achieve the required accuracy of calibration, testing laboratories that are charged with the responsibility of certifying the accuracy of the camera calibrations have been steadily improving their techniques. The press of work and the need for speed have, in general, precluded close cooperation between the various laboratories. In consequence, many methods of camera calibration have been developed. In each instance, the method developed has been one that is capable of yielding the information required but these methods are not all equally simple or capable of handling the same volume of work.

In the United States and Canada, the emphasis has been on the photographic method of camera calibration while in Europe greater emphasis has been placed on visual processes. The photographic method has been employed in a variety of ways depending upon the location of the

laboratory and the equipment readily available. Thus the National Bureau of Standards has developed the precision lens testing camera (7) and the camera calibrator (8). At Wright Field, the field method has been developed and perfected to a high degree by Sewell (9) and associates. Additional field methods that yield a high degree of accuracy and are suited to a variety of conditions have been developed by Merritt (10). Laboratory methods that yield the required information have been developed in Canada by Howlett (11) and Field (12).

Visual methods that employ a goniometer have been developed in Europe (13, 14, 15) and are described in the literature. In the United States, Merritt (16) has developed a successful goniometer method.

However divergent the approaches to the problem of precise camera calibration, the end results are necessarily the same because all are seeking the same type of information, namely accurate values of the scale factor to be used in map interpretation. Consequently, the factors affecting the final accuracy are much the same regardless of method. In the present paper, an analysis of several sources of error is given and the manner in which these errors affect the final accuracy is shown for several photographic methods.

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† This is the first paper in a series of four papers by Dr. Washer. The remaining three will be in following issues of this JOURNAL.—EDITOR.

Particular emphasis is given to those errors that affect the determination of values of focal length and distortion. Detailed tabulations of actual data are shown in some instances in order to give a better understanding of the magnitudes involved.

2. PRECISION OF DETERMINATION OF EQUIVALENT FOCAL LENGTH AND DISTORTION

The accuracy of the measured values of equivalent focal length and distortion for a given airplane camera lens is dependent upon several factors which may produce error. A prime source of variation in the measured value of the equivalent focal length itself is the actual selection of the preferred focal plane; this gives rise to definite measured differences in the equivalent focal length but has no appreciable effect on the distortion values. More serious and definite errors arise from inaccuracies present in the determination of the angles separating targets, and in the determination of distances on the negative from which the values of equivalent focal length and distortion are obtained. These errors may be systematic or accidental, but so far as this investigation is concerned only the accidental or random errors will be considered. Curvature or waviness of the registering surface also contributes to error in the values of equivalent focal length and distortion; this error is here treated as a random error although at times it may be regarded as a systematic error.

These factors affecting the variation of measured values of the equivalent focal length and the accuracy of determination of equivalent focal length and distortion are herein discussed as they apply to photographic methods of measurement. It is, however, apparent that these same factors may be present in visual methods and will affect the final accuracy of measurement in much the same manner.

2.1 SELECTION OF THE FOCAL PLANE

When a lens is tested prior to mounting in a camera there is some uncertainty as to the location of the focal plane that will yield best average definition (17). Because of field curvature, the plane of best average definition seldom coincides with the plane of best axial focus. For a lens of aperture $f/6.3$, there exists a range of

approximately 1.2 mm. along the axis wherein the resolving power will be not less than 20 lines per mm. For the same lens, the depth of focus along the axis may be as high as 1.8 mm. wherein the resolving power will not be less than 14 lines per mm. Consequently if the resolving power requirement is 20 lines per mm. on the axis and not less than 14 lines per mm. in other portions of the field, a range may exist of 0.6 to 0.8 mm. wherein the specified values of resolving power are obtained even if the curvature of field amounts to as much as 0.5 mm.

It is usually possible by careful examination of the test negative to locate the plane of best average definition within ± 0.1 mm. It must be remembered, however, that it is probable that the performance of the lens may satisfy the specified tolerances even if the selected focal plane may be separated as much as 0.4 mm. from the plane of best average definition.

It is this depth of focus that makes it possible for two laboratories to report values of the equivalent focal length for the same lens differing by as much as 0.2 to 0.3 mm. Both laboratories may be doing equally good work but may be using different criteria in locating the plane of best average definition. In order to judge whether or not the focal length determinations from two laboratories are consistent, values of both equivalent focal length and back focal length must be considered. The consistency may then be judged by comparing the arithmetic differences between corresponding equivalent and back focal lengths. This difference should show no appreciable variation.

This variation in the measured value of the equivalent focal length arising from choice of a focal plane cannot occur when the lens is mounted in an airplane camera having a fixed focal plane provided that the equivalent focal length is measured for the lens as mounted in the camera.

2.2 THE EFFECT OF ERRORS IN R AND β

a. Determination of the equivalent focal length.

The equivalent focal length is defined (18) by the equation

$$f = \lim_{\beta \rightarrow 0} r \cot \beta \quad (1)$$

where r is the transverse distance from the principal focus to the center of the image in the image-space focal plane of an

infinitely distant object which lies in a direction making an angle β with the axis of the objective. The principal focus is defined as the axial image point of an object at infinity (2). For a lens free from distortion the value of f would be invariant with respect to the value of β . For many photographic purposes the distortion is negligible for points distant from the center of the useful field not more than one-fifth of its radius and within this region the above equation may, with sufficient accuracy, be written as

$$f = r \cot \beta. \quad (2)$$

It is evident that the accuracy of the value of f obtained from this equation is dependent on the accuracy of the measured angle, β . If the probable errors in r and β are known, the probable error in f can be determined (22). This can be done by differentiating the above equation which yields

$$df = dr \cot \beta - r \operatorname{cosec}^2 \beta d\beta. \quad (3)$$

It is accordingly clear that if Δf_r is the probable error* in f arising from the probable error Δr inherent in the measurement of r , then

$$\Delta f_r = \cot \beta \cdot \Delta r. \quad (4)$$

Similarly, if Δf_β is the probable error in f arising from the probable error $\Delta \beta$ inherent in the measurement of β , then

$$\Delta f_\beta = r \operatorname{cosec}^2 \beta \Delta \beta \quad (5)$$

which may be written

$$\Delta f_\beta = \frac{0.000004848}{\sin \beta \cos \beta} f \Delta \beta = B f \Delta \beta \quad (6)$$

for values of $\Delta \beta$ expressed in seconds. The total probable error in the value of f arising from probable errors in both r and β may be found from the relation

$$\Delta f = \pm \sqrt{(\Delta f_r)^2 + (\Delta f_\beta)^2}. \quad (7)$$

Values of the probable errors $\Delta \beta$ are usually obtained from the analysis of experimental data containing repeated measurements of r and β .

Values of $\cot \beta$ and B for those values of

* Throughout this paper, the symbol Δ is used to designate probable error. Thus Δr is the probable error in the measurement of r ; Δf_r is the probable error in focal length arising from a determination of f based on a value of r that has a probable error Δr . It is understood that all probable errors are plus or minus and that \pm signs are often omitted herein.

β frequently used in camera calibration are listed in the second columns of Tables 1 and 2 respectively. These values can be used to determine the probable errors Δf_r and Δf_β when the magnitudes of the probable errors Δr and $\Delta \beta$ are known. For convenience in computation, values of Δf_r are listed in Table 1 for a series of values of Δr . Similarly, values of Δf_β are shown in Table 2 for a series of values of $\Delta \beta$. The values of Δf_β in Table 2 are given for a lens having a focal length of 150 mm. since the value of Δf_β is also dependent on the focal length of the lens.

To illustrate the use of Tables 1 and 2 in evaluating the probable error in equivalent focal length, Table 3 shows the values of Δf arising from assumed specific values of Δr and $\Delta \beta$ for a lens having a focal length of 150 mm. It is clear from this table that for a distortion-free lens, the accuracy of an equivalent focal length determination increases with increasing angle β . The usual lens is, however, not free from distortion and consequently the magnitude of the angle β that may be used is limited. The presently accepted rule (18) is that the angle β , used in an equivalent focal length determination, should not be greater than the angle subtended at the lens by the axial image and a point one-fifth of the distance between the center and edge of the useful field. For a lens whose half-angular field is 45° , this limits the angle β to approximately 11.3° .

The foregoing discussion applies primarily to the equivalent focal length of a camera lens. In the case of precision cameras, the calibrated focal length is of greater importance to the user. Ordinarily, the calibrated focal length is thought of as adjusted value of the focal length calculated to minimize distortion over the entire field. It has been shown (8) that values closely approximating the calibrated focal length can be obtained by using a value of β in the determining equation of focal length that corresponds to a zero point of distortion computed with respect to the calibrated focal length. Such a zero point occurs when β has a value of approximately 40° . From Table 3, Δf is ± 0.007 mm. at $\beta = 40^\circ$ as compared with $\Delta f = \pm 0.025$ mm. at $\beta = 10^\circ$. It is clear, therefore, that the value assigned to the calibrated focal length for a given lens has appreciably less error than the value assigned to the equivalent focal length for

TABLE 1

THE PROBABLE ERROR, Δf_r , IN FOCAL LENGTH ARISING FROM ERRORS IN r ALONE FOR VARIOUS ANGULAR SEPARATIONS (β) FROM THE AXIS FOR A SERIES OF VALUES OF Δr

These values are computed with the aid of eq. 2

β	cot β	Δf_r for values of Δr in millimeters of					
		0.001	0.002	0.003	0.004	0.005	0.006
degrees		mm.	mm.	mm.	mm.	mm.	mm.
5	11.430	0.011	0.023	0.034	0.046	0.057	0.069
7.5	7.596	.007	.015	.023	.030	.038	.046
10	5.671	.006	.011	.017	.023	.028	.034
12.5	4.511	.005	.009	.014	.018	.023	.027
15	3.732	.004	.007	.011	.015	.019	.022
20	2.747	.003	.005	.008	.011	.014	.016
22.5	2.414	.002	.005	.007	.010	.012	.014
25	2.145	.002	.004	.006	.009	.011	.013
30	1.732	.002	.003	.005	.007	.009	.010
35	1.428	.001	.003	.004	.006	.007	.009
37.5	1.303	.001	.003	.004	.005	.007	.008
40	1.192	.001	.002	.004	.005	.006	.007
45	1.000	.001	.002	.003	.004	.005	.006

the same conditions of test.

Recently mapping lenses having very low distortions are being supplied to various mapping organizations. The maximum distortion referred to the calibrated focal length frequently does not exceed ± 0.020 mm. For such lenses, the value of β used in computing the equivalent focal length may properly be as high as 20° with a resultant

decrease in the value of Δf as compared with the value computed at $\beta = 10^\circ$

b. *Evaluation of the distortion*

Distortion D is defined by the equation (10)

$$D = r - f \tan \beta \quad (8)$$

where f is the equivalent focal length

TABLE 2

THE PROBABLE ERROR, Δf_β , IN FOCAL LENGTH ARISING FROM ERRORS IN β ALONE FOR VARIOUS ANGULAR SEPARATIONS (β) FROM THE AXIS FOR A SERIES OF VALUES OF $\Delta \beta$

These values are computed with the aid of eq. 6 and with the focal length, f , assumed to be 150 mm.

β	$B \times 10^6$	Δf_β for values of $\Delta \beta$ in seconds of					
		1	2	3	4	5	6
degrees	1-sec	mm.	mm.	mm.	mm.	mm.	mm.
5	55.84	0.008	0.017	0.025	0.033	0.042	0.050
7.5	37.46	.006	.011	.017	.022	.028	.034
10	28.35	.004	.009	.013	.017	.022	.026
12.5	22.94	.003	.007	.010	.014	.017	.020
15	19.39	.003	.006	.009	.012	.014	.017
20	15.08	.002	.004	.007	.009	.011	.014
22.5	13.71	.002	.004	.006	.008	.010	.012
25	12.66	.002	.004	.006	.008	.010	.011
30	11.20	.002	.003	.005	.007	.008	.010
35	10.32	.002	.003	.005	.006	.008	.009
37.5	10.04	.002	.003	.004	.006	.008	.009
40	9.85	.001	.003	.004	.006	.007	.009
45	9.70	.001	.003	.004	.006	.007	.009

TABLE 3

THE PROBABLE ERROR, Δf , IN FOCAL LENGTH ARISING FROM ERRORS IN BOTH r AND β FOR VARIOUS ANGULAR SEPARATIONS FROM THE AXIS

These values of Δf are computed with the aid of eq. 7. For these computations, it is assumed that $\Delta r = \pm 0.002$ mm.; $\Delta \beta = \pm 5$ seconds; and $f = 150$ mm.

β	Δf_r	Δf_f	$\Delta f = \pm \sqrt{(\Delta f_r)^2 + (\Delta f_f)^2}$
degrees	mm.	mm.	mm.
5	± 0.023	± 0.042	± 0.048
7.5	.015	.028	.032
10	.011	.022	.025
12.5	.009	.017	.019
15	.007	.014	.016
20	.005	.011	.012
22.5	.005	.010	.011
25	.004	.010	.011
30	.003	.008	.008
35	.003	.008	.008
37.5	.003	.008	.008
40	.002	.007	.007
45	.002	.007	.007

determined for a particular set of values of r and β . The probable error in distortion ΔD arising from the errors Δf , $\Delta \beta$, and Δr can be found by differentiating the above expression which yields

$$dD = dr - df \tan \beta - f \sec^2 \beta d\beta. \quad (9)$$

From which it is clear that if ΔD_r is the probable error in distortion arising from

uncertainties in the measurement of r , then

$$\Delta D_r = \Delta r. \quad (10)$$

Similarly, if ΔD_f is the error in distortion arising from errors in the value of f based on particular values of r and β , then

$$\Delta D_f = \tan \beta \Delta f. \quad (11)$$

Likewise, if ΔD_β is the error in distortion dependent on uncertainties in the measurement of β , then

$$\Delta D_\beta = f \sec^2 \beta \Delta \beta \quad (12)$$

$$= \frac{0.000004848}{\cos^2 \beta} f \Delta \beta = C f \Delta \beta. \quad (13)$$

The probable error, ΔD , arising from all three sources is

$$\Delta D = \pm \sqrt{(\Delta D_r)^2 + (\Delta D_f)^2 + (\Delta D_\beta)^2}. \quad (14)$$

Values of $\tan \beta$ and C for use in computing ΔD_f and ΔD_β are listed in Table 4. This table was used in the preparation of Tables 8, 10, 11, and 12.

2.3 FLATNESS OF THE REGISTERING SURFACE

Appreciable errors in the values of the equivalent focal length and distortion can result if the glass negative upon which the images are registered is not truly flat (19, 20). Figure 1 illustrates one case of departure from flatness. The photographic plate is assumed to be concave toward the lens L with a radius of curvature R where R is very great compared with the focal

TABLE 4
VALUES OF $\tan \beta$ AND C FOR VARIOUS ANGULAR SEPARATIONS (β) FROM THE AXIS

Values of $\tan \beta$ are for computing ΔD_f when using eq. 11 and the values of C are for computing ΔD_β when using eq. 13

β	$\tan \beta$	C
degrees	mm./mm.	mm./sec.
5	0.0875	0.000004894
7.5	.132	.000004933
10	.176	.000005000
12.5	.222	.000005086
15	.268	.000005194
20	.364	.000005489
22.5	.414	.000005678
25	.466	.000005906
30	.577	.000006461
35	.700	.000007222
37.5	.767	.000007706
40	.839	.000008261
45	1.000	.000009722

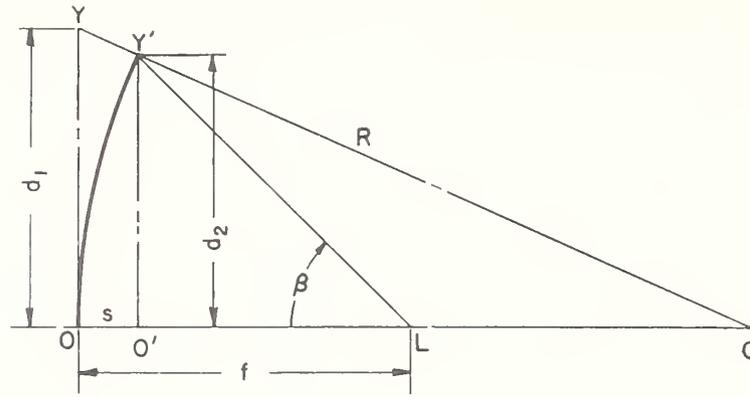


FIG. 1. Schematic drawing showing the effect of plate curvature on image position. OY is the focal plane, R is the radius of curvature of the plate. The distance d_2 which is the distance measured on the negative is less than d_1 because of the plate curvature.

length f , and O is the focal point of the lens L . Normally, Y is the point where a ray at angle β would be imaged but because of plate curvature the image appears at Y' . The plane $Y'O'$ is nearer to the lens than YO by the magnitude of the sagitta S . From the figure it is clear that

$$d_1 = f \tan \beta \quad (15)$$

and

$$d_2 = (f - S) \tan \beta. \quad (16)$$

For very large R and small S

$$d_2 = \sqrt{2RS} \quad (17)$$

from which it can be shown that

$$S = \frac{f^2 \tan^2 \beta}{2(R + f \tan^2 \beta)} \quad (18)$$

For $\beta = 45^\circ$, this equation becomes

$$S_{15^\circ} = \frac{f^2}{2(R + f)} \quad (19)$$

Then for S very small compared to R , values of S for other values of β can be computed from the approximate formula

$$S_\beta = S_{15^\circ} \cdot \tan^2 \beta. \quad (20)$$

Values of the distortion, D , are obtained from the relation

$$D = d_2 - d_1 \quad (21)$$

or

$$D = (f - S) \tan \beta - f \tan \beta \quad (22)$$

whence

$$D = -S \tan \beta \quad (23)$$

which yields the values of distortion computed with respect to the equivalent focal length.

It is not unusual to find magnitudes of the sagitta, S , as large as 0.200 mm. for glass plates that have not been selected for flatness. This is equivalent to a radius of curvature of $R = 56.1$ M for a chord of approximately 300 mm. in length. Table 5 shows the values of the distortion, induced in the image of a distortion-free lens, having a focal length of 150 mm. when a curved plate having a sagitta S of 0.200 mm. is used to register the image. The second column shows the values of S

TABLE 5

DISTORTION INTRODUCED BY PLATE CURVATURE FOR A DISTORTION FREE LENS HAVING A FOCAL LENGTH OF 150 MM.

The plate is assumed to be concave toward the lens with a maximum departure from flatness of 0.200 mm. at $\beta = 45^\circ$, which is equivalent to $R = 56.1$ M. Values of the distortion based upon the equivalent focal length EFL and two values of the calibrated focal length CFL are shown for selected values of β .

β	S	Distortion for		
		EFL of 150.000 mm.	CFL of 149.848 mm.	CFL of 149.899 mm.
degrees	mm.	mm.	mm.	mm.
0	0.000	0.000	0.000	0.000
7.5	.0035	-.0005	.020	.013
15	.014	.004	.037	.024
22.5	.034	.014	.049	.030
30	.067	.039	.048	.022
37.5	.118	-.091	.025	-.010
40	.141	-.118	.009	-.030
45	.200	-.200	-.048	-.095

computed for each angle β . The third column, headed *EFL* of 150.000 mm. shows the magnitude of the distortion when it is computed with respect to the equivalent focal length. The fourth column, headed *CFL* of 149.848 mm., shows the extent by which this type of distortion can be minimized over the entire image plane by proper selection of a calibrated focal length. The fifth column, headed *CFL* of 149.899 mm., shows the magnitude of the distortion when a calibrated focal length is selected to minimize distortion over that portion of the image plane that lies within 40 degrees from the axis.

Table 6 gives the same type of information as Table 5 except that a value of the sagitta $S=0.0250$ mm., is used in the computation. This is an especially interesting case because the values shown in this table are those which can be expected when the plate is plane to within ± 0.0005 inch which is the tolerance usually specified for camera platens. The values of $+0.006$ mm. at $\beta=22.5^\circ$ and 30° and -0.006 mm. at $\beta=45^\circ$ are particularly significant when it is recalled that the distortion requirements on some wide-angle lenses are set at ± 0.020 mm. referred to the calibrated focal length.

Unfortunately, actual photographic plates are neither truly flat nor do they usually exhibit a uniform curvature. All measurements made on a glass negative are affected to some extent by the varying

curvature or waviness of the surface. Consequently it is probable that the values of focal length and distortion derived from such measurements are affected by the departures from flatness of the photographic plate used in the calibration. While no method existed at the time* this study was made for yielding a quick and accurate contour map of the surface of the photographic plate as it exists at the moment of exposure, it is possible from the measurements on pairs of similar negatives to deduce the probable magnitude of the effect of plate curvature on the values of focal length and distortion. This is done by comparing the distances between the same two corresponding points on successive negatives made under the same conditions. The differences found should either be zero or proportional to tangent Δ , if the two plates are truly flat or have identical figures. It so happens that the measurements

* It is worthy of mention that Carman (21) has recently devised an interferometric method employing infrared light that enables one to bend an emulsion coated plate to minimize the departures from flatness. The photographic plate with the constraints still in place is then used in that condition to make the calibration negative. By this procedure, one is assured that the departures from flatness of the photographic plate are held to a few microns during exposure. It seems likely that this process should appreciably reduce the errors arising from plate curvature.

TABLE 6
DISTORTION INTRODUCED BY PLATE CURVATURE FOR A DISTORTION-FREE LENS
HAVING A FOCAL LENGTH OF 150 MM.

The plate is assumed to be concave toward the lens with a maximum departure from flatness of 0.0250 mm. at $\beta = \pm 45^\circ$, which is equivalent to $R=449.85$ M. Values of the distortion based upon the equivalent focal length, *EFL*, and two values of the calibrated focal length are shown for selected values of β .

β	<i>S</i>	Distortion for		
		<i>EFL</i> of 150.0000 mm.	<i>CFL</i> of 149.9811 mm.	<i>CFL</i> of 149.9868 mm.
	mm.	mm.	mm.	mm.
0	0.0000	0.0000	0.0000	0.0000
7.5	.0004	-.0001	.0024	.0016
15	.0018	.0005	.0046	.0030
22.5	.0043	.0018	.0060	.0037
30	.0083	-.0048	.0061	.0028
37.5	.0147	-.0113	.0032	-.0012
40	.0176	-.0148	.0010	-.0037
45	.0250	-.0250	-.0061	-.0118

used in determining the prism effect in lenses can be used for this purpose.

The results of measurement have been analysed for 12 cameras using 1/16 inch glass plates, 7 cameras using 1/8 inch glass plates, and 7 cameras using 1/4 inch glass plates. All cameras were equipped with lenses having a focal length of six inches. The probable errors of calibrated focal length for a single camera were found to be ± 0.020 mm. for the 1/16 inch plates;

3. ANALYSIS OF ERRORS IN FOCAL LENGTH AND DISTORTION FOR SEVERAL METHODS OF CALIBRATION

In section 2.2, it was shown that, for the case of lens, focal plane, and targets properly positioned with respect to one another, the errors in focal length and distortion arise from errors in the measurement of the test negative and errors in the measured value of the angles separating the the targets.

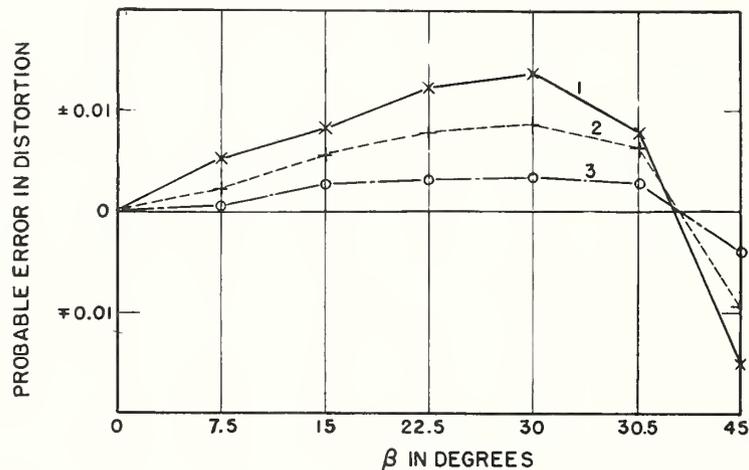


FIG. 2. Probable error in distortion versus angle for three plate thicknesses. Curve 1 shows the variations in the probable error in distortion of 1/16" plates; curve 2 shows results for 1/8" plates; and curve 3 shows the results for 1/4" plates.

± 0.008 mm. for the 1/8 inch glass plates; and ± 0.004 mm. for the 1/4 inch glass plates. The values of the probable errors in distortion for a single camera are shown in Figure 2. It is interesting to note the striking reduction in the error with increasing thickness of the photographic plates. This is undoubtedly a consequence of the departures from planeness becoming progressively less as the thickness of the plates increase. It is clear from these curves that the contribution to the error in distortion is appreciable for both the 1/16 and 1/8 inch glass plates, and that the 1/4 inch plates are the best of the three for use in camera calibration. All three varieties of plates were initially selected for planeness so the analysis indicates that the thicker plates are less likely to warp or depart from their initial state of planeness than the thinner plates. Consequently in this type of work, it is usually advantageous to use thick plates.

In this section the magnitudes of the probable errors that arise from these sources are computed for several methods of lens and camera calibration. The methods are (a) precision lens testing camera, (b) camera calibrator, and (c) field calibration method. In making these computations, the magnitudes assigned to the errors are those which are reported by the various operators. It is further assumed that the errors arising from plate curvature of the type described in section 2.3 can be neglected. This assumption is justified as the primary aim of these computations is for the purpose of comparing the reliability of these methods. The presence of appreciable plate curvature would impair the accuracy to approximately the same extent in all three methods.

3.1 PRECISION LENS TESTING CAMERA

This is the instrument used by the National Bureau of Standards for

TABLE 7

THE PROBABLE ERROR IN AN EQUIVALENT FOCAL LENGTH DETERMINED WITH THE PRECISION LENS TESTING CAMERA

In the computation of Δf , it is assumed that $\Delta r = \pm 0.002$ mm.; $\Delta\beta = \pm 3$ seconds per 5° angle; and $f = 150$ mm.

β	Δr	$\Delta\beta$	Δf_r	Δf_β	Δf
degrees	mm.	seconds	mm.	mm.	mm.
5	± 0.002	± 3.0	± 0.023	± 0.025	± 0.033
10	± 0.002	± 4.2	± 0.011	± 0.018	± 0.021

photographically determining the focal length and distortion of lenses intended for use in airplane mapping cameras (7). Two negatives are usually made which together cover a single diameter. Measurements are made along each of the two radii and the results are averaged. The probable error in the determination of angle between adjacent collimators is believed to not exceed ± 2 seconds but for the purpose of the following calculations, a more conservative estimate of ± 3 seconds is used. Because the angles between adjacent collimators are measured separately, the probable error in the angle between the first and third collimator is $\pm 3\sqrt{2}$ seconds; between the first and fourth, it is $\pm 3\sqrt{3}$ seconds, and so on. The probable error of measurement of a distance on the negative does not exceed ± 0.002 mm. for moderately good images.

To compute the focal length, the distance between the 0° and 5° images is used for one determination, and the distance between the 0° and 10° images is used for the second determination. The final value of f is found by taking a weighted average of the values obtained for $\beta = 5^\circ$ and $\beta = 10^\circ$. It is customary to assign weights to the separate determinations that are inversely proportional to their probable errors. The probable errors of f_{5° and f_{10° are determined from Tables 1 and 2 for the given values of β , Δr , and $\Delta\beta$. The computed values of Δf are shown in Table 7.

The probable error, Δf , of the weighted average of f_{5° and f_{10° is given by the relation

$$\Delta f = \pm \frac{1}{\sqrt{\left(\frac{1}{\Delta f_{5^\circ}}\right)^2 + \left(\frac{1}{\Delta f_{10^\circ}}\right)^2}} \quad (24)$$

Using the values in Table 7 in this equation, the probable error in f for a single negative is

$$\Delta f = \pm 0.018 \text{ mm.} \quad (25)$$

As two negatives are usually made, the above value is divided by $\sqrt{2}$, so that the final value of the probable error for the average of the two negatives is

$$\Delta f = \pm 0.013 \text{ mm.} \quad (26)$$

The probable errors in the distortion for each value of β are computed in accordance with the procedure shown in section 2.2 (b). Table 8 shows the value of ΔD_r , the probable error arising from errors in the measurement of r ; ΔD_f , the probable error arising from errors in the determination of f ; and ΔD_β , the probable error arising from errors in β . Table 4 is used in determining the values of ΔD_f and ΔD_β for the given conditions, ΔD_r remaining constant. The final value of ΔD for a single negative is obtained by finding the square root of the sum of the squares of each error as shown in eq. 14. The final value for two negatives is shown in the column headed ΔD for $n = 2$.

Because of the manner in which the angles are measured, there is little gain in using a calibrated focal length in evaluating the probable errors in distortion. In this particular case, when the approximate calibrated focal length is based on $D = 0$ for $\beta = 40^\circ$, the probable error Δf becomes ± 0.009 instead of ± 0.013 mm. In turn this reduces ΔD to ± 0.009 mm. and ± 0.011 mm. at $\beta = 40^\circ$ and $\beta = 45^\circ$ respectively.

In the past few years precision theodolites have become readily available that permit the measurement of β within 2 seconds of arc. With an instrument of this type, the angle separating the first collimator and any of the other collimators can be measured directly. Consequently there is no cumulative error such as occurs when each 5° interval is measured separately and $\Delta\beta$ is the same for each

TABLE 8

THE PROBABLE ERROR, ΔD , IN DISTORTION FOR MEASUREMENTS MADE WITH THE PRECISION LENS TESTING CAMERA FOR VARIOUS ANGULAR SEPARATIONS (β) FROM THE AXIS

These values of the probable error are for a lens having an equivalent focal length of 150 mm. It is estimated that $\Delta f = \pm 0.013$ mm.; $\Delta r = \pm 0.002$ mm.; and $\Delta\beta = \pm 3$ seconds for each 5° interval.

β	$\Delta\beta$	ΔD_r	ΔD_f	ΔD_β	$\frac{\Delta D}{n=1}$	$\frac{\Delta D}{n=2}$
degrees	sec.	mm.	mm.	mm.	mm.	mm.
5	3.0	0.002	0.001	0.002	0.003	0.002
10	4.2	.002	.002	.003	.004	.003
15	5.2	.002	.003	.004	.005	.004
20	6.0	.002	.005	.005	.007	.005
25	6.7	.002	.006	.006	.009	.006
30	7.3	.002	.008	.007	.011	.008
35	7.9	.002	.009	.008	.012	.008
40	8.5	.002	.011	.010	.015	.011
45	9.0	.002	.013	.013	.018	.013

value of β . If the error $\Delta\beta$ is assumed to be ± 4 seconds, no reduction in the error in equivalent focal length is found but the error in distortion is reduced, the maximum error falling to ± 0.010 mm. at $\beta = 45^\circ$ for the average obtained from determinations for two negatives. If the calibrated focal length for $\beta = 40^\circ$ is used, the error in *CFL* falls to ± 0.006 mm. as does the error in distortion for $\beta = 45^\circ$ for $n = 2$.

3.2 CAMERA CALIBRATOR

The camera calibrator (8) was developed at the National Bureau of Standards and has been in use since the latter part of 1949. It was designed especially to simplify calibration of precision airplane mapping cameras; to increase the accuracy of measurement of focal length, radial distortion, tangential distortion, and the location of the principal point; and to reduce markedly the time required for calibration. It has performed in accordance with the expectations. Moreover, it is a compact piece of equipment so designed that all work is performed in the laboratory under controlled conditions. The cost of construction is relatively low. The calibration is sufficiently simple that the instrument can be recalibrated in the course of a day. Because of its rugged and compact nature and the simplicity of recalibration, it could easily be transported from one place to another and put into operating condition following the move within a very few days.

In the course of calibration, it is customary to determine the equivalent focal

length and distortion of the lens as mounted in the camera. A single negative, made with a camera on the calibrator, contains images at 7.5° intervals along four radii from the centers to the four corners of the square image area. Measurements are made along each of the four radii and the results averaged. While all of the necessary information can be obtained from a single negative, a second negative is usually taken particularly when measurements on tangential distortion and prism effect are required.

There are now two methods available for determining the angle β . In the first method, angles separating adjacent collimators are measured one at a time using a calibrated reflecting prism. The angles separating the central collimator and any of the other collimators are obtained with consequent increase in the error $\Delta\beta$ with increasing β . In the second method, the total angle β is measured in one step with the aid of a precision theodolite.

Table 9 shows the resultant error in focal length for a lens having a focal length of 150 mm. for $\Delta r = \pm 0.002$ mm. and $\Delta\beta = \pm 4$ seconds for each value of β for both methods of determining β . The individual value of Δf_r and Δf_β are determined with the aid of Tables 1 and 2. The column, headed $\Delta f(n=1)$, gives the magnitude of the probable error in focal length for a determination based on measurements along a single radial bank. The column headed $\Delta f(n=4)$, gives the magnitude of the probable error in focal length based on the average obtained from measurements along

TABLE 9

PROBABLE ERROR, Δf , IN THE FOCAL LENGTH DETERMINATIONS FOR THE CAMERA CALIBRATOR

For the computations, it is assumed that $f=150$ mm.; that $\Delta r = \pm 0.002$ mm.; that $\Delta\beta = \pm 4$ seconds per 7.5° interval in the first part of the table and that $\Delta\beta = \pm 4$ seconds per angle in the second part of the table. The calculations are for a single negative. If two negatives are used, the error of the average is 0.7 of the value listed for $r=4$.

β	$\Delta\beta$	Δf_r	Δf_β	Δf	Δf
	$\Delta\beta = \pm 4$ seconds per 7.5° angle			$n=1$	$n=4$
degrees	seconds	mm.	mm.	mm.	mm.
7.5	4	± 0.015	± 0.022	± 0.207	± 0.014
15	5.6	.007	.016	.017	.009
22.5	6.9	.005	.014	.015	.008
30	8.0	.003	.013	.013	.007
37.5	8.9	.003	.013	.013	.007
45	9.8	.002	.014	.014	.007

$\Delta\beta = \pm 4$ seconds per angle					
7.5	4	± 0.015	± 0.022	± 0.027	± 0.014
15	4	.007	.012	.014	.007
22.5	4	.005	.008	.009	.005
30	4	.003	.007	.008	.004
37.5	4	.003	.006	.007	.003
45	4	.002	.006	.006	.003

the four radii. If the results from two negatives are averaged, the resultant error is reduced by the ratio of the square root of two.

For an equivalent focal length determination for a lens having appreciable distortion, the values of Δf for $\beta=7.5^\circ$ is assigned. For lenses having near zero distortion, a larger value of β can be used with a resultant decrease in Δf . In assigning the error in a calibrated focal length, the value of Δf nearest to the outermost zero point of distortion may be properly used. It is clear from a consideration of these tables that the error in calibrated focal length arising from these magnitudes of error in r and β is almost negligible.

The probable errors in the values of distortion, based on the assumed errors in r and β , are listed in Table 10. The error in equivalent focal length used in the computation is that associated with $\beta=7.5^\circ$. The tabulation is based on the first method of measurement of β with the consequent increase in $\Delta\beta$ with β . The values of ΔD are given for a single radial bank under the column headed, $n=1$; and the average for the four radial banks is given under the column, $n=4$. The table lists the error for the least favorable conditions of the given error in r and β . If the distortion is based

on the calibrated focal length, there is a reduction in the maximum value of ΔD . Also, if $\Delta\beta$ is assumed constant, the error is still further decreased as shown in Table 11.

3.3 FIELD CALIBRATION METHOD

The field calibration method (9) employs a calibration range consisting of a camera station and a number of targets spaced along a line at a moderately great distance therefrom. This distance is usually so great that it is believed that the imagery is comparable to that which usually obtains for infinitely distant targets. When targets at a finite distance are used there is always the possibility of an error arising because the camera does not occupy the correct position with respect to that occupied by the central axis of the theodolite when the angle was measured. This is the supreme advantage of the infinitely distant target. Neglecting spherical aberration of collimator, the value obtained is independent of placement of cameras so long as the aperture is filled.

In performing a test, the camera is so placed that the image of the line of targets falls along one of the diagonals of the focal plane frame. The camera is aimed at the central target by approximate methods.

TABLE 10

THE PROBABLE ERROR, ΔD , IN DISTORTION FOR MEASUREMENTS MADE WITH THE CAMERA CALIBRATOR FOR VARIOUS ANGULAR SEPARATIONS FROM THE AXIS

These values of the probable error are for a lens having an equivalent focal length of 150 mm. It is estimated that $\Delta f = \pm 0.014$ mm.; $\Delta r = \pm 0.002$ mm.; and $\Delta\beta = \pm 4$ seconds for each 7.5° interval.

β	$\Delta\beta$	ΔD_r	ΔD_f	ΔD_β	$\frac{\Delta D}{n=1}$	$\frac{\Delta D}{n=4}$
degrees	sec.	mm.	mm.	mm.	mm.	mm.
7.5	4.0	0.002	0.003	0.002	0.004	0.002
15	5.6	.002	.004	.004	.006	.003
22.5	6.6	.002	.006	.006	.009	.004
30	8.0	.002	.008	.008	.012	.006
37.5	8.9	.002	.010	.011	.015	.008
45	9.8	.002	.014	.014	.020	.010

These errors are further reduced if an average is made for two negatives.

The angles separating the central target from the other targets are measured with an accurate theodolite. The probable error in the determination of the angle between selected targets is believed to be not in excess of ± 5 seconds. The probable error of measurement of distance on a test negative does not exceed ± 0.003 mm. Targets separated by 12.5° from the central target are used in determining the equivalent focal length. So for a single determination given that

$$\Delta\beta = 5 \text{ seconds} \quad (27)$$

$$\Delta r = 0.003 \text{ mm.} \quad (28)$$

the values of the probable error from Tables 1 and 2 for $\beta = 12.5^\circ$ are

$$\Delta f_r = 0.014 \text{ mm.} \quad (29)$$

$$\Delta f_\beta = 0.017 \text{ mm.} \quad (30)$$

whence

$$\Delta f = \pm 0.022 \text{ mm.} \quad (31)$$

Two determinations of the equivalent focal length are made from a single negative, so the probable error for one diagonal is

$$\Delta f = \frac{\pm 0.022}{2} = \pm 0.016 \text{ mm.} \quad (32)$$

The probable errors in the distortion have been computed on the basis of the error in r and β , reported for the field calibration method, and the results are shown in Table 12. The values are slightly higher than those present in the camera calibrator method. Some reduction in the error is possible by computing with respect to a calibrated focal length but not as much as for the two previous methods because the equivalent focal length is

TABLE 11

THE PROBABLE ERROR, ΔD , IN DISTORTION FOR MEASUREMENTS MADE WITH THE CAMERA CALIBRATOR FOR VARIOUS ANGULAR SEPARATIONS (β) FROM THE AXIS

The distortion values are based on a calibrated focal length of 150 mm. calculated to yield zero distortion for $\beta = 40^\circ$. It is estimated that $\Delta f = \pm 0.003$ mm.; $\Delta r = \pm 0.002$ mm.; and $\Delta\beta = \pm 4$ seconds.

β	$\Delta\beta$	ΔD_r	ΔD_f	ΔD_β	$\frac{\Delta D}{n=1}$	$\frac{\Delta D}{n=4}$
degrees	sec.	mm.	mm.	mm.	mm.	mm.
7.5	± 4.0	± 0.002	± 0.000	± 0.003	± 0.004	± 0.002
15	4.0	.002	.001	.003	.004	.002
22.5	4.0	.002	.001	.003	.004	.002
30	4.0	.002	.002	.004	.005	.002
37.5	4.0	.002	.002	.005	.006	.003
45	4.0	.002	.003	.006	.007	.004

TABLE 12

THE PROBABLE ERROR, ΔD , IN DISTORTION FOR MEASUREMENTS MADE WITH THE FIELD CALIBRATION METHOD FOR VARIOUS ANGULAR SEPARATIONS (β) FROM THE AXIS

These values for the probable error are for a lens having an equivalent focal length of 150 mm. It is estimated that $\Delta f = \pm 0.016$ mm.; $\Delta r = \pm 0.003$ mm.; and $\Delta\beta = \pm 5$ seconds.

β	ΔD_r	ΔD_f	ΔD_β	ΔD $n=1$	ΔD $n=2$
degrees	mm.	mm.	mm.	mm.	mm.
5	0.003	0.001	0.004	0.005	0.004
10	.003	.003	.004	.006	.004
15	.003	.004	.004	.006	.004
20	.003	.006	.004	.008	.006
25	.003	.007	.004	.009	.006
30	.003	.009	.005	.011	.008
35	.003	.011	.005	.012	.008
40	.003	.013	.006	.015	.011
45	.003	.016	.007	.016	.011

initially based on a large value of β .

The field method of camera calibration when performed carefully should yield reliable values of the calibrated focal length and distortion. It can give reliable information on the location of the focal point in the absence of prism effect. It is, however, deficient in some respects, although in most instances these defects do not seriously impair the accuracy of the calibration. The features most subject to criticism are finite distance of targets and alignment of camera.

a. Finite distances of targets

The fact that the targets are located approximately 400 ft. from the camera station places the film plane of the camera 0.188 mm. nearer to the lens than the true image plane, assuming the camera is set at infinite focus. It is probable that this will not materially affect the focal length and distortion determinations for the film plane of the camera. However, it is likely to produce some changes in the values of resolving power. This effect will be increased with increasing focal length of the lens, and for 36 inch lenses it will mean evaluating the resolving power for a plane 1 mm. inside the best image plane.

b. Alignment of camera

An auto-collimating telescope should be used to make the focal plane of the camera normal to the line of sight between telescope and central target. When this is not done, the camera is almost certain to be

tipped with respect to the line of sight and asymmetrical distortions are sure to result. This has been compensated in the field method by operating on the data to find a point of symmetry. These calculations, in the absence of prism effect, serve to locate the focal point of the camera (referred to as the center cross by the present writer and also referred to as the principal point of auto-collimation in recent literature (3)). Since only one diagonal of the camera is used at a time, it is probable that the camera is also tipped about a horizontal axis as well as the vertical. It is possible that this tipping of unknown magnitude may sometimes affect the final results, although such effects will generally be very small. For example, a one degree tip about the horizontal axis will produce an increase of 0.02 mm. in the measured value of the focal length of a 150 mm. lens.

While the location of the focal point, in the absence of prism effect, is sufficiently accurate for the purpose by this method, it is accomplished by difficult and time consuming computation. The greater part of this computation can be avoided by using the auto-collimating telescope in aiming the camera.

When prism effect is present, this method locates a point about which the distortion values at a specified angle are equalized. Asymmetries will still exist at all other angles although they are reduced from the values originally found. Fortunately the prism effect in modern cameras

is so small that the remaining assymetries are usually small.

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The Testing of Photographic Lenses at the National Bureau of Standards

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ABSTRACT—A summary of the methods employed at the National Bureau of Standards for the evaluation of photographic lens performance is given. This includes a brief description of equipment such as the precision lens testing camera and the camera calibrator, developed for the determination of equivalent focal length, distortion, and resolving power.

The methods used in determining the resolving power of photographic lenses are discussed. These include the original high contrast transparency, the charts issued as part of Circular C428, the variable contrast chart and the charts recently issued which form part of Circular 533. The subject of high and low contrast charts is considered briefly. Objective methods of evaluating lens performance such as microdensitometric analysis of negatives and electronic scanning procedures are discussed.

Introduction

The National Bureau of Standards has been conducting tests on photographic lenses and devising methods for evaluating the lens constants and the image forming qualities for many years. The early methods of test were predominantly visual. As the science of photography advanced, visual methods of evaluating the performance of lenses intended for use with photographic emulsions were superseded by photographic methods so that lenses would be tested under conditions more closely approximating the conditions of use. The photographic method has

proved eminently successful and many pieces of equipment have been developed at this Bureau to facilitate the accurate evaluation of lens performance by this means.

Precision Lens Testing Camera

The precision lens testing camera, shown in Fig. 1, was developed in the early 1930's by Gardner and Case.¹ The instrument is still in use, although numerous modifications have been made on it through the years, the principal changes being the addition of three collimators to extend the half angular-field to 45°, better methods of light control, and an improved camera back. The instrument now consists of 10 collimators, spaced at 5° intervals, which effectively provide infinitely distant targets within the confines of the laboratory. Each collimator is

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equipped with a resolution chart, the original form of which is shown in Fig. 2. By means of appropriate controls, it is possible with this equipment to explore the range of usable imagery and to select the plane of best average definition for the lens, which plane for many lenses does not necessarily coincide with the plane of best visual focus. On the test negative, a complete photographic record is obtained of the quality of imagery throughout the field from which the curvature of the image field can be readily determined.

After selection of the row of images lying in the plane of best average definition, the separations of these images are measured. From these measurements, the equivalent focal length and distortion of the lens are calculated. The equivalent focal length is one of the lens constants of principal interest to the user; it is the scale factor used in interpreting distance measurements on the negative. Unfortunately, the equivalent focal length, or scale factor, is not invariant over the entire picture area, so that a value of the equivalent focal length that is valid in the

central area may not give a true measure in the outer areas. This failure of the lens to yield a true perspective projection of the area photographed is called distortion and was formerly one of the principal causes of rejection of lenses intended for use in aerial mapping. No lens is completely free from distortion, so it is customary to establish tolerances and to accept lenses having distortion within the tolerance range.

For some years, the determination of equivalent focal length, back focal distance, distortion, and resolving power satisfied the needs of the users. In the meantime the science of photogrammetry was advancing and a demand arose for calibrating the lens in the actual aerial mapping camera. This new calibration included not only the previous information but also the exact location of the principal point with respect to four index markers located on the focal plane frame. For an ideal lens, the principal point is the point of intersection of a ray, normal to the focal plane from an infinitely distant object. Initially this work was performed on the precision lens testing camera.²

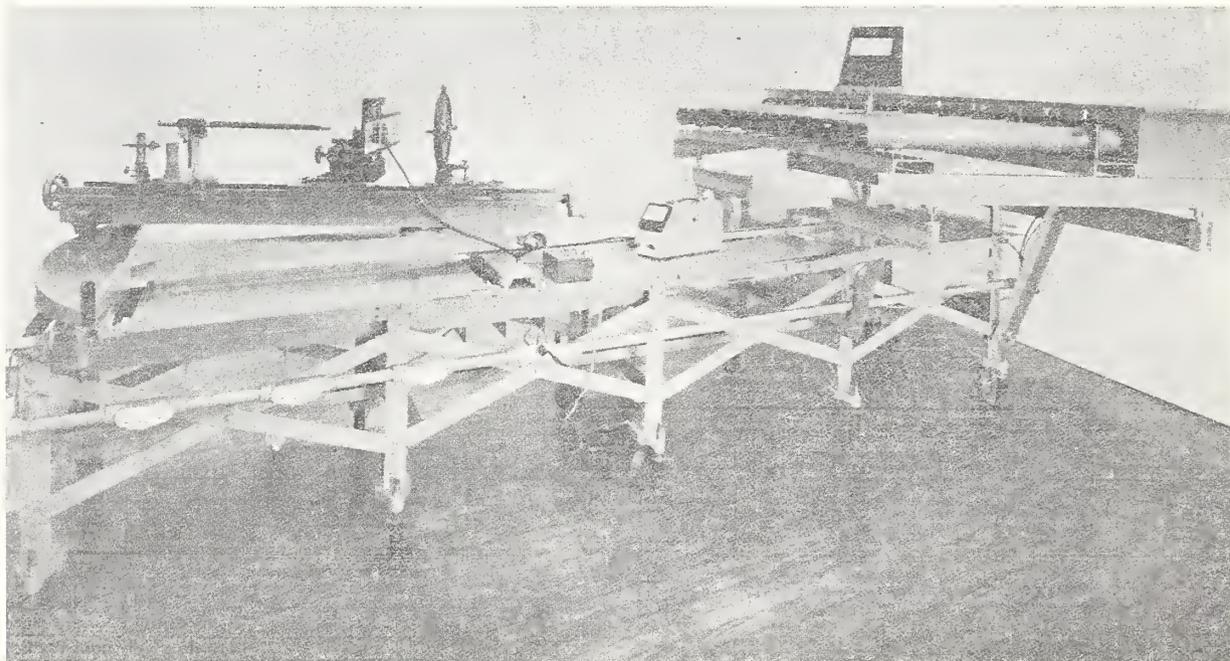


Fig. 1—Precision lens testing camera. This photograph shows the new camera back with a light-metering device in place. The values of illumination in the focal plane are read from the large meter above the collimator bank.

TESTING PHOTOGRAPHIC LENSES

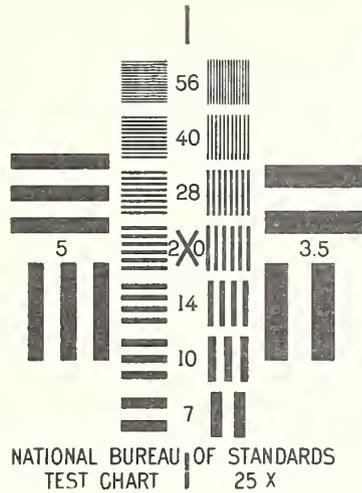


Fig. 2—High contrast resolution chart. A transparency of this chart is provided for each collimator. The ratio of the line spacings in successive patterns is equal to $\sqrt{2}$. The range of resolving power is dependent upon the focal length of the lens tested; conditions are usually such that the range is from 3.5 to 56 lines/mm in the image plane.

Camera Calibrator

It was a difficult and time consuming task to conduct this type of calibration on the lens testing camera, and thus the camera calibrator³ was developed, from sheer necessity, and put into operation in January 1949. This instrument, shown in Fig. 3, embodied many of the features employed in the precision lens testing camera. It is however designed so that the camera under test points vertically down thus simplifying its orientation with respect to the central collimator. It is equipped with 25 collimators arranged in the form of a cross. Each collimator is provided with a resolution chart such as shown in Fig. 4. This chart differs from the chart shown earlier in that the spacings between the lines of adjacent patterns differ by the $\sqrt[4]{2}$ instead of $\sqrt{2}$. For a six inch lens, the range of resolving power covered is from 4.1 to 289 lines/mm. The cross in the center of the chart is the fiducial mark with reference to which the angles separating the collimators are measured. When imaged on a camera test negative, the measured sep-

arations of the crosses serve for the determination of equivalent focal length and distortion.

A schematic drawing of a typical negative is shown in Fig. 5. The location of the collimation index markers of the camera are registered on the photographic plate at the same time as the collimator targets. Consequently, a single negative contains all the information necessary for the calibration of the precision camera. Two negatives are usually taken however with the camera rotated 180° between exposures and the results averaged to insure accuracy.

The location of the principal point with respect to the collimation index markers of a precision camera is complicated to some extent by the presence of prism effect. This effect may arise from lack of plane parallelism of the surfaces of the lens filter or from small decentration of the lens elements. It is possible from measurements on the negative to compute the displacement of the principal point from its apparent position and to make allowance for it in the course of calibration. This is done for all precision cameras calibrated in our laboratory.

Distortion is vitally important to the users of precision mapping cameras and the usual definition of distortion referred to the equivalent focal length⁴ which yielded low values of distortion in the central area was soon discontinued in favor of a definition of distortion so determined as to minimize the error over the entire picture area. This is the distortion referred to the calibrated focal length. It is perhaps not generally realized that the shape of the distortion curve one obtains during a calibration is affected by the region of the negative used in determining the focal length. This shape variation is shown in Fig. 6. Here, the values of the focal length are based successively upon the distances separating the images at 0 to 7.5 degrees; 0 and 15 degrees; and so on. The distortion is then determined for each value of focal length and the results plotted. The commonly accepted value of the distortion referred to the calibrated focal length is shown at the bottom of the figure. It is clear that for this lens, this is equivalent to basing the distortion upon the focal length determined by the separation of images located at 0 and 42.5.

Resolving Power Test Methods

While the metrical characteristics of photographic lenses are of primary importance in the field of precision cameras, the subject of quality of definition has also been of considerable interest. Numerous attempts have been made to achieve a simple objective method of evaluating the performance of a lens so far as its image forming characteristics are concerned. To date, the measurement of its resolving power, which is one of the

earliest methods, continues as the simplest and best. It is very possible that a better method, than those now used, will some day be developed; but until such time, we are obliged to continue to use resolving power as a basis upon which to judge lens performance.

There have, however, been major improvements in the methods of measurement of resolving power. At first, the resolving power was determined visually with high contrast targets but it early became evident that the results so obtained



Fig. 3—Camera calibrator. Instrument as arranged for use. The camera under test points vertically downward. Its proper orientation is affected by the auto-collimating telescope and the plane-parallel piece of glass placed on the focal plane of the camera. Images on a finished negative appear every 7.5° along each diagonal, with the collimation index marks registered on each side of the negative.

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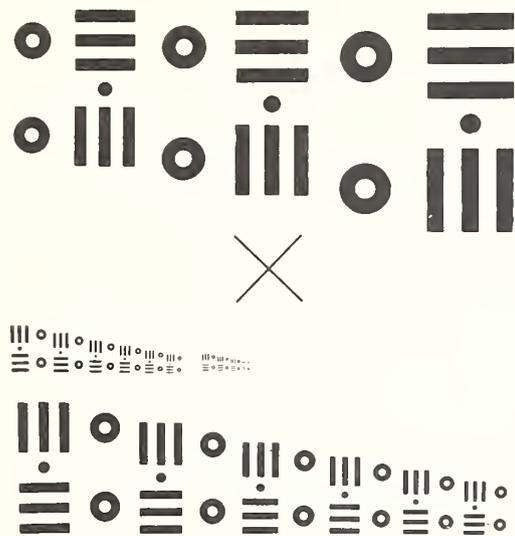


Fig. 4—Test chart for camera calibrator. The test chart that is used in preparing the reticle for use in each collimator consists of patterns of parallel lines in two orientations with spaces varying in geometric progression by steps equal to $\sqrt[4]{2}$. The circle targets are included for comparative study of line and circle targets.

were not well suited to the evaluation of the performance of a photographic lens as used in practice. When the precision lens testing camera was built at the National Bureau of Standards, resolution charts were used as targets of the collimators. The resolution, as recorded in the negative image, was thereafter determined throughout the image field for each lens submitted for test. The resolution, thus determined, is of course the resolution of a lens-emulsion combination and not of the lens alone. However, since the photographic lens is always used with an emulsion, the values of resolving power so obtained are a reliable measure of the performance of the lens for the particular lens-emulsion combination.

From the time the routine measurement on resolving power was first initiated at the Bureau to the present, it has been the practice to use a high contrast transparency as a test chart and a fine grained emulsion (such as Eastman Spectroscopic Plate, type V-B) to record the imagery. It is true that these conditions of test do

not coincide with the conditions generally prevailing during the practical use of the lens. For example, in practice the contrast between discrete objects in the area photographed is rather low and high speed coarse grain emulsions are used. However, there is as yet not sufficient justification to warrant discontinuing the present methods in favor of methods that more nearly approximate the conditions of use. The use of the high contrast target and fine grain emulsion leads to higher measured values of the resolving power with consequent greater variation among lenses and makes easier the rating of the relative performance of different lenses.

NBS Resolution Test Chart of 1940

In the late 1930's, the Bureau was constantly receiving inquiries from camera users concerning the best method of evaluating the quality of lenses used in small hand cameras. Because of this wide-spread interest in lens performance, Dr. Gardner prepared the circular C428 entitled "A Test of Lens Resolution for the Photographer" which was issued in 1940.⁵ This circular contained two sheets of six high contrast resolution charts, printed on glossy paper and complete instructions for use. The resolution charts were copies of the standard resolution chart, shown in Fig. 2, and were printed by the Bureau of Printing and Engraving. These charts have enjoyed wide popularity and have proved extremely useful.

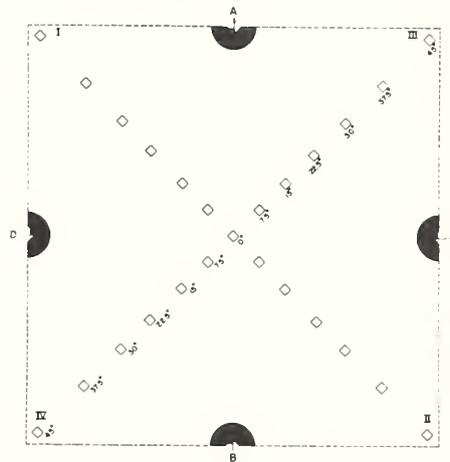


Fig. 5—Schematic drawing of test negative obtained with camera calibrator.

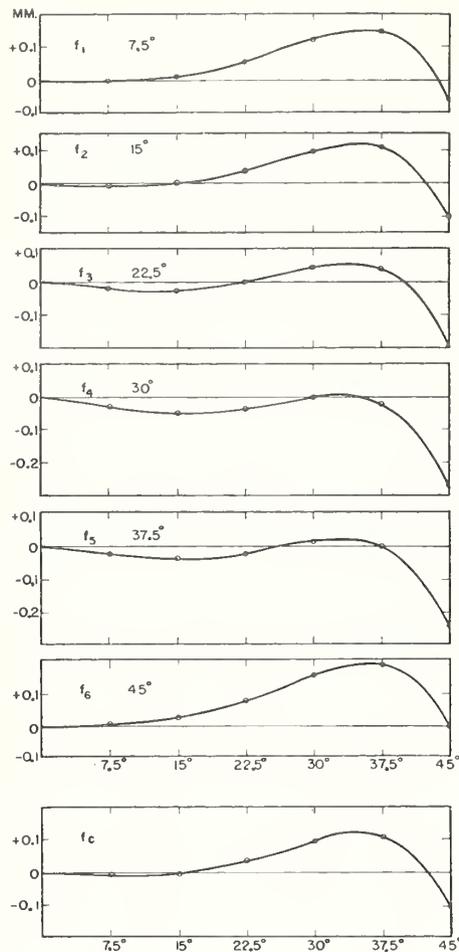


Fig. 6—Variation of distortion in the same image plane with equivalent focal length computed from the relation $f = y / \tan \beta$ for varying angles of β . The lowest curve showing the distortion referred to the calibrated focal length indicates that the calibrated focal length is equal to the equivalent focal length for values of β of approximately 16° and 42.5° .

During the early 1940's, the measurement of resolving power by photographic methods began to receive greater emphasis in other laboratories. Different types of charts were evolved and different conditions of test were imposed. In England, the two-line low contrast Cobb chart⁶ was developed and is still used. In Canada, the annulus or ring low contrast chart⁷

was developed. The U. S. Air Force developed its own high contrast 3-line chart.⁸ In these other laboratories, it is customary to use coarse grain high speed emulsion to record the imagery. The subject of contrast in the target is emphasized and low contrast charts are sometimes preferred because the contrast in natural objects is low. In addition, different series of line spacings were used. For most charts, the successive fine patterns reduce in size by some geometric ratio. For the NBS chart of 1940, this ratio is $\sqrt{2}$. The first Air Force chart used a ratio of $\sqrt[3]{2}$. For the Cobb chart, the ratio is $\sqrt[4]{2}$. The aim of using a smaller ratio is the hope of achieving greater accuracy in determining the limit of resolution for a particular lens-emulsion combination.

There are sound reasons favoring the use of each of the many types of charts developed. Each contributed toward the better understanding of photographic imaging processes and there is difficulty in deciding which, if any, of the various charts and test processes are to be preferred. In reviewing the various types of charts used, it may be noted that visual examination of the negative image is required generally in the course of the analysis of lens performance. The usual resolving power test may accordingly be regarded as subjective. There is likelihood of different criteria being applied by different observers and consequent variation in the results reported by various laboratories. For example, some observers declare a pattern resolved only if the lines in it and all coarser patterns are sufficiently well defined so that the separate lines are clearly distinguishable and readily counted. Other observers are content to call a pattern resolved if one can determine with reasonable certainty the orientation of the lines in the pattern. When one examines a photographic image of a resolution chart, it is clearly evident that the contrast between the lines and spaces in successively finer patterns decreases steadily as one proceeds from the coarse to the fine patterns. At the limit of resolution of the lens emulsion combination, the contrast between line and space becomes zero. However, the contrast between line and space is frequently so low for several patterns coarser than that determined by the limit of resolution that

it is extremely difficult to state with certainty at just what pattern the limit is reached.

The Variable Contrast Chart

Because of the general interest in the effect of object contrast on lens resolution, Dr. Gardner suggested, in the early part of 1950, the use of a variable contrast chart.⁹ With a chart of this nature, it is possible to evaluate the performance of a lens for a series of contrasts ranging from high to low with a single exposure on a single plate. One is thus certain that identical conditions prevail throughout the test for both the high and low contrast targets.

Figure 7 shows the master high contrast chart that is used in making the variable contrast chart.¹⁰ The line fre-

quency is a linear function of the distance measured across the lines. The zero of the chart is at the inner edge of the first broad dark line. The range of this chart is from 0.2 to 2.0 lines/mm.

The variable contrast chart is made by contact printing the high contrast target on a photographic plate under conditions such that the exposure time varies in a predetermined manner. The high contrast target is then removed and a second exposure is given to the photographic plate in such a manner that the transmittance averaged for a small number of lines and spaces is constant over the entire negative. This double exposed plate is then processed, and the finished negative is the final master target having continuously varying contrast in the direction along the lines. A typical variable con-

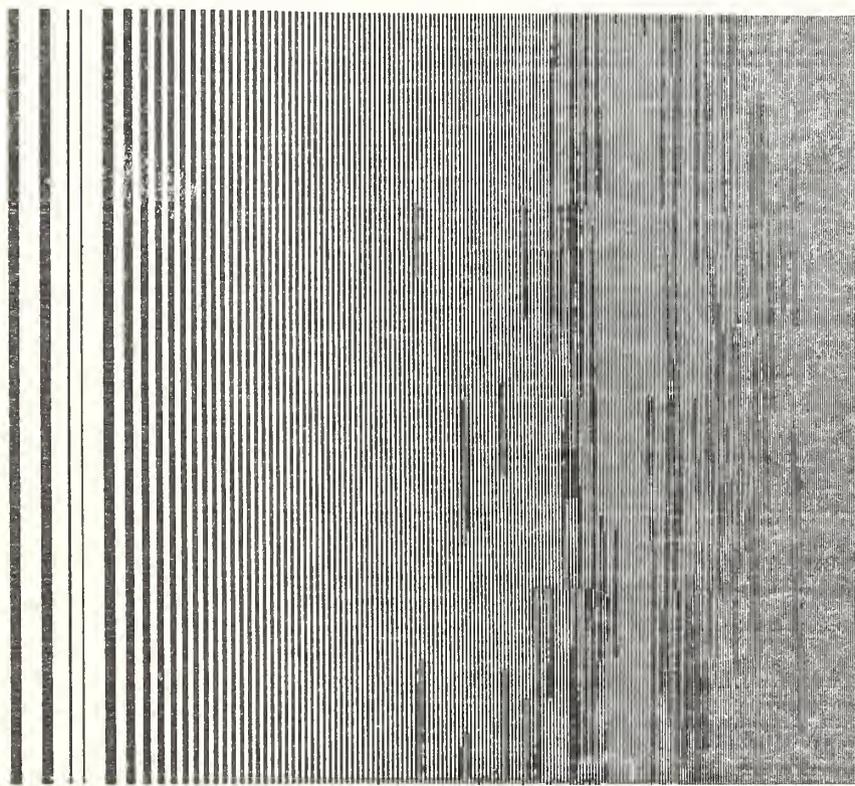


Fig. 7—Master high contrast test chart. The line frequency is a linear function of the distance measured across the lines. The zero of the chart is at the inner edge of the first broad dark line. The range of this chart is from 0.2 to 2.0 lines/mm.

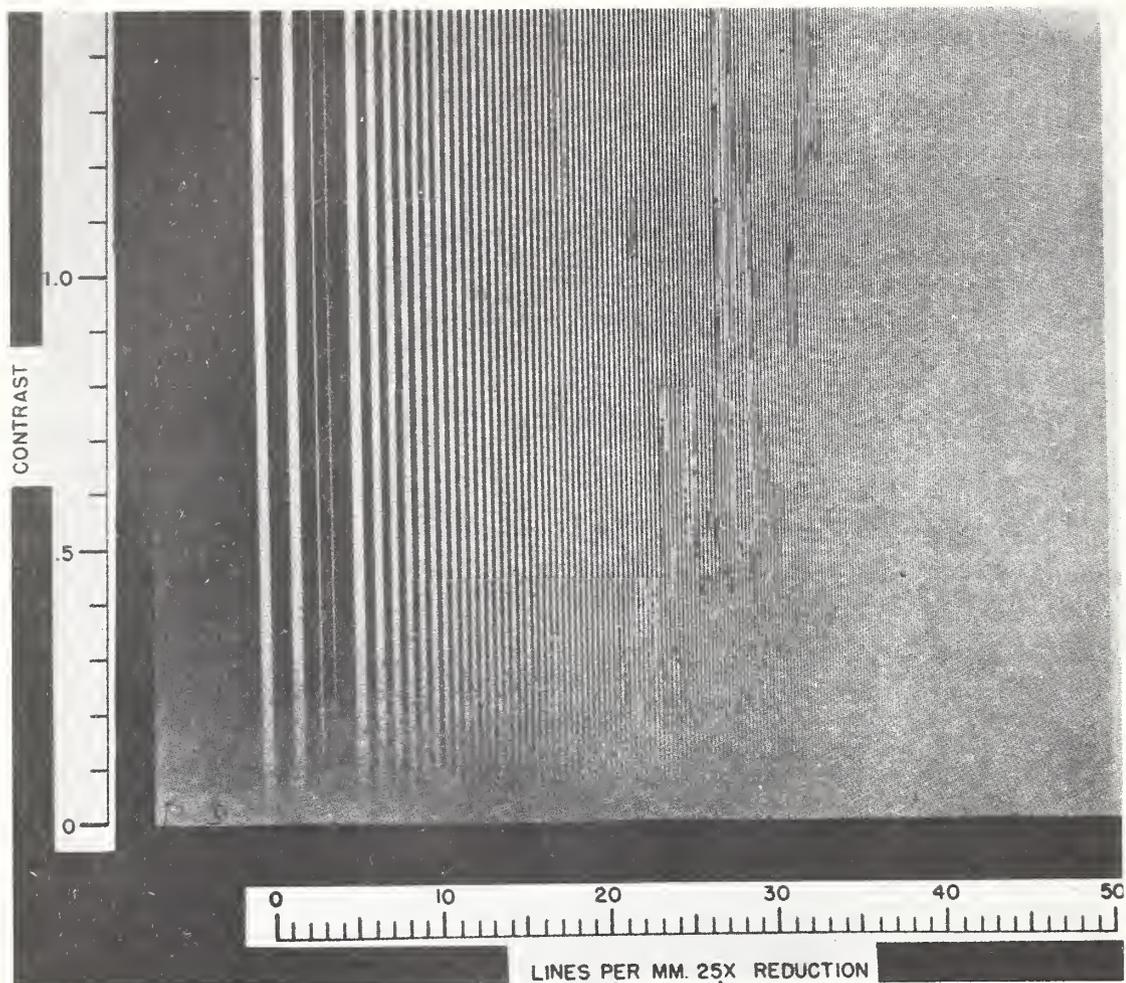


Fig. 8—Variable contrast master test chart. In addition to the features contained in the master high contrast test chart, the contrast varies in a linear manner from 0 to 1.5. The "moire" effect that appears in the illustration is produced by the interaction of the periodic pattern of the half-tone screen and the pattern of continuously varying line spacings in the chart. Consequently, the above figure is not a true reproduction of the chart, although it is a fairly close approximation in some areas.

trast target is shown in Fig. 8. For this particular target, the scale at the left marked "Contrast" shows that in the original transparency the contrast varies in linear manner from 0 to 1.5. Contrast, as here used, is simply the difference in photographic density between dark and light areas as measured on a densitometer. The lower scale gives the line frequency or resolving power in lines/mm at a 25X reduction. For the chart illustrated, the

marked scale is correct only when the distance from 0 to 50 is equal to 8.0mm in the image on the test negative.

The chart is used in the following manner. The target is set up at $(M+1)$ focal lengths in front of the test lens, where M is the desired reduction ratio of object to image size. It is lighted from the rear by a broad uniform source and negatives are made using a given emulsion with the lens set at a selected aperture. Figure

9 shows the results for a given lens obtained with Eastman Spectroscopic Plate Type V-G. The reduction ratio is 50 to 1, so that the frequency range of the chart is from 10 to 100 lines/mm. Value of the contrast in the image obtained from microdensitometer recording at three different heights across the negative image are shown as a function of the line frequency. The contrast in the target at a given height is approximately constant so that the decrease in contrast in the image results from action by the lens-emulsion combination. Curve 1 shows the results for a moderately high contrast of 1.3 in the target; curve 2 is for a medium contrast of 0.5; and curve 3 is for a low contrast of 0.2. The results appear to be in agreement with what one might anticipate on the basis of the literature dealing with the subject of target contrast and resolving power. However, the chief point to emphasize here is that the data for drawing these curves were obtained from a single negative, with consequent marked reduction in the number of factors that might conceivably make difficult the correlation of resolving power with contrast in the target.

It is clear that it would be difficult to determine the limiting value of resolving power of the lens for a given target contrast from curves of the nature shown in Fig. 9. The resolving power reaches its maximum when the contrast in the image reaches zero. However, because of the slow rate of change of contrast with frequency as one nears the limit of resolving power and because of local irregularities in the image it is difficult to determine with certainty at just what value of resolving power the contrast in the image reaches zero. As a practical expedient it may be preferable in reporting the performance of a lens under given conditions to state the value of the resolving power for a selected value of contrast in the image for a specified value of contrast in the target. For example we may wish to report the values of the resolving power for an image contrast of 0.1 for several values of target contrast. This information is readily obtainable from the curves and is illustrated in Table 1 for three emulsions.

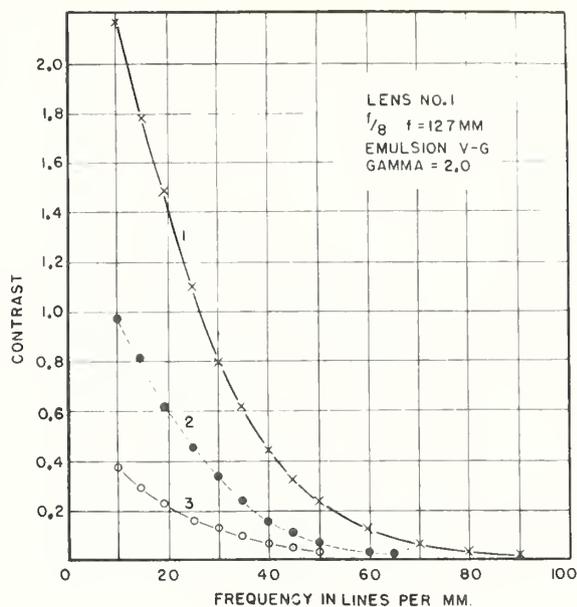


Fig. 9—The variation in contrast for increasing line frequency in the image for Type V-G, Spectroscopic emulsion. Target contrast is 1.3 for curve 1; 0.5 for curve 2; and 0.2 for curve 3. (The average density of the negative is 0.45).

TABLE I. Resolving power at image contrast of 0.1 for three emulsions at three target contrasts.
 Resolving power in lines/mm

Target contrast	V-G	Pan process	Pan-atomic X
1.3	64	36	30
0.6	46	25	20
0.2	33	13	13

High Contrast Multi-Line Chart

It is worthy of mention that the master high contrast target used in the preparation of the variable contrast target is itself a very useful test chart. This high contrast chart, shown in Fig. 7, with continuously varying frequency has been found to be well suited to the study of some phases of spurious resolution.¹¹ When a typical out-of-focus image of the chart is examined it is readily seen that the contrast between adjacent lines and spaces quickly falls to approximately zero as one proceeds toward the finer patterns.

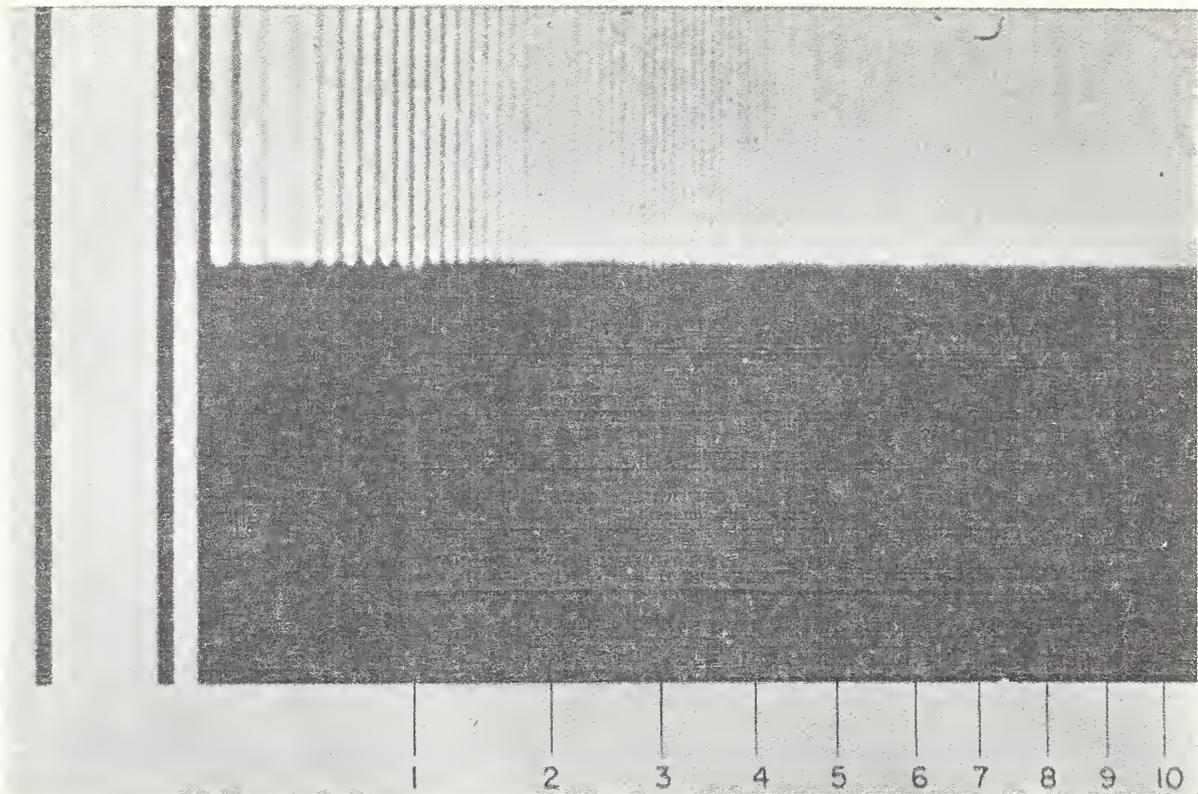


Fig. 10—Typical out-of-focus exposure showing spurious resolution.

However, instead of remaining zero, the contrast rises and falls as one continues in the direction of the finer patterns. This effect is shown in Fig. 10. The lower half of the chart used in making this photograph was opaqued with the exception of every tenth space which is left as a reference space. In the original negative, several of the reference spaces are easily visible. Most of these spaces have been lost in reproduction and so lines 1, 2, 3, etc., have been added to indicate their positions. The apparent resolution after the first minimum of contrast is spurious or false resolution. It may be noted that the final reference space in the lower half of the target image does not join a space in the upper half, but seems now to join a line indicating that something analogous to a phase shift of contrast has taken place. The microdensitometric trace of this waxing and waning effect is illustrated in Fig. 11. Here, true resolution is present, only in the left portion of the figure for the lower set of curves, the additional maxima do not represent true resolution. This phenomenon can be ex-

plained fairly simply on a geometric basis.

NBS Resolution Test Chart of 1952

Because of the widespread interest in the subject of resolving power and contrast it was decided in the early part of 1952 to revise circular C428 in order to incorporate some of the very appreciable gains in knowledge that had taken place since 1940. This has been done and a new publication entitled "Method of Determining the Resolving Power of Photographic Lenses" was issued in May 1953 as NBS Circular No. 533.¹² The most striking change is the inclusion of a somewhat different design of test chart, shown in Fig. 12. This test chart differs from the former standard chart in that the ratio of change in size between patterns has been reduced from $\sqrt{2}$ to $^4\sqrt{2}$ in order to achieve greater precision in the rating of lenses. Another innovation is the inclusion of low contrast charts, shown in Fig. 13, in addition to the high contrast charts.

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The contrast between lines and spaces is approximately 1.4 for the high and 0.2 for the low contrast chart. The range of the charts under standard conditions of a 25X reduction is from 12 to 80 lines/mm. However, because of the symmetrical nature of the pattern its range can be extended by appropriate copying and compounding of prints as shown in Fig. 14 to achieve any range desired. For the composite shown in the figure, the range is from 3.0 to 80 lines/mm.

The use of long lines is somewhat of a departure from the usual practice but it serves to extend the usefulness of the chart in that microdensitometric analysis of the test images can be readily performed. Figure 15 shows microdensitometric traces made by scanning the negative images of the high- and low-contrast charts. The light area at the bottom of the figure shows the area of the chart actually scanned. The variation of contrast in the image is shown in Fig. 16, where curve 1 shows the results for the high contrast chart and curve 2 shows

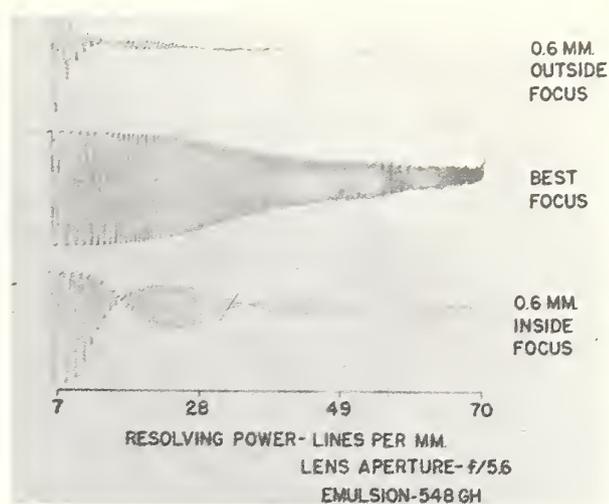


Fig. 11—Microdensitometer traces for lens of $f/5.6$; 548GH emulsion.

the results for the low contrast chart. It is evident that higher values of resolution are obtained with the high contrast chart.

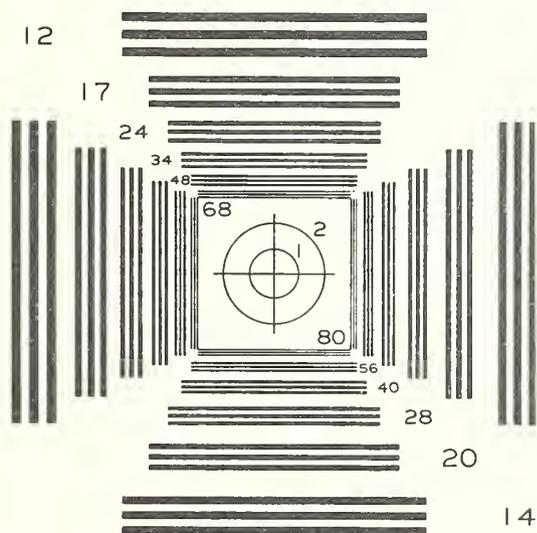


Fig. 12—Left—High-contrast resolution test chart. The ratio of the line spacings in adjacent groups is equal to $\sqrt{2}$; however, the ratio of the line spacings of corresponding opposite patterns is $^4\sqrt{2}$. When this chart is photographed at the standard distance of $26 f$, the values of resolving power that can be measured range from 12 to 80 lines/mm in a geometric series proceeding by $^4\sqrt{2}$.

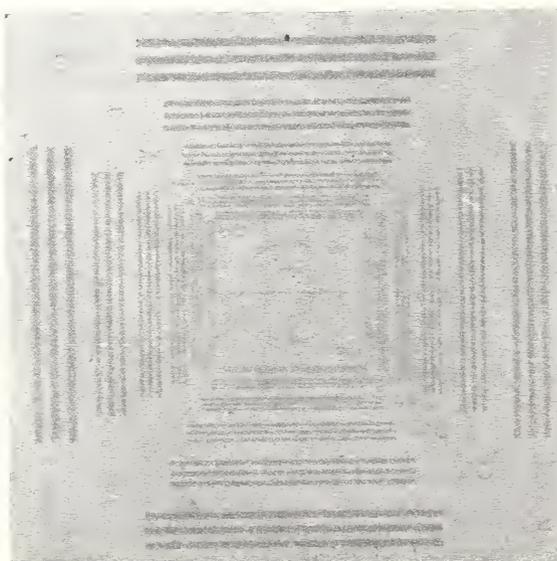


Fig. 13—Right—Low-contrast resolution test chart. The metrical characteristics of this chart are identical with those of the high contrast chart shown in Figure 12. The contrast (ratio of reflection densities) between the gray ink of the lines and the gray background is approximately 0.2.

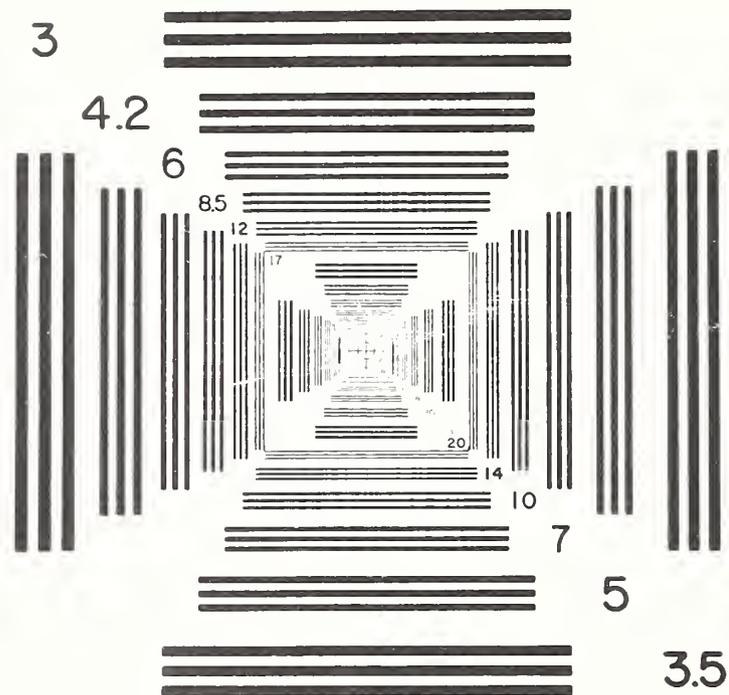


Fig. 14—NBS resolution test chart with range extended by photographic process. This chart is a composite formed by superimposing the high-contrast chart upon an enlarged copy of the master chart. The ratio of magnification is 4. The range of the composite chart is from 3 to 80 lines/mm as compared to the original range of 12 to 80 lines/mm.

Electronic Optical Bench

In addition to visual and microdensitometric analysis of images on negatives, there have recently been developed objective methods which depend upon analysis of the actual image formed by the lens. The method developed by Schade¹³ is of especial interest. Figure 17 shows the electronic moving-target optical bench constructed by Schade in connection with a research project sponsored by the Special Devices Center of the Office of Naval Research. This optical bench is now at the National Bureau of Standards. The target pattern is printed on the sound track portion of a loop of 35mm film which is moved past a rectangular aperture. The pattern consists of a set of sections having sinusoidally varying transmittances, each section containing a different spatial frequency. This pattern as seen through the target aperture is imaged by

the lens under test. A microscope is used to project the image onto the plane of a slit behind which is a multiplier phototube connected to its associated amplifier, meters, and recorder.

The target pattern produces a spatial variation in the target flux distribution, this variation consisting of a sinusoidal component superimposed on a constant component. The ratio of the amplitude of the sinusoidal component to the magnitude of the constant component is the target modulation, which is measured on the apparatus by projecting the target pattern directly onto the plane of the receiver slit with a good low-power microscope objective.

When the target is imaged by a lens, the modulation is reduced, the degree of reduction depending on the spatial frequency of the pattern and the quality of the lens. The ratio of the modulation in the image to that in the object is called

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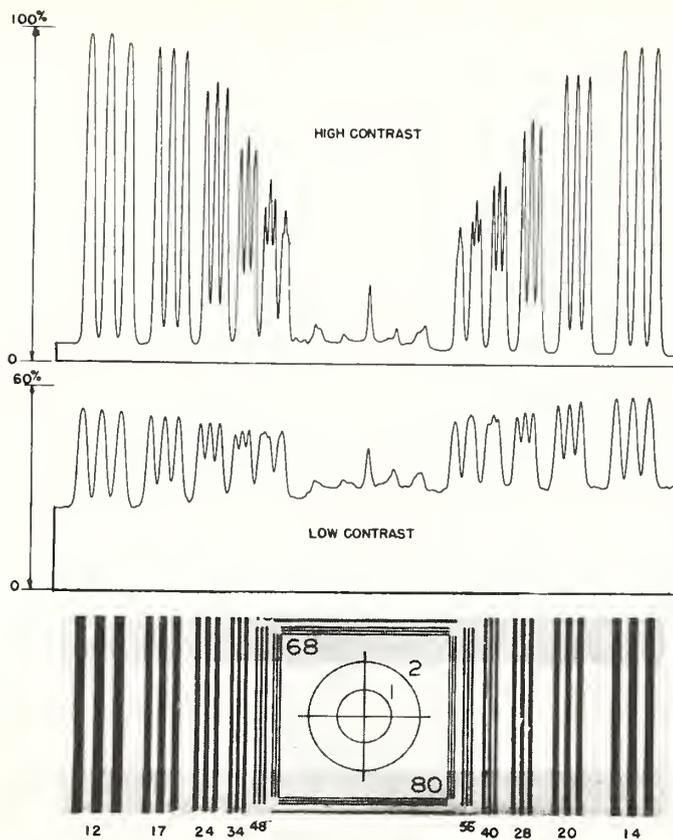


Fig. 15—Comparative results on axis for a lens tested with high- and low-contrast charts. These curves are microdensitomer traces made by scanning the negative images of the high- and low-contrast charts. The light area of the reproduction of the chart at the bottom of the figure shows the portion of the image actually scanned.

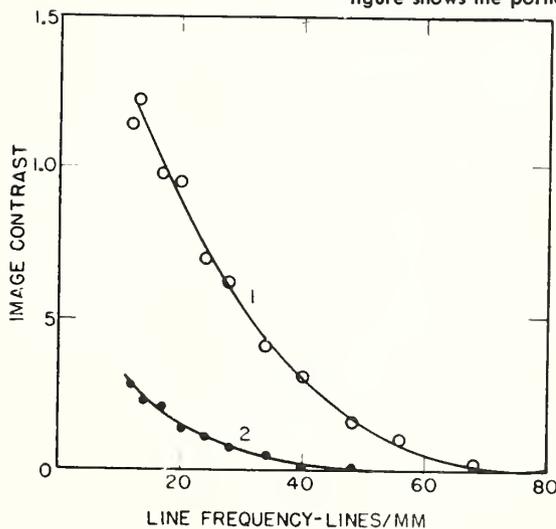


Fig. 16—Variation of contrast in the image as a function of line frequency. These curves show the measured values of image contrast taken from the curves shown in Figure 15. Curve 1 shows the results for the high-contrast target and curve 2 shows the results for the low-contrast target.

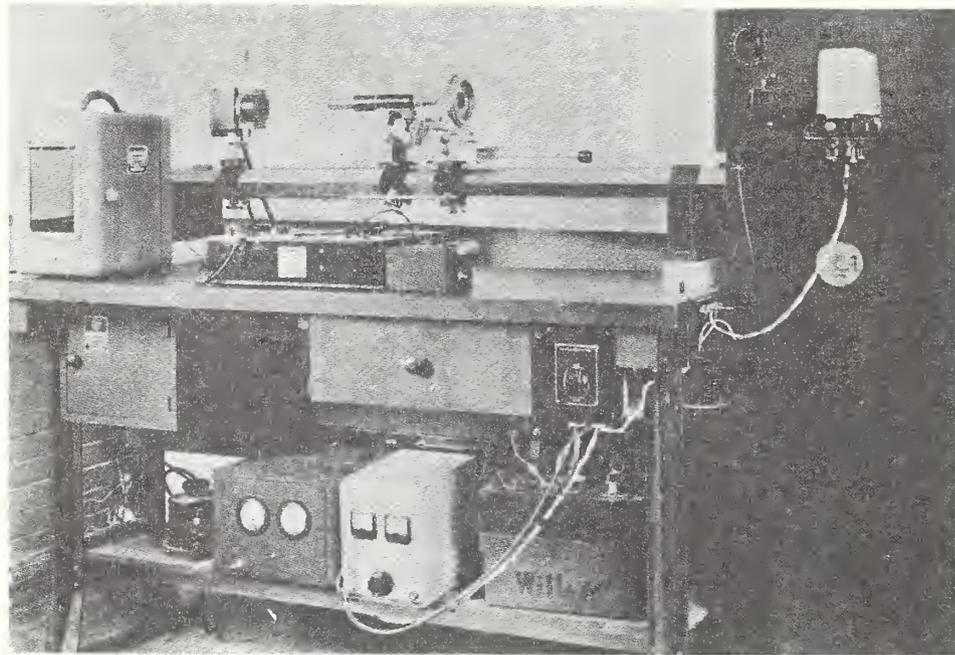


Fig. 17—The electronic lens bench. The test film is located in the housing at the right of the photograph. The image formed by the lens under test (center) is projected by the microscope into the phomultiplier tube at the left. Results are registered on the recorder.

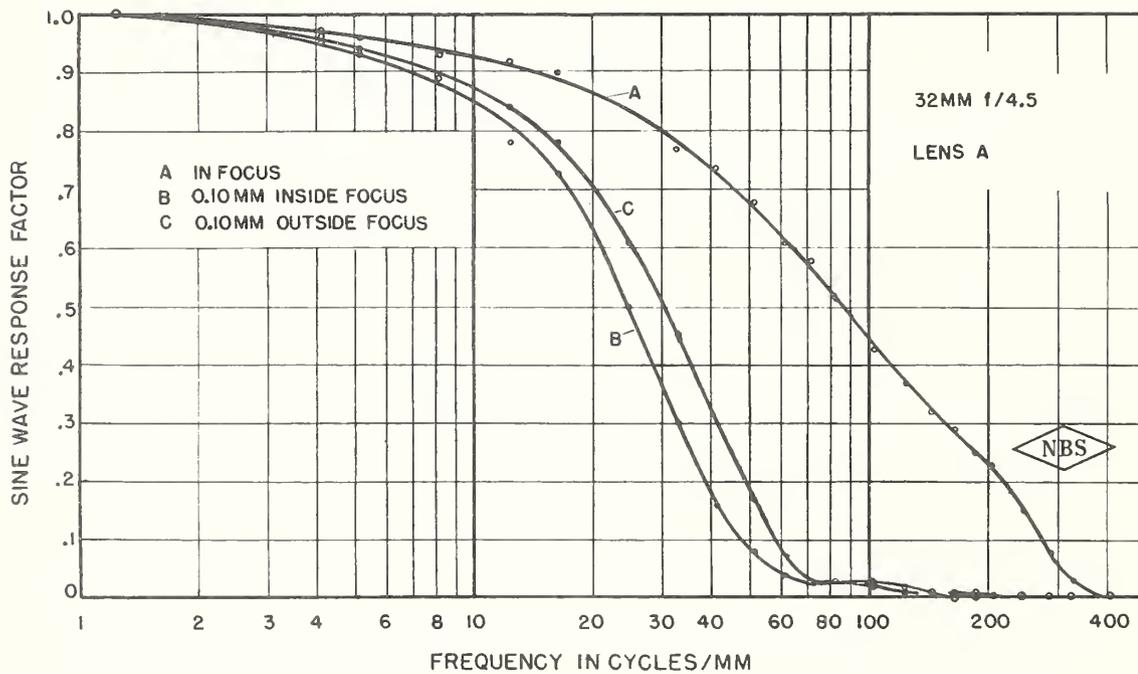


Fig. 18—Sine wave response for lens A.

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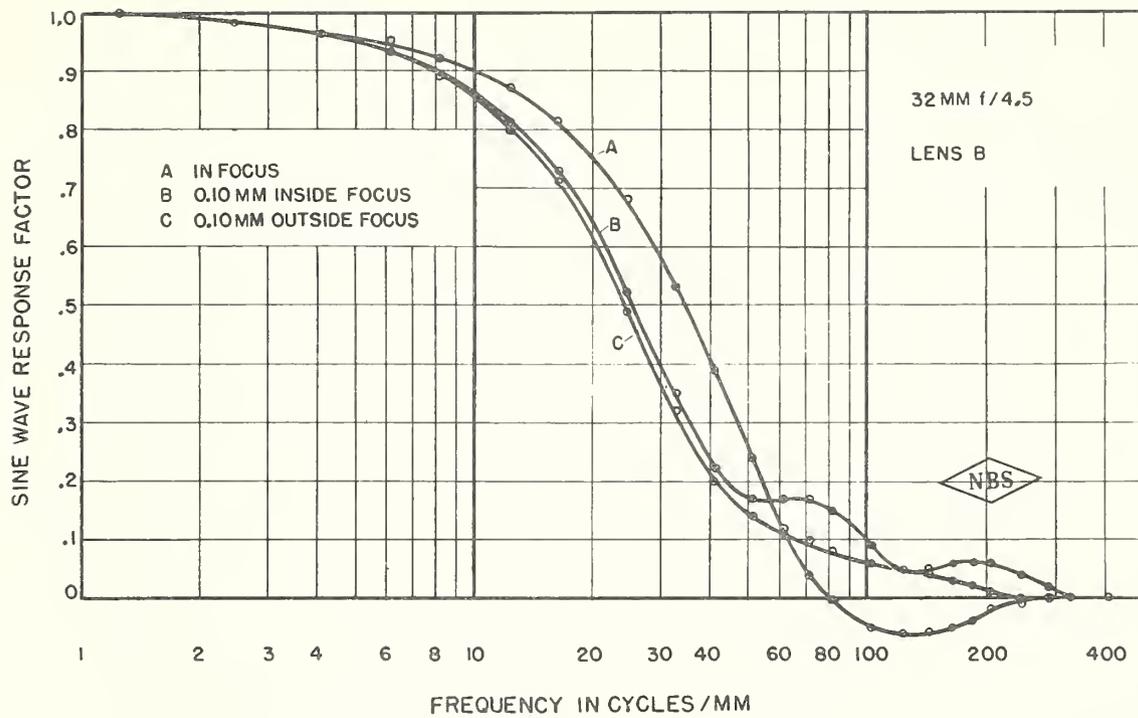


Fig. 19—Sine wave response for lens B.

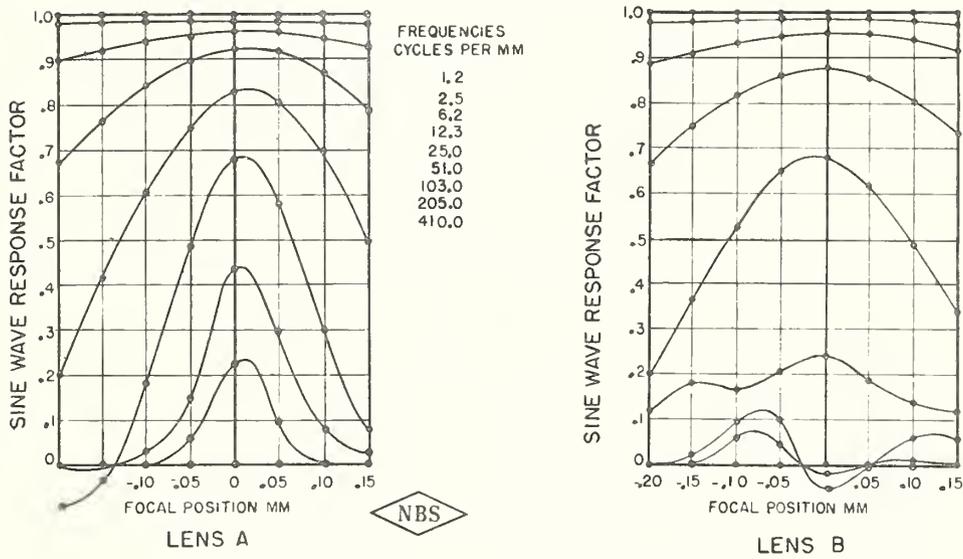


Fig. 20—Change in response through focus at constant line frequencies for lenses A and B.

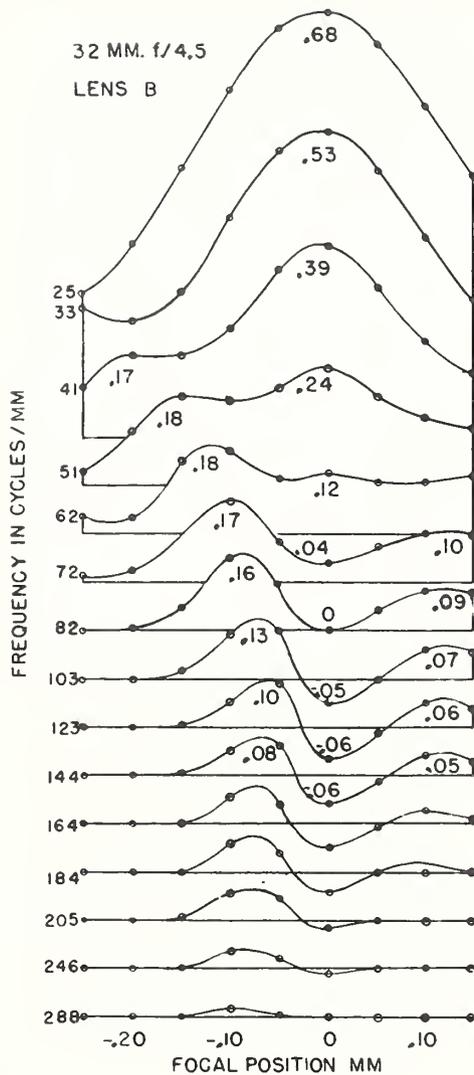


Fig. 21—Change in response through focus at constant line frequencies for lens B in more detail at higher frequencies.

the sine-wave response factor. This factor generally diminishes as the spatial frequency of the pattern increases, the lens acting as a low-pass filter on spatial frequencies. Figure 18 shows the sine-wave response curve for lens A test on axis for several conditions of focus. For this lens, the response reaches zero at a frequency of 400 lines/mm. The lower responses at 0.1mm away from the best axial focus are clearly distinguishable. Figure 19 shows similar curves for lens B. Although

lens B is nominally identical to lens A, there are pronounced differences in the responses. Lens B shows a region of false resolving power for the frequency band ranging from 80 to 200 lines/mm, that is, the dark parts of the target are rendered as light parts of the image. These results clearly show that this method can be readily used in rating lens performance.

It is interesting to study the change in response for several constant line frequencies as the receiver passes through focus. Figure 20 shows a comparison of the responses for lens A and lens B as the receiver passes through focus. For the lower frequencies, shown at the top, there is little difference between the two, but for the higher frequencies, A shows a relatively symmetrical pattern about the position of best focus, while B appears to exhibit a change in focus with frequency. This is shown in greater detail in Fig. 21. To avoid confusion, the response at each frequency is plotted at a different level, and the value of peak response is given for each frequency next to the proper curve.

The advantage of this type of analysis is that the results are not complicated by the characteristics of a photographic emulsion. It is probable that it will eventually be possible to evaluate the image forming properties of a lens and an emulsion separately, and to predict the performance of a given lens-emulsion combination.

Most of our work of this nature to date has been done on axis. However, work in the extra-axial regions now in process is yielding good results.

The data, used in making the curves, shown in Figs. 18 through 21, was kindly supplied by Mr. R. V. Shack and Mr. J. J. Ball of this laboratory.

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Selected Papers on

Radiation

(Contents on page VI)

Infrared High-Resolution Grating Spectrometer

Earle K. Plyler and Lamdin R. Blaine

An infrared grating spectrometer which can be used as a single- or double-pass instrument has been built and set in operation. The collimating mirror has a focal length of 235 centimeters and the instrument has the highest resolution from 1,600 to 3,500 cm^{-1} . In order to have flexibility in scanning the spectra at different speeds, a special drive mechanism has been built which is made of spur gears and worm gears. The speeds range from 2.5 to 200 minutes of time per degree of rotation. Examples of the spectra resolved by the instrument are given. Lines separated by 0.05 cm^{-1} are completely resolved and two lines separated by 0.025 cm^{-1} are partially resolved.

1. Introduction

In recent years, cooled lead sulfide detectors have been made of high signal to noise ratio. By the use of these cooled PbS cells, much smaller slits can be used on the spectrometer and the diffracting grating may be the limiting factor in the resolution of the instrument. By double passing the radiation on a high-grade grating, a resolution approaching the theoretical resolving power has been obtained by Rank and his coworkers¹ in the region of 5,000 cm^{-1} . The instrument that is described in the present work is best suited for measurements in the region from 1,600 to 3,500 cm^{-1} , and partial resolution of 0.025 cm^{-1} has been obtained in this region.

2. Description of Instrument

A spectrometer has been designed which can be used as a single- or double-pass instrument. Two optical systems have been used for double passing the grating. One arrangement of the optical components is shown in figure 1. After the radiation has passed through the entrance slit it falls on an on-axis paraboloidal mirror (focal length 235 cm) and

is rendered parallel. The mirror has a diameter of 16 in., and by using 7 in. on one side of the center it is effectively equal to a 3° off-axis system. From the grating, the parallel beam is intercepted by the optical flat and is reflected back to the grating where the beam almost retraces the incident path and falls on the second slit. The single-pass diffraction beam also falls on the second slit but its wavelength is slightly different from that of double-pass radiation. For the optical arrangement of figure 1, the dispersion is almost equal in the single- and double-pass spectra and is different only due to the inequality of the angle of the incident and reflected rays; and the wavelength regions are so close together that they cannot be separated by the transmission filters. In order to separate the two spectra, the optical flat is tilted a few minutes of arc from the vertical so that the two spectra are focused on the second slit with one above the other. The exit slit is then masked so that only the double-pass spectrum reaches the detector. When the instrument is used single pass, the optical flat is screened and the mask on the second slit is removed. The receiving surface of the lead sulfide cell is 2.5 by 0.2 mm and the radiation on 18 mm of the height of the slit is detected by the use of an ellipsoidal mirror giving a 7 to 1 reduction of image size.

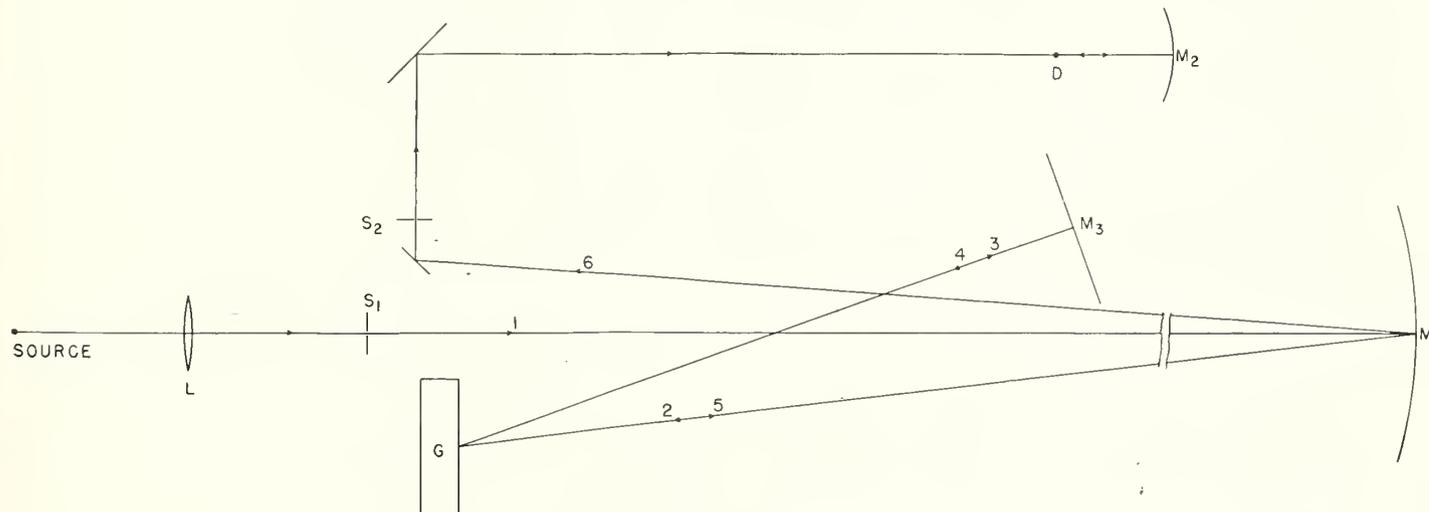


FIGURE 1. The optical arrangement for the double-pass spectrometer using an optical flat M_3 .

M_1 is a paraboloidal mirror of 235 cm^{-1} , and M_2 is an ellipsoidal mirror with foci of 15 and 105 cm^{-1} .

The second optical system for double passing the radiation is shown in figure 2. The essential part of this system is the two small plane mirrors behind the entrance slit which send the energy back to the grating the second time. In order to remove the single-pass radiation, the beam is chopped in front of one of the small 45° mirrors. In this system the dispersion is, for all practical purposes, identical to its single-pass dispersion as the angle of the incident and reflected radiation are practically equal. The image on the second slit for single pass is curved. The two mirrors reverse the image and its curvature so that the double-pass image on the second slit is approximately straight.

In addition to the optical parts of the spectrometer, a number of auxiliary components are used. These consist of sources, filters, absorption cells, Fabry-Perot interferometers, PbS and PbTe photo-detecting cells, amplifiers, and recorder. From 1 to 3.6 μ , a 300-w Sylvania enclosed zirconium arc is used for the source, and from 3.6 to 6 μ , a Nernst glower is used. In order to remove the higher order spectrum, transmission filters are set into the path of the radiant energy in front of the entrance slit of the spectrometer. A silicon filter is used from 1.2 to 2 μ , a germanium filter from 2 to 3.5 μ , and transmission filters made by Baird-Atomic from 3.5 to 6 μ . The filters transmit about 90 percent of the energy and are used instead of a foreprism arrangement. The absorption cell is 1 m in length and with the use of mirrors a 6-m path is obtained. Fluorite windows are used and the transmission of the cell for the 6-m path length is about 75 percent.

The spectrum is recorded on a two-pen recorder. One pen is used for the interferometer fringe system and the other pen records the absorption spectrum. The method of measuring the spectra from the charts has been described previously.² A disk

chopper is used to modulate the radiation at 90 cps. The radiation falling on the cooled PbS cell is amplified by a 90-cps Wilson narrow-band-pass type amplifier³ of low noise level. The spectrum is scanned by rotating the grating and for the highest resolution slow speeds are required. For example, in the region of 3,020 cm^{-1} , about 30 min of time is required to scan one wavenumber for the best formation of the lines. In order to adequately survey different regions of the spectrum under different levels of resolution a versatile drive system is desirable. A large number of speeds are possible by use of a gear box between the motor and the worm gear attached to the grating mount. A synchronous motor of 900, 1,800, and 3,600 rpm of about 1/30 hp is attached to a 10-to-1 reduction gear, which drives a four-speed gear box with reductions of 1 to 4, 1 to 1, 3 to 1, and 5 to 1. The gear box is reduced by a 60-to-1 precision worm, which then drives a pair of precision worms on the grating turn table which have reductions of 120 to 1, and 180 to 1. This arrangement of gears makes it possible to rotate the grating mount at 12 different speeds, the fastest speed being 2.5 min of time per degree of rotation and the slowest being 200 min/deg. Additional speeds which are faster and slower than those described above, can be obtained by changing the gears in the first reducer.

The uniformity of rotation of the system has been checked by the regularity of interference fringes of about 0.3- cm^{-1} spacing. The variation of the distance between successive fringes is less than 4 percent, which indicates that the irregularities of the drive do not introduce errors in measurement greater than 0.006 cm^{-1} . For rapid scanning or for moving from one spectral region to another, an auxiliary motor of 1,800 rpm can be automatically coupled directly to the output of the transmission gear box, which provides a rotational speed of 1° per half minute of time.

Earle K. Plyler, L. R. Blaine, and E. D. Tidwell, *J. Research NBS* 55, 279 (1955) RP2630.

³ R. C. Nelson and W. R. Wilson, *Proc. Nat. Elec. Conf.* 3 (1947).

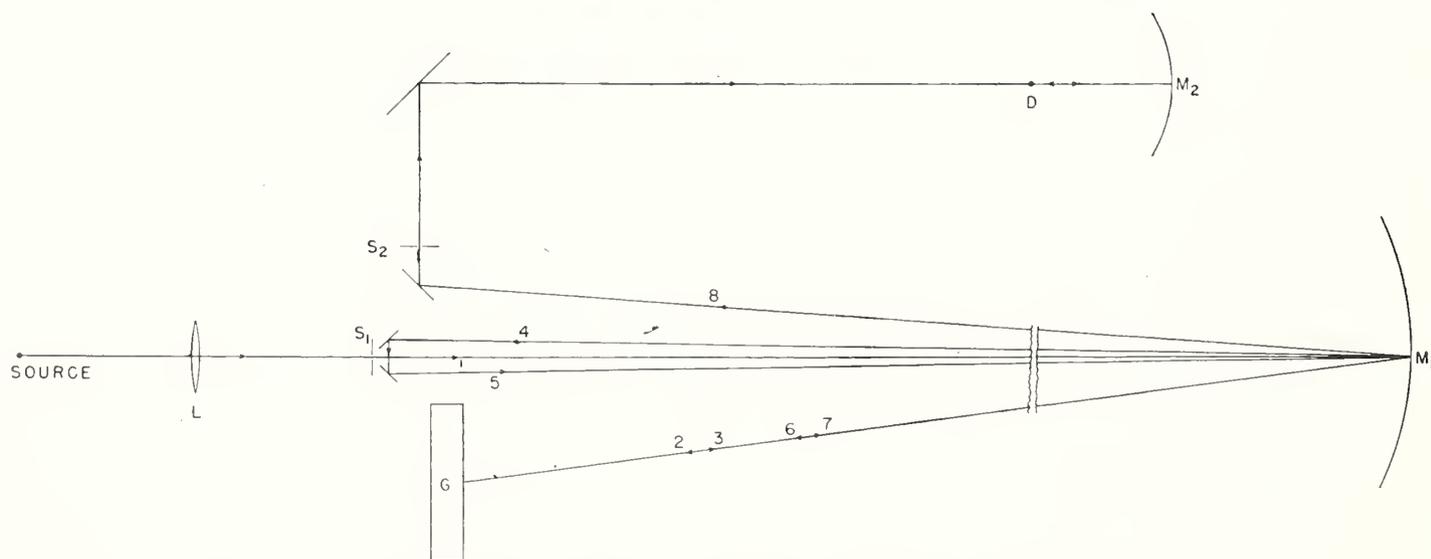


FIGURE 2. The optical system for double pass using two small mirrors behind the entrance slit.

The other mirrors are the same as those represented in figure 1.

3. Examples of Spectra Measured

The spectrometer has been in operation for about a year and some bands of several molecules such as methane, ethylene, ethane, ammonia, and cyclopropane have been measured. The classification of these spectra and the calculation of the molecular constants are now being made and the results will be given in other publications.

Some examples of the high resolution obtained by the instrument are given of parts of bands observed with slow scanning speeds. Figure 3 shows the P13 lines of methane as observed with a 10,000 lines/in. Bausch and Lomb grating using the optical system represented in figure 1. The pressure used was 0.1

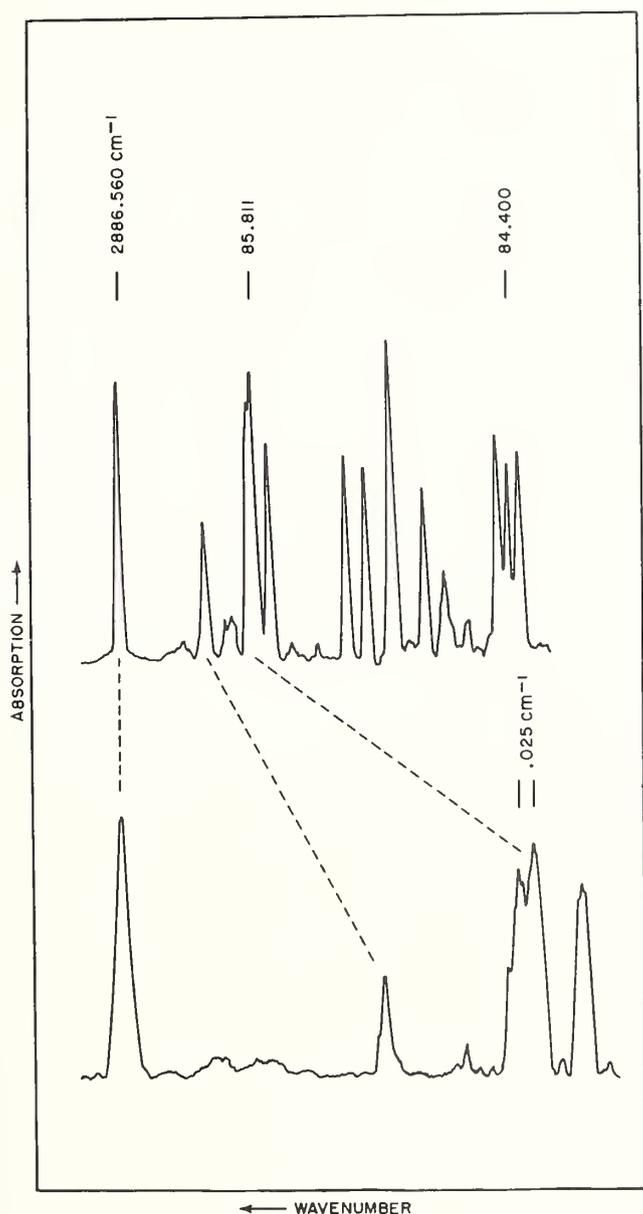


FIGURE 3. The fine structure of the P13 line of the ν_3 band of methane.

The lower curve is a part of the P13 line of methane scanned at the slowest rate of the drive. The two partially resolved lines are separated by 0.025 cm^{-1} . The pressure was 0.1 mm (Hg) and the path length 6 m. A grating with 10,000 lines/in. was used.

mm (Hg) and the path length was 6 m. As the resolution of the instrument is increased, lower pressures of the gas can be used. When the P-13 transition of methane was measured on a single-pass spectrometer the last three lines were not so well separated and for the same maxima of absorption about 1 mm (Hg) pressure was necessary. The last two lines are separated by 0.055 cm^{-1} and the line widths at half intensity are about 0.023 cm^{-1} .

The lower part of the figure shows a slower scan (one-third speed) of several lines of the upper spectrum. The separation of the two partially resolved lines is 0.025 cm^{-1} . It is believed that the optical system is capable of still higher resolution, but until the noise of the electronics and the detector is reduced, the limit of resolution of the gratings and the optics cannot be determined.

An example of pressure broadening is shown in figure 4. Part of the ν_3 band of ammonia at $3,500 \text{ cm}^{-1}$ overlaps the atmospheric absorption lines of water vapor. The width of the water vapor lines is about 0.15 cm^{-1} and the observed width of the ammonia lines at 2-mm pressure (Hg) is 0.03 cm^{-1} . On account of the large difference in line widths of the two substances it is easy to identify the two spectra. The spectra shown in figure 4 were observed with a Babcock 15,000 lines/in. grating which had a total of 120,000 lines.

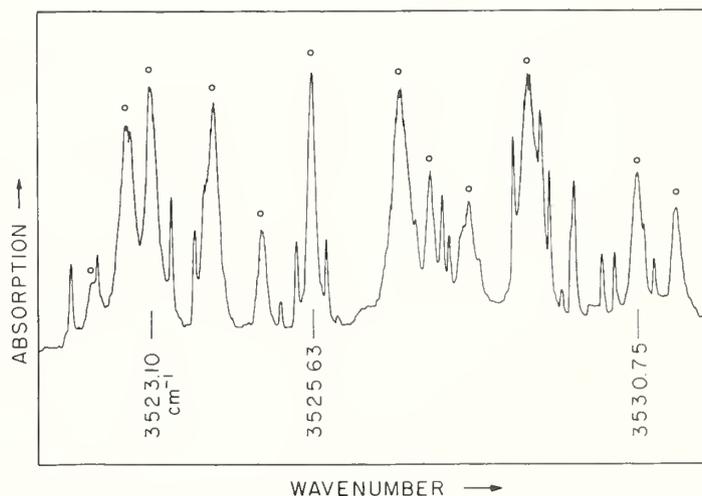


FIGURE 4. The absorption spectrum of ammonia 2 mm (Hg) pressure, in the region of $3,500 \text{ cm}^{-1}$.

The broad lines, marked with a circle over them, are produced by the atmospheric absorption of water vapor.

The actual limit of resolution of the instrument at the present time under the best conditions appears to be about 0.02 cm^{-1} . Larger gratings or more sensitive detectors, or both, will be required to make a further improvement in resolution.

The authors are indebted to Horace Babcock for making available to them one of his 15,000 lines/in. gratings.

WASHINGTON, September 29, 1958.

Infrared Measurements With a Small Grating From 100 to 300 Microns¹

Earle K. Plyler and L. R. Blaine

Good resolution has been obtained with a small grating in the region from 100 to 300 microns. A high-pressure mercury lamp, having a quartz inner envelope, was used as a source and a Golay cell as a detector. The radiation was reflected from three filtering plates before passing through the entrance slit. This filtering served to reduce the stray radiation to about 5 percent.

1. Introduction

In the past several years small gratings have been used in spectrometers for the near-infrared region. It has also been shown that a small prism spectrometer could be converted, with only minor changes, into a far-infrared instrument.² Good resolution has been obtained in the region from 25 to 125 μ using a thermocouple as the detector. Further work has been done on the instrument, which increased the resolution and extended the wavelength range.

2. Experimental Procedure

In order to extend the measurements beyond 125 μ three changes were made in the instrument. A Golay cell with a crystal quartz window replaced the thermocouple, a high-pressure mercury lamp was substituted for the globar source, and a different arrangement of the filters replaced the previous three-filter arrangement described in footnote 2.

The Golay cell was found to have a greater sensitivity in the detection of the long-wavelength radiation than the thermocouple, making it possible to use narrower slits and to obtain better resolution.

High-pressure mercury lamps have much greater intensity in the far-infrared region than the globar source. It was found that an AH4 mercury lamp was adequate for the measurements made in the region from 100 to 300 μ . A section 1 by $\frac{1}{2}$ in. was removed from the outer glass envelope which enclosed the quartz lamp. A small glass tube was introduced through the opening and nitrogen was circulated about the electrodes, which greatly added to the life of the lamp.

Ultraviolet and visible radiation are intense from the lamp, and the observers' eyes should be shielded. At first the lamp was housed in a black box with an aperture that permitted the radiation to fall upon the slit of the spectrometer. Later the lamp was coated with a layer of magnesium oxide. The MgO film absorbed only a small fraction of the radiation at 100 μ , but it was effective in scattering the short-wavelength region. A crystal of magnesium oxide 1-mm thick absorbed about 80 percent of the radiation at 100 μ , but with thinner layers the energy loss with the coating became negligible.

As measurements are carried out to longer wavelengths, increased filtering is required for the removal of the stray radiation. A new arrangement of the optical system has been made so that three reflection filters are employed in front of the entrance slit of the spectrometer. Figure 1 represents the system that was used. The filters that were placed in positions A, B, and C were reststrahlen plates of CsBr and CsI, gratings, and roughened silver scatterer plates. In the measurements shown in figures 2 and 3, one filter of each type was used. For the region from 70 to 100 cm^{-1} the external filters as shown in figure 1 were a CsBr reststrahlen plate and two roughened silver plates, one of which was blackened. For the spectral region from 53 to 70 cm^{-1} a CsI reststrahlen plate was substituted for the CsBr plate. In some of the measurements a grating with 1,000 lines/in. was substituted for one of the silver plates.

Some spectra have been obtained in the region from 25 to 50 cm^{-1} by the use of small gratings with 60 lines/in. and 100 lines/in. In the measurements in this region it was necessary to use wider slits, and in the region of 400 μ the slitwidth was 4 mm. In order to obtain the wide slits, the regular slit jaws were removed and replaced by two metal jaws so that there was a separation of 2 mm when the slit drum read zero. This change enabled the observer to set the slits between 2 and 4.5 mm.

The strong water-vapor line at 26 cm^{-1} was observed, but it is estimated that the spectrum con-

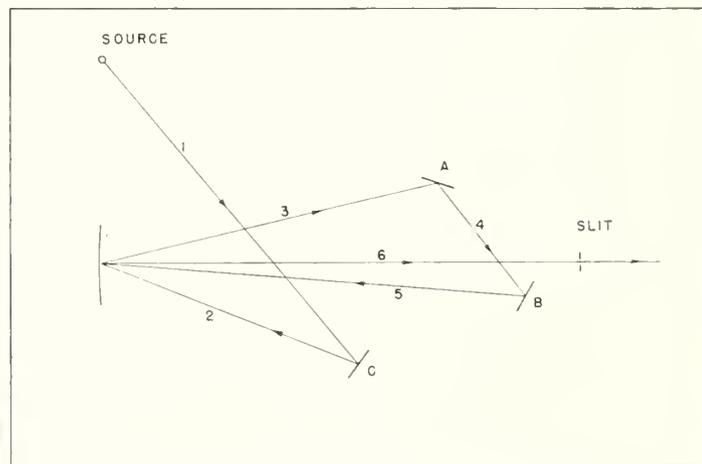


FIGURE 1. Optical arrangement for the filters.

The reflection filters are placed in positions A, B, and C.

¹ This work was supported by the U. S. Atomic Energy Commission.

² Earle K. Plyler and Nicolo Acquista, J. Research NBS 56, 149 (1956) RP2660.

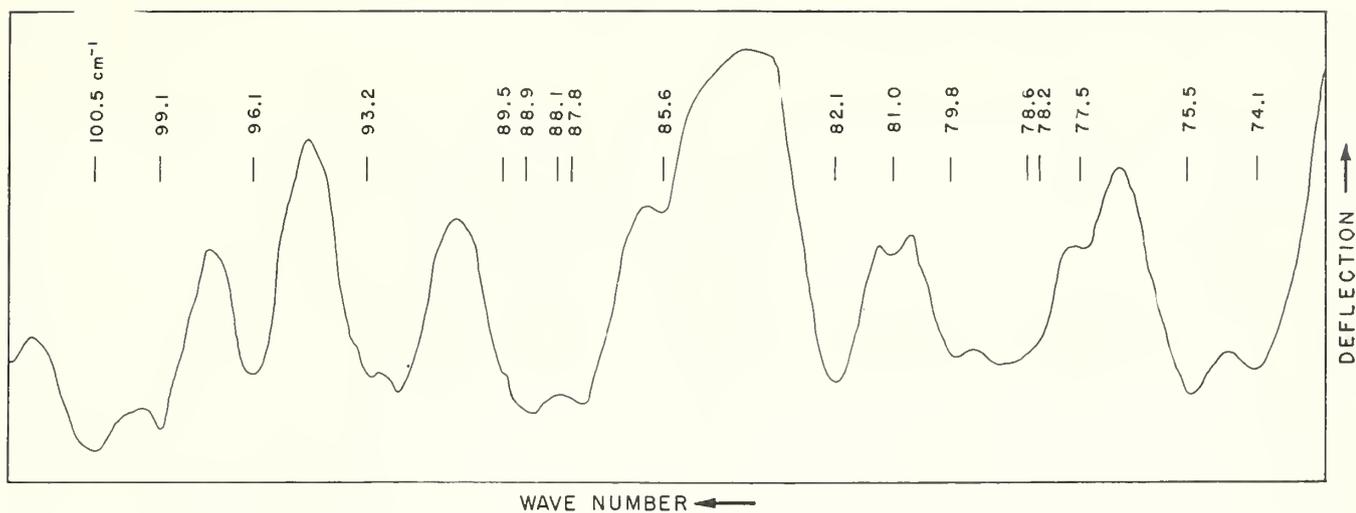


FIGURE 2. Absorption spectrum of water vapor from 75 to 100 cm^{-1} .

The slitwidths were 1.5 mm and a 180-lines/in. grating was used.

tained about 40 percent of stray radiation in this region. Before quantitative measurements can be made between 300 and 400 μ on the instrument a more efficient filtering system will be required.

3. Results

As the present investigation has been primarily concerned with the methods of measurements, only three examples of the results will be given. The absorption spectra of several molecules have been reported in other publications.³

Figure 2 represents the absorption spectrum of water from 73 to 100 cm^{-1} as measured with a 180-lines/in. grating. This region was included in the previous publication (see footnote 2), but now the stray radiation has been almost completely removed and the absorption lines of high intensity cause the deflection to approach the zero line. The wave numbers marked on the lines are the calculated values determined by Benedict.⁴

³ D. E. Mann, J. H. Meal, and Earle K. Plyler, *J. Chem. Phys.* **24**, 1018-1022 (1956).

The amount of water vapor in the path was sufficient to produce nearly 100-percent absorption for the lines of moderate and high intensity; thus some of the weak lines could be observed. The observed wave numbers of some of the lines of low intensity agree closely with the calculated values. The wavelengths were determined experimentally by the measurement of the angle of the grating with reference to the central image.

Figure 3 represents the region from 53 to 70 μ . The more intense lines of the spectra have been identified and the wave numbers are marked on the chart. The two lines near 60 cm^{-1} are slightly distorted by second-order lines. The linewidths are about 0.6 cm^{-1} , which is a measure of the resolution obtained. The spectrum of water vapor recorded in figures 2 and 13 is for a path length of 2 m and relative humidity of 40 percent. The slitwidths were 1.5 mm for the region from 75 to 100 cm^{-1} , and 2.5 mm from 53 to 72 cm^{-1} .

⁴ W. S. Benedict, private communication.

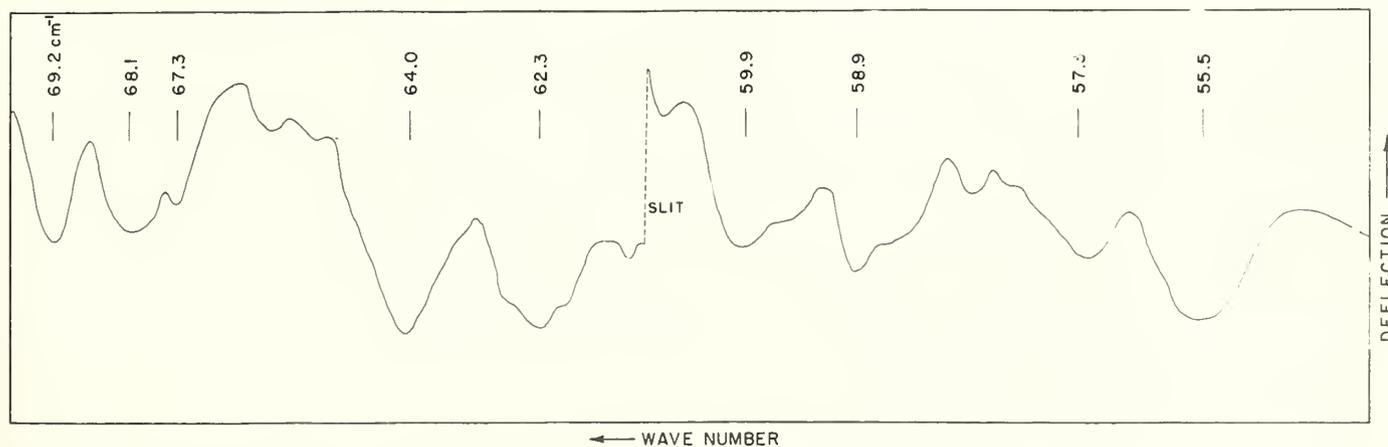


FIGURE 3. Absorption spectrum of water from 55 to 70 cm^{-1} as measured with a 180-lines/in. grating.

The slitwidths were 1.5 mm to 62 cm^{-1} and 2.5 mm from 55 to 62 cm^{-1} .

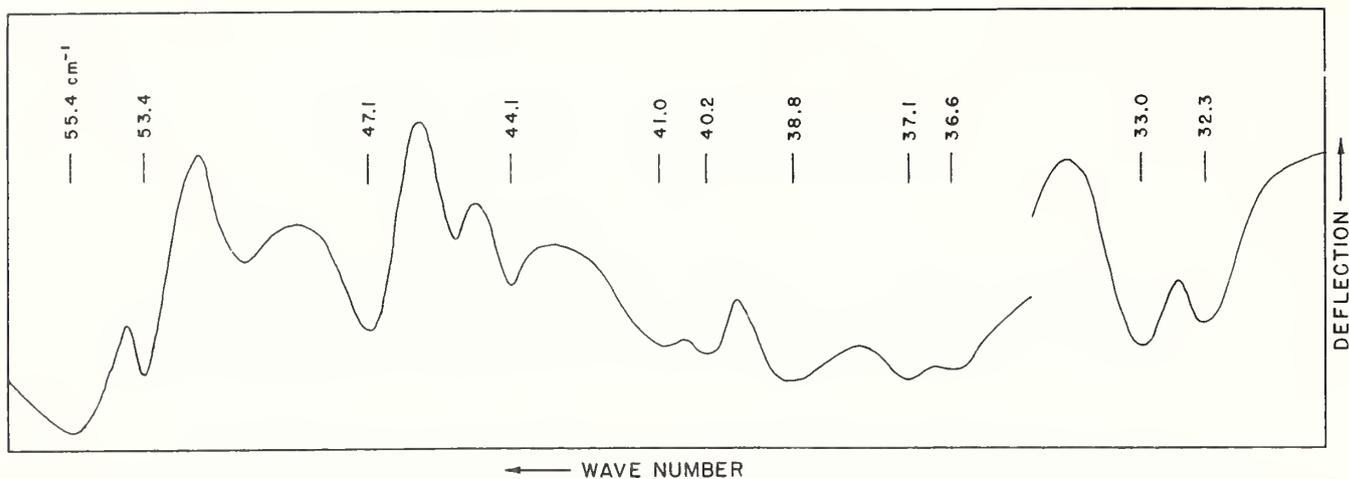


FIGURE 4. Absorption spectrum of water vapor from 36 to 55 cm^{-1} as measured with a 100-lines/in. grating and from 30 to 36 cm^{-1} with a 60-lines/in. grating.

The slitwidth was 4 mm for the entire region.

Figure 4 shows the absorption spectrum of water vapor from 30 to 55 cm^{-1} . The stray radiation was reduced to a small percentage by using a 0.2-mm window of LiF as a transmission filter. The slitwidths were 4 mm, and a 100-lines/in. grating was used from 36 to 55 cm^{-1} and a 60-lines/in. grating was used from 30 to 36 cm^{-1} . In observing the two lines at 32.3 and 33 cm^{-1} a window of KBr, 0.4-mm thick, was found effective in eliminating higher orders from the grating. The line at 47 cm^{-1} could

be observed through the KBr window, but the energy in the higher wavelength region (47 to 100 cm^{-1}) was almost zero.

In conclusion it can be stated that a small grating is adequate for measuring spectra from 25 to 300 μ , and with further improvements in the filter system the measurements can be extended to 400 μ .

WASHINGTON, July 18, 1957.

Vibration-Rotation Structure in Absorption Bands for the Calibration of Spectrometers From 2 to 16 Microns¹

Earle K. Plyler, Alfred Danti,² L. R. Blaine, and E. D. Tidwell

(September 23, 1959)

Suitable bands of common gases have been tabulated and remeasured wherever necessary from 2 to 16 microns to obtain an accuracy of about 0.03 cm^{-1} throughout the region and to provide good calibrating points at frequent intervals. Some 600 rotation-vibration lines are illustrated in 20 spectrograms and wavenumbers are listed in companion tables with considerable intercomparison with worthy data obtained in other laboratories. The absorption bands were remeasured or calibrated by using either a precisely graduated grating circle or standard atomic lines with the fringe system formed by a Fabry-Perot interferometer. Characteristic features of the individual bands are discussed briefly and references to other publications are given. The substances used for calibration include H_2O , CO_2 , CO , HCl , HBr , NH_3 , C_2H_2 , CH_4 , N_2O , and polystyrene film.

1. Introduction

In recent years, infrared spectroscopists have expressed concern regarding satisfactory methods and wavelengths for calibrating good prism and small grating spectrometers [1, 2, 3, 4].³ In the past, a number of publications [5, 6, 7, 8, 9, 10, 11] from several laboratories did tend to ease the immediate requirements; but with the influx of finer instruments the need for more thorough, suitable, and precise coverage has arisen. Professor Mizushima and fellow workers have clearly indicated in their detailed measurements [12] that the small grating instruments are certainly capable of producing rather precise data. The present work addressed itself to the need for bringing together and remeasuring suitable bands of common gases in order to certify absolute accuracy to within several hundredths of a cm^{-1} throughout the region and to provide calibration points at frequent intervals.

2. Utility of the Molecular Band Method

Although in several cases (extremely precise measurements on CO [13, 14, 15] and HCN [16]) the use of molecular bands for calibration purposes is probably as good as the use of atomic lines, this method should not take preference over the use of atomic lines when precise measurement is being considered. However, when measurements good to several hundredths of a cm^{-1} are sufficient or in cases where other limiting factors enter, viz, broad bands, then this method may

offer a number of advantages. In the case of prism instruments, the use of appropriate atomic lines for calibration is rather limited to the visible and near infrared regions and the need for single order coverage throughout is apparent. Present day commercial instruments are more easily calibrated in absorption than in emission and the molecular band method obviates the need for a full battery of atomic line equipment as well as personnel capable of using it. In addition, only available or easily obtainable substances have been used in this work so that laboratories should experience little difficulty in using this method. Most of the gases are available in typical chemistry laboratories and it will be mentioned that work in this laboratory has indicated that, in general, natural gas from commercial lines and acetylene drawn from the welder's tank are suitable for calibration purposes. Should impurity peaks arise in these bands, use can still be made of the lines which are clearly those tabulated and illustrated in the following sections.

Some 600 lines, which provide rather thorough coverage, are tabulated and illustrated in 20 spectrograms. For certain bands, the number can be appropriately increased by having reference to recommended publications given in the discussion section. Attempts were made to keep the number of different gases used at a minimum and they include: H_2O , CO_2 , CO , HCl , HBr , NH_3 , C_2H_2 , CH_4 , and N_2O . Also a polystyrene film has been included.

3. Instruments and Methods Used in Calibrating the Standard Lines

Two instruments in the Radiometry Section of the NBS were used in measuring the bands and in the 2- to 4- μ region most bands were measured on both instruments at different resolution and the results compared.

¹ This research was supported by the Air Force Cambridge Research Center and by a NASF grant to MIT.

² Guest worker from MIT.

³ Figures in brackets indicate the literature references at the end of this paper.

3.1. 5 to 16 Micron Region

a. Instrument

The previously described [17] grating spectrometer with an off-axis collimating mirror of 1-m focal length was used in conjunction with an extremely precise grating circle reported to be accurate to at least 3 sec of arc for any angle [18]. For the longer wave work (5 to 16 μ) the lead telluride detector was replaced with a thermocouple detector. When this instrument was used in the 2- to 4- μ region, the thermocouple detector was purposefully used so that resolution could be lowered and made comparable to that of small grating instruments. A KBr foreprism arrangement with fixed foreprism slits and a manually adjustable prism was used for order separation throughout this work. Under these stated conditions the instrument was capable of resolving lines separated by 0.3 cm^{-1} in the 7- to 16- μ region and up to about 1 cm^{-1} in the 2.5- to 6.5- μ region.

b. Method of Calibration

With a microscope attachment, angles were read off the inscribed circle, centered with respect to the spindle of the cone which rotates the grating, and fiducial marks recorded on the chart. During measuring runs, readings were taken every 5 min of arc and a slow drive speed (~ 23 min per deg) was used. It was found that proper positioning of the foreprism energy upon the entrance slit of the spectrometer proper was necessary in the sense that for extreme settings, where the maximum of energy being admitted to the spectrometer would come about 5 grating degrees away from the point of interest, the frequencies of measured lines would be in error by several hundredths of a cm^{-1} . By making frequent settings of the foreprism (about every 2 deg of grating angle) so that the energy would be maximized and hence placed symmetrically on the entrance slit, these effects could be ignored. Such considerations were, of course, of greater importance in regions where wide spectrometer slits were required or at long wavelengths where the dispersion of KBr is greatest. (This matter was considered in some detail, since it is felt that it is a frequently overlooked source of error occurring with the use of foreprisms.) In the course of any complete run (standards plus spectrum to be measured) no changes were made in instrumental conditions (viz, retardation resulting from changes in amplifier response settings may shift the line center) unless absolutely necessary.

At the start of the work, it was decided that the precisely measured CO fundamental lines (see ref. [13]) (table 4) be used as standards wherever possible. The procedure devised was that of using the simplified grating relation, $n\lambda = K \sin \theta$, where an "effective" grating constant, K_{eff} , incorporating spectrometer characteristics, could be calculated by measuring about 15 CO lines and the physical central image. This was done with the grating being turned first in one direction then in the reverse direction. If slightly different, the K 's obtained separately for each direction would be averaged. Once a good

value of K had been determined for a grating, the central image was always calculated from the 10 (or so) standard lines recorded either before or immediately following a measuring run. Using this procedure, the frequency of standard CO lines could always be recalculated from chart measurements to within about $\pm 0.01 \text{ cm}^{-1}$ of the accepted value. With good spectrometer and grating alignment, it was felt that CO standards could be used to calibrate NH_3 , CH_4 , and H_2O lines even though the grating angle for the standards differed appreciably from that of the lines in question. In calibrating the lines, both standards and runs to be measured would be recorded with the grating being turned in the same direction. As is often the case, it was found that slightly different values would be obtained for the two directions. Final values represented the average of an equal number of measurements in each direction. It is believed that the tabulated values are accurate to $\pm 0.02 \text{ cm}^{-1}$ in this region.

c. Chart Reduction

Wherever possible single lines were picked out for measuring, but in some instances certain complex lines were measured since they are particularly suited for direct prism observation. It is advisable to record spectra on ruled chart paper where the ruled lines are reproducible perpendicular to the chart edge. The determined center point of the line to be measured can then be easily projected to a suitable measuring base line on the chart by using a pair of dividers and the nearest ruled line. The center of the line should be determined with a pair of stiff dividers at about the 2/3 absorption point and not at the peak or apparent maximum of absorption. If the spectrum is particularly noisy the center should be determined at a number of places from the 2/3 to 3/4 point of the maximum of absorption and the average taken. Fractional distances between "pip" marks should be measured to within 0.005 in. using a steel rule and visual enlarger if necessary. For the majority of measuring runs, 5 min of grating are corresponded to 2 in. on the chart.

d. Instrument and Calibration

The instrument, which uses the double-pass system and is capable of resolving about 0.03 cm^{-1} has been described [19] and only a few details need be added. Standard atomic lines and the fringe system described in a previous paper (see ref. [17]) were used to calibrate the molecular bands. By scanning the spectrum slowly and averaging several runs, a set of relative values for the lines of a band can be determined to a few thousandths of a cm^{-1} and the absolute to $\pm 0.01 \text{ cm}^{-1}$. With scanning at a medium rate, absolute values are probably in error by $\pm 0.03 \text{ cm}^{-1}$. The accuracy of measurement of this instrument is increased by the lines being very narrow (0.03 cm^{-1}). Measurements made under lower resolution (about 1 cm^{-1}) with the other instrument in this region agreed with these to about $\pm 0.05 \text{ cm}^{-1}$. This indicates that low resolution

spectrometers can measure with an error of about 1/10 of the resolution.

e. Refractive Index

In this laboratory measurements on both instruments are carried out with the grating in air so that reported frequencies were corrected to vacuum by using refractive index tables, and temperature and pressure corrections given by Penndorf [20].

4. Comments on Measuring Technique and Use of the Standard Lines

For the present-day molecular spectroscopist who wishes to carry out precise measurements, useful points on measurement technique, whether on standard or unknown lines, have already been mentioned in previous sections on method of calibration, instrument performance, chart reduction and also in reference [17]. Concerning a spectrum to be measured, in cases where the instrument is being used at or near its resolution limit (ripple spectrum) or in the case of complex lines or shoulders, then the best possible "center" point must be picked and this may necessarily have to be near the absorption peak since lines may not be well enough developed to permit measurement at the 2/3 to 3/4 absorption point.

It is advisable to record both spectra and standards for each direction of grating turn if the driving mechanism is equally accurate in both directions. Provisions or modifications should be made on commercial or laboratory instruments so that the grating can be used in both directions with near equal accuracy and the results averaged. Undoubtedly, in most laboratories the standards will be used in conjunction with reproducible pip marks. Data will be extracted from simple pip or drum number versus frequency (cm^{-1}) relations by fitting a straight line to two end points and an extended deviation curve plotted. In view of extremely precise data for certain bands (CO, HCN) the utility of using these standard lines in higher orders for the same grating angle should not be overlooked. In terms of precision measurement, it would appear that the *R* branch (1,000 to 1,200 cm^{-1}) of the $\mu/2$ NH_3 band might also be classified in this category. This higher order procedure, of course, requires refractive index considerations for conversion to vacuum and each spectral region must be handled independently.

In making use of the standard lines under lower or higher resolution, workers should exercise necessary care. For example, in the CO bands where (apart from some isotopic overlap) the lines are single and suitably spaced throughout, even ripple peaks observed with a prism instrument would serve as satisfactory calibration points. Whereas, in many other bands proper usage under lower resolution may require judicious use of sufficiently low pressures and slow scanning speeds to assure the proper development of lines and eliminate "pulling" by strong neighboring absorption.

5. Discussion of Spectograms

In general, the runs made on white chart paper for illustration purposes were made at somewhat faster speeds and with much more chart concentration than was the case for the measuring runs. On some of the illustrations, sloping or bowing backgrounds are due either to foreprism effects or the joining of separate sections.

5.1. CO_2 , ν_2 Fundamental

Some 20 lines in this band were remeasured and comparison is made with the work of Rossman, Rao, and Nielsen [21-A, 21-B] and also with the values reported by Mizushima et al. (see ref. [12]). Although our measurements were somewhat handicapped by the overly strong absorption (7-m path) and also by poorer grating resolution in this region, the agreement is quite good. It appears that the values reported by Rossman, Rao, and Nielsen (also additional unpublished work communicated by Dr. Rao) are accurate and that lines they report both at higher and lower frequency as well as those given in table 1 may be used for calibration purposes provided the atmospheric absorption is augmented by CO_2 used in a vapor cell. The spectrum is illustrated in figure 1.

TABLE 1. Absorption lines of ν_2 fundamental of CO_2 from 635 to 700 cm^{-1}

<i>J</i>	<i>R</i> (<i>J</i>)			<i>P</i> (<i>J</i>)		
	Rossmann, Rao, and Nielsen ^a ν cm^{-1} (vac.) observed	This work ν cm^{-1} (vac.) observed	Mizushima et al. ^b ν cm^{-1} (vac.) observed	Rossmann, Rao, and Nielsen ^a ν cm^{-1} (vac.) observed	This work ν cm^{-1} (vac.) observed	Mizushima et al. ^b ν cm^{-1} (vac.) observed
2-----	669.75	-----	-----	665.83	-----	-----
4-----	71.34	-----	.28	64.28	-----	-----
6-----	72.88	.88	.87	62.71	-----	-----
8-----	74.45	.46	.43	61.18	.15	.15
10-----	76.02	.06	.02	59.64	.55	.60
12-----	77.60	-----	.59	58.08	.06	.09
14-----	79.20	-----	.16	56.55	.51	.53
16-----	80.78	-----	.76	55.02	4.98	.00
18-----	82.36	.39	.35	53.48	.48	.47
20-----	83.95	.98	.95	51.95	.91	.94
22-----	85.55	.56	.55	50.41	.39	.41
24-----	87.16	.17	.14	48.90	-----	.89
26-----	88.77	.79	.78	47.39	-----	.38
28-----	90.38	.36	.36	45.90	.89	.87
30-----	91.99	.97	.99	44.37	-----	.39
32-----	93.59	-----	.60	42.86	-----	.88
34-----	95.19	-----	.20	41.35	.40	.38
36-----	96.82	-----	.82	39.83	-----	.90
38-----	98.44	-----	.42	38.34	-----	.35
40-----	700.07	-----	.04	36.87	-----	.89
42-----	01.69	-----	.68	35.38	-----	.40
Q Branch--	^c (10°0→10°0)	720.46	-----	-----	-----	-----

^a Rossman, Rao, and Nielsen [21].

^b Mizushima et al. [12].

^c The measuring point of this band was taken at the 2/3 absorption point.

5.2. Acetylene, ν_3 Fundamental

This band was included in order to provide useful calibration points between the end of the strong 15- μ CO_2 band and the ammonia band. On the low-

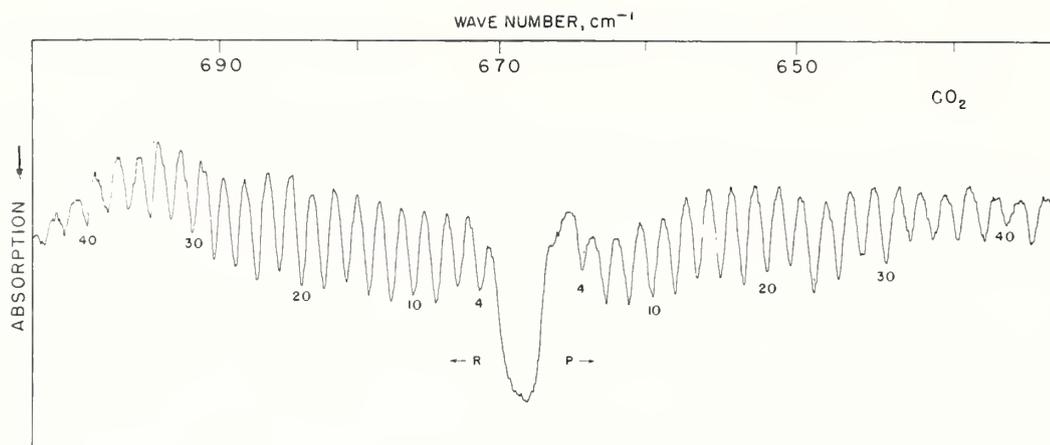


FIGURE 1. The ν_2 fundamental of CO_2 recorded in the first order of an 1,800 lines/in. grating under atmospheric conditions and 7-m path.

Spectral slit width about 0.5 cm^{-1} .

frequency end, our values are in excellent agreement with the recently published values of Rao, Ryan, and Nielsen [22] and table 2 includes additional values as reported by these authors. At higher frequency, results are also compared with the work of Christensen, Eaton, Green, and Thompson [23]. The agreement is quite good except between lines 29 to 39, where several lines appear to be pulled out of position by an overlapping band. These lines are set out by parentheses. Results of recent measurements by Jones and Nadeau of the National Research Council, Ottawa, Canada are also included. The spectrogram is illustrated in figure 2.

5.3. NH_3 , ν_2 Fundamental

Table 3 gives values obtained in four different laboratories for this band. Since these four works are of about equal quality, the average value reported in the last column represents a simple numerical average (within arbitrary $\pm 0.04\text{-cm}^{-1}$ exclusion conditions). The average values are probably better than individual determinations, and it is estimated that they are good to within $\pm 0.01\text{-cm}^{-1}$ absolute on the average. The region from 1,000 to 1,200 cm^{-1} is particularly good. We have cause for suspicioning some of our low-frequency lines (numbers 1, 3, and 6) due to the fact that the grating was being used at exceedingly high angle. As more experimental and theoretical data become available for ammonia, a good set of calculated values may ultimately replace the average value column. The spectrum is shown in figure 3.

5.4. Methane, ν_4 Fundamental

This band serves the purpose of providing connection between the ammonia and water bands and is included mainly because workers with small instruments may have difficulty in observing weak water lines in this region. We are unable to compare these

TABLE 2. Absorption lines of ν_5 fundamental of acetylene from 680 to 790 cm^{-1}

Line serial number	This work $\nu \text{ cm}^{-1}$ (vac.) observed	Rao, Ryan, and Nielsen ^a $\nu \text{ cm}^{-1}$ (vac.) observed	C., E., G., and T. ^b $\nu \text{ cm}^{-1}$ (vac.) observed	Jones and Nadeau ^c $\nu \text{ cm}^{-1}$ (vac.) observed
1		686.77		686.66
2		89.11		89.15
3		91.47		91.39
4		93.80		93.79
5		96.19		96.15
6	698.53	98.52	698.50	98.57
7		700.89	700.88	700.88
8	703.28	03.24	03.19	03.23
9	^d (05.66)	05.60	05.56	05.53
10	07.96	07.96	07.94	07.94
11	10.30	10.30	10.37	10.29
12	12.68		12.67	12.60
13	15.08		15.03	15.12
14	16.36			
15	17.35		17.35	17.35
6	19.90 (peak)		19.90	19.84
17	(22.15)		22.06	21.96
18	24.39		24.35	
19	36.17		36.18	36.14
20	38.56		38.56	38.54
21	40.89		40.88	40.85
22	43.29		43.31	43.20
23	45.64		45.64	45.65
24	47.97		48.02	47.95
25	50.36		50.37	50.29
26	52.66		52.74	52.70
27	(55.12)		55.14	55.10
28	57.42		57.45	57.43
29	(59.85)		59.86	59.68
30	62.11		62.18	62.10
31	(64.46)		64.52	64.50
32	66.74		66.87	66.68
33	69.08		69.21	69.13
34	71.39		71.53	71.46
35	73.71		73.84	73.80
36	(76.02)		76.17	76.12
37	78.42		78.52	78.42
38	(80.70)		80.82	80.73
39	83.07		83.09	83.00
40	85.43		85.45	85.37
41	87.75		87.74	
42	90.10		90.12	

^a Rao, Ryan, and Nielsen [22].

^b Christensen, Eaton, Green, and Thompson [23].

^c Jones and Nadeau, National Research Council, Ottawa, Canada, unpublished work.

^d Parentheses around values in second column indicate poorer lines.

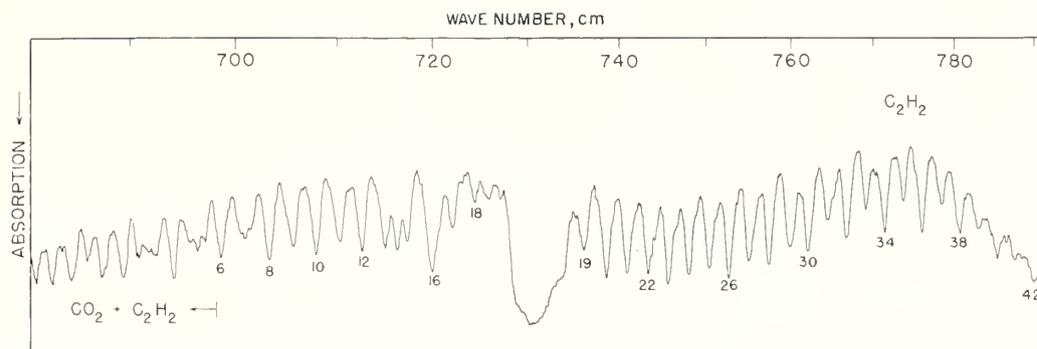


FIGURE 2. The ν_5 fundamental of acetylene recorded in the first order of an 1,800 lines/in. grating with 8-cm pressure and 5-cm path.

Spectral slit width about 0.7 cm^{-1} . The low frequency end of the P branch overlaps with atmospheric absorption of CO_2 and the strong Q branch (hot band) of CO_2 at 720.46 cm^{-1} also contributes to absorption at this point.

TABLE 3. Absorption lines of ν_2 fundamental of NH_3 from 760 to 1,210 cm^{-1}

Line serial number	This work ν cm^{-1} (vac.) observed	Garing and Nielsen ^a ν cm^{-1} (vac.) observed	Price and coworkers ^b ν cm^{-1} (vac.) observed	Mizushima et al. ^c ν cm^{-1} (vac.) observed	Average value ^d ν cm^{-1} (vac.)
1	760.72	.69	.69	-----	.70
2	70.96	.91	.90	.96	.93
3	78.33	.29	.27	.33	.30
4	91.76	.72	.76	.75	.75
5	96.17	.14	.16	.15	.16
6	809.76	.72	.71	.74	.73
7	14.27	.25	.22	.24	.24
8	30.68	.65	.70	.67	.68
9	34.86	.82	.84	.83	.84
10	51.36	.32	.36	.32	.34
11	52.77	.71	.76	.73	.74
12	67.83 (blend)	(.53, .74, .93)	(.53, .73, .96)	.82	.82
13	71.77	.73	.74	.79	.76
14	72.59	.56	.58	.58	.58
15	87.99	.99	8.02	.96	.99
16	92.06 (blend)	(1.88, .14)	(1.88, .14)	.10	.08
17	908.21	.17	.15	.24	.18
18	18.65	.62	.61	-----	.63
19	35.85 (poor)	.90	.90	-----	.88
20	48.25	.22	.25	.27	.25
21	51.83	.77	.80	-----	.80
22	61.01 (poor)	.06	(0.89, .12)	-----	-----
23	71.91	.89	.92	.90	.90
24	91.71	.68	.71	-----	.70
25	92.60 (blend)	(.45, .70)	(.46, .71)	.64	.62
26	1,007.55	.54	.56	.48	.55
27	11.20	.20	.22	.24	.22
28	27.04	.04	.04	6.92	.04
29	32.14	.13	.13	.12	.13
30	46.41	.40	.42	.27	.41
31	53.14	.13	.14	.14	.14
32	65.58	.57	.55	.46	.57
33	70.60	.59	.59	.53	.59
34	74.17	.14	.17	.13	.15
35	84.61	.60	.62	.56	.61
36	89.38	.42	.39	.31	.40
37	95.15	.15	.16	.15	.15
38	1,103.44	.46	.42	.44	.44
39	10.67	.69	.69	.67	.68
40	16.02	.03	.03	5.97	.03
41	22.15	.14	.13	.10	.14
42	31.85	.86	.89	-----	.87
43	36.78	.76	.75	-----	.76
44	40.62	.65	.65	.58	.64
45	47.53	.52	.54	-----	.53
46	52.86	.86	.87	-----	.86
47	58.93	.98	.95	.90	.95
48	77.09	.08	.10	-----	.09
49	95.02	.00	4.99	-----	5.00
50	1,212.66	.68	.69	-----	.68

^a Garing, Nielsen, and Rao [24].

^b Price and coworkers [33].

^c Mizushima et al. [12].

^d Numerical average of the 4 determinations. If a number differed by 0.04 cm^{-1} or more from the average of the other 3, it was excluded from the average.

numbers with others, as there are no other precision measurements available but our results (see table 4) would appear to be good to about $\pm 0.02\text{-cm}^{-1}$ absolute. The spectrum is shown in figure 4.

5.5. H_2O , ν_2 Fundamental

Some 50 good lines of varying intensity were picked out for measurement. (See table 5.) Wherever possible, the lines are compared with recent precision determinations of Rao, Ryan, and Nielsen (see ref. [22]). These authors indicate that their values are, on the average, about 0.07-cm^{-1} lower than those reported by Dalby and Nielsen [25] for the region 1,450 to 1,650 cm^{-1} . Our results, when compared with Dalby and Nielsen's values, would indicate that this is the case throughout the band. Wherever possible, mild flushing or pumping down of the spectrometer housing is to be recommended for cleaner development of the lines but our results indicate that good calibration measurements can be made even on saturated lines provided the line is not a complex one. This band is illustrated in figure 5.

TABLE 4. Substructure of ν_4 band of CH_4 from 1,200 to 1,370 cm^{-1}

Serial number	This work ν cm^{-1} (vac.) observed
1	1,216.26
2	30.12
3	33.47
4	41.01
5	47.81
6	56.60
7	65.52
8	75.20
9	81.59
10	92.61
11	97.63
12	1,311.42
13	22.09
14	27.20
15	32.41
16	41.79
17	46.70
18	53.06
19	66.04

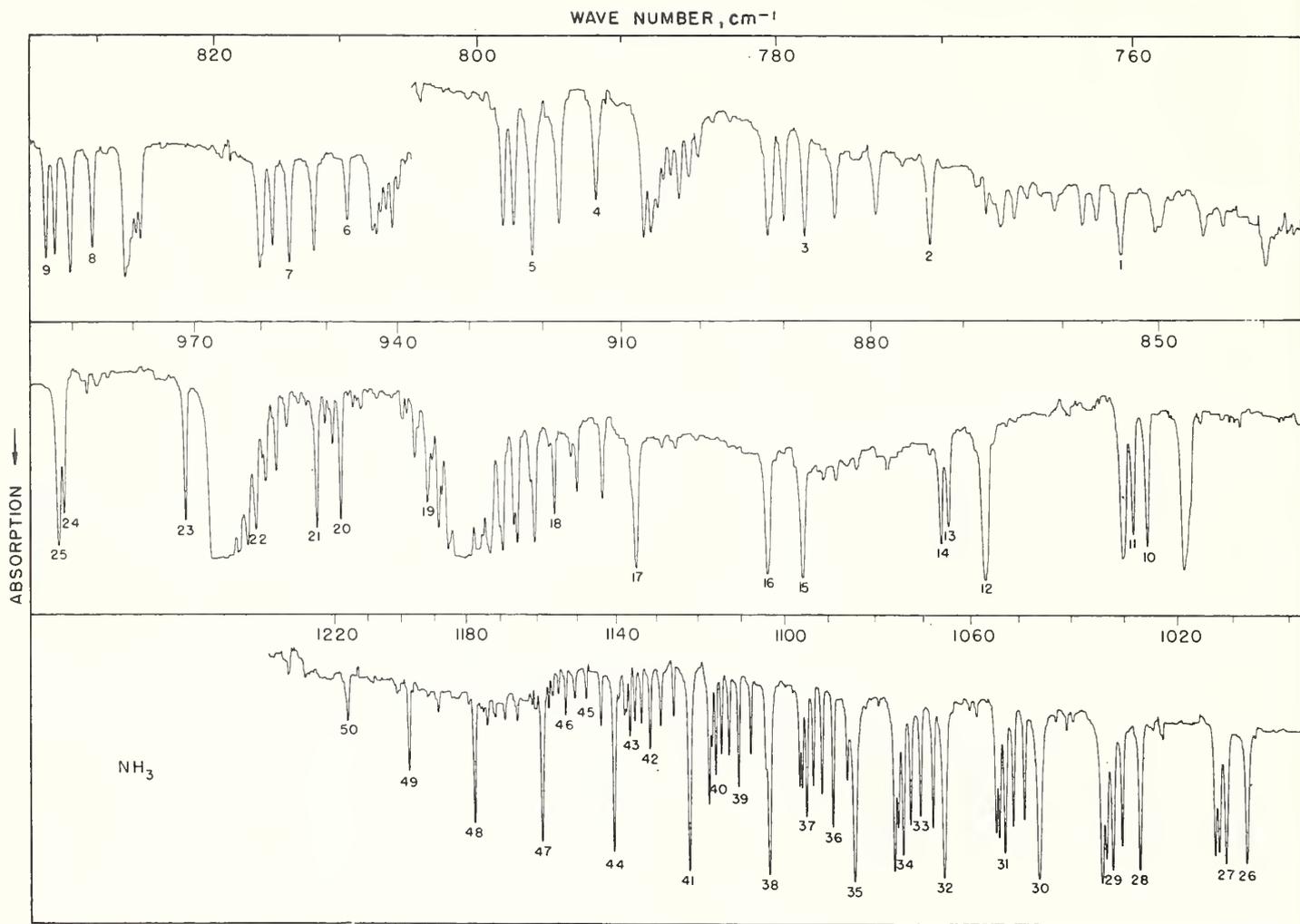


FIGURE 3. The ν_2 fundamental of NH_3 recorded in the first order of a 3,600 lines/in. grating with 7-cm pressure near the band center and 12-cm for the wings, both with 5-cm path. Spectral slit width about 0.3 cm^{-1} .

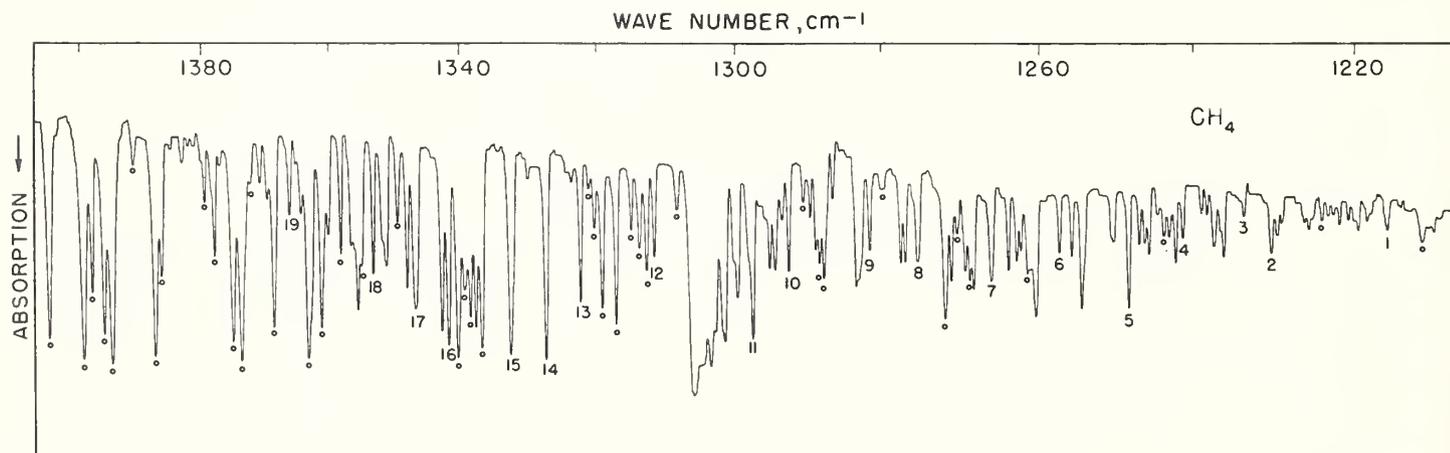


FIGURE 4. The ν_4 fundamental of CH_4 recorded in the first order of a 3,600 lines/in. grating with 12-cm pressure and 5-cm path. Spectral slit width about 0.5 cm^{-1} . The open circles refer to atmospheric water peaks.

TABLE 5. Absorption lines of ν_2 fundamental of H_2O from 1,300 to 2,000 cm^{-1}

Line serial number	This work ν cm^{-1} (vac.) observed	Rao, Ryan, and Nielsen ^a ν cm^{-1} (vac.) observed	Line serial number	This work ν cm^{-1} (vac.) observed	Rao, Ryan, and Nielsen ^a ν cm^{-1} (vac.) observed
1	1,312.55	-----	26	16.77	16.72
2	18.92	-----	27	27.88	-----
3	36.68	-----	28	37.53	-----
4	54.83	-----	29	40.31	-----
5	61.08	-----	30	62.82	-----
6	68.62	-----	31	71.49	-----
7	75.07	-----	32	90.15	-----
8	94.46	-----	33	1,704.49	-----
9	1,404.99	-----	34	10.23	-----
10	17.42	-----	35	23.49	-----
11	29.94	-----	36	56.81	-----
12	47.93	-----	37	68.17	-----
13	52.04	-----	38	75.64	-----
14	64.92	64.91	39	84.95	-----
15	81.25	-----	40	^b (90.94)	-----
16	87.29	-----	41	99.60	-----
17	98.80	-----	42	1,810.63	-----
18	1,512.30	-----	43	25.24	-----
19	28.57	-----	44	47.80	-----
20	^b (45.09)	-----	45	69.35	-----
21	64.93	64.91	46	89.59	-----
22	94.52	94.51	47	95.19	-----
23	96.29	96.27	48	1,918.05	-----
24	1,601.25	01.23	49	42.60	-----
25	09.46	-----	50	88.43	-----
			51	^b (2,016.79)	-----

^a Rao, Ryan, and Nielsen [22].
^b These lines are less accurate.

5.6. CO Fundamental

The CO fundamental band at 4.67 μ was measured accurately by Plyler, Blaine, and Connor (see ref. [13]) in 1955. With the measurement of the CO harmonic (2.33 μ), it was possible to calculate the position of the fundamental band. This has been done by Rank and his colleagues (see ref. [14]) and a very good agreement with the observed values was obtained. There was a slight difference in values for the high J 's in the R branch. Recently the R branch has been re-measured and a better correspondence with the calculated values is obtained. The accuracy of the calculated values approaches a few thousandths of a cm^{-1} . The spectrum is shown in figure 6 and the values are listed in table 6.

5.7. $C^{13}O_2$, ν_3 Fundamental

The $C^{13}O_2$ band is excellent for calibration from 2,240 to 2,280 cm^{-1} and connects with the ν_3 band of $C^{12}O_2$. This spectrum was measured in 1955 by Plyler, Blaine, and Tidwell [26]. In this work, the lines of this band have been re-measured and the values check closely with the first determination. Unfortunately, the J values were incorrectly listed in table 1 of the above publication and each J of the P branch should be reduced by 2; that is, P 44 should

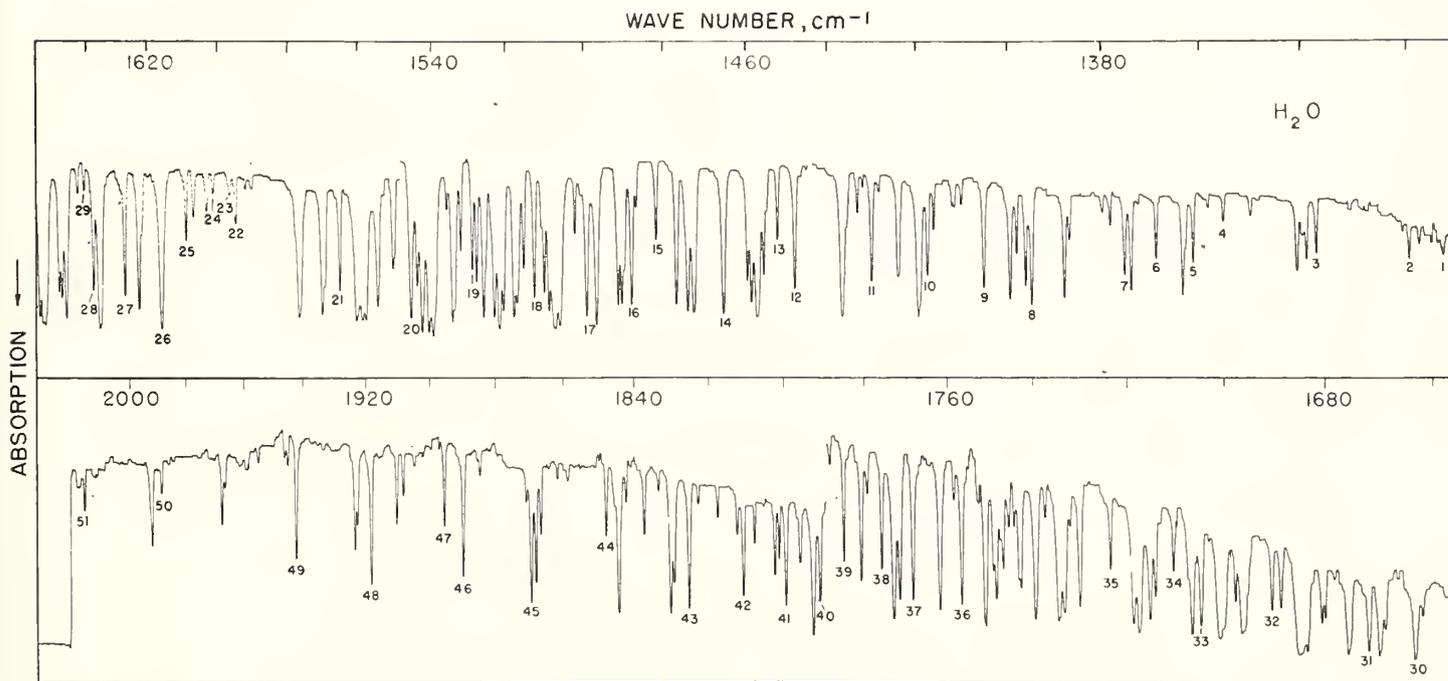


FIGURE 5. The ν_2 fundamental of H_2O recorded with a 3,600 lines/in. grating under atmospheric conditions ($T=21^\circ C$, humidity 21%) and 7-m path.

The upper spectrogram was recorded in the first order and the lower in the second order. Spectral slit width about 0.6 to 0.7 cm^{-1} for both spectrograms.

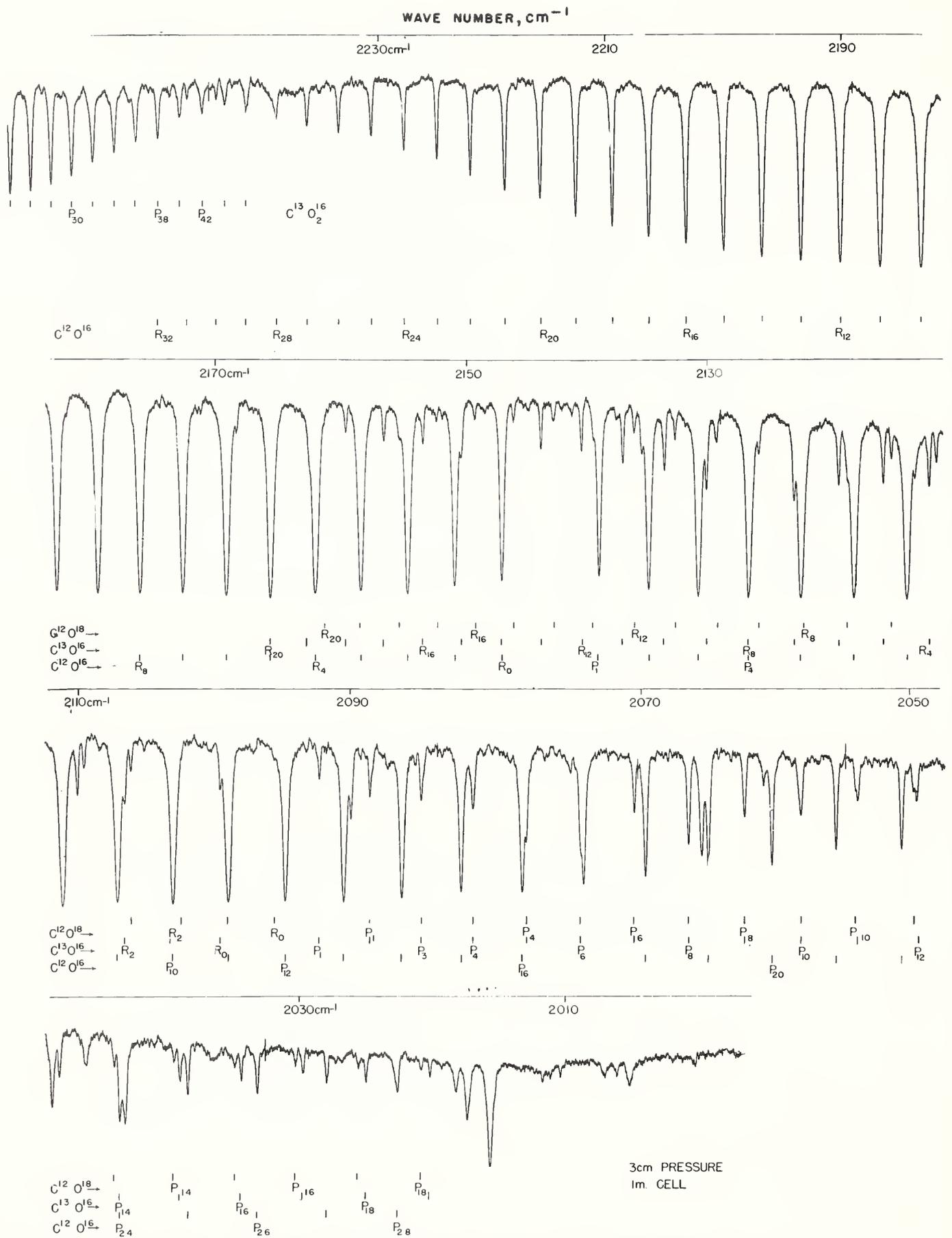


FIGURE 6. Absorption band of CO recorded in the first order of a 7,500 lines/in. grating with 3-cm pressure and 1-m path. Spectral slit width about 0.3 cm^{-1} .

TABLE 6. Absorption lines of CO from 2,020 to 2,240 cm^{-1}

Line No.	Plyler, Blaine, and Connor ^a ν cm^{-1} (vac.) observed	Rank et al. ^b ν cm^{-1} (vac.) calculated	Line No.	Plyler, Blaine, and Connor ^a ν cm^{-1} (vac.) observed	Rank et al. ^b ν cm^{-1} (vac.) calculated
P 28	2,022.899	.915	R 0		
27	27.635	.650	1		
26	32.349	.354	2	2,154.596	.588
25	37.030	.026	3	58.309	.302
24	41.663	.668	4		
23	46.271	.278	5	65.602	.604
22			6		
21	55.391	.402	7	72.759	.761
20	59.911	.916	8	76.287	.286
19			9	79.761	.774
18	68.851	.849	10	83.226	.226
17			11	86.636	.641
16	77.650	.652	12	90.010	.020
15	82.009	.005	13	93.357	.361
14	86.322	.324	14	96.661	.665
13	90.603	.611	15	99.929	.933
12	94.870	.835	16	2,203.147	.163
11	99.096	.085	17	06.345	.355
10	2,103.265	.272	18	09.498	.510
9	07.413	.426	19	12.600	.626
8	11.555	.546	20	15.685	.705
7			21	18.733	.746
6	19.677	.684	22	21.732 ^c 21.750	.749
5	23.700	.702	23	24.694 24.712	.713
4			24	27.638	
3	31.639	.635	25	30.526	
2	35.554	.549	26	33.362	
1	39.432	.429	27	36.186	
			28	38.958	

^a Plyler, Blaine, and Connor [13].

^b Rank et al. [14].

^c New measurements agreeing more closely with calculated values.

be P 42, etc. The work with a small grating spectrometer of Mizushima and his colleagues (see ref. [12]) has also been included and there is very good agreement between the two lists of frequencies. The spectrum of this band is shown in figure 7-A and the values are listed in table 7-A.

5.8. C^{12}O_2 , ν_3 Band

The only precision work for this band is the work which was done at the NBS. The frequencies were first published in 1955 (see ref. [26]) and since then two other determinations have been made. With the long paths employed in this laboratory, it is not

TABLE 7-A. Absorption lines of ν_3 fundamental of C^{13}O_2 ¹⁶ from 2,240 to 2,300 cm^{-1}

Line number	This work ν cm^{-1} (vac.) observed	Previous work Plyler, Blaine, and Tidwell ^a ν cm^{-1} (vac.) observed	Mizushima et al. ^b ν cm^{-1} (vac.) observed	Line number	This work ν cm^{-1} (vac.) observed	Previous work this laboratory ^c ν cm^{-1} (vac.) observed	Mizushima et al. ^b ν cm^{-1} (vac.) observed
P 40	2,247.68	2,247.86		R 0	2,284.24		
38	49.68	.68		2	85.79	.76	.83
36	51.70	.67		4	87.31	.33	.34
34	53.66			6	88.81	.80	.83
32	55.59			8	90.23	.20	.29
				10	91.62	.62	.73
30	57.52	.52	2,257.48	12	93.10	.12	
28	59.41	.42		14	94.45	.46	
26	61.29	.30	.35	16	95.89		.91
24	63.13	.14	.30	18	97.18	.20	.26
22	64.95	.96	5.04	20	98.50		.57
				22	99.76	.74	
20	66.75	.76	.85	24	2,301.05	.06	
18	68.54	.55	.65				
16	70.29	.31	.37				
14	72.03						
12	73.73	.74	.73				
10	75.42						
8	77.09	.08	.03				
6	78.71	.71	.64				
4	80.33	.31	.18				
2	81.94						

^a Plyler, Blaine, and Tidwell [26].

^b Mizushima et al. [12].

^c E. K. Plyler and L. R. Blaine, unpublished work.

possible to measure the central part of the band. Figure 7-B shows how use can be made of the entire band by purging the instrument housing and containing the CO_2 in a cell. (Dr. Norman Jones of the National Research Council of Canada has furnished the spectogram as observed with a purged small grating instrument.) Table 7-B gives observed values for the band wings and calculated values for the central part. In view of the fact that molecular constants calculated from the observed lines agree well with those of Courtoy [27] obtained from many bands and agreement with observed lines is good, it is felt that workers can use either the observed or calculated values of table 7-B equally well provided the intensity and resolution of the band is close to that of figure 7-B. Both

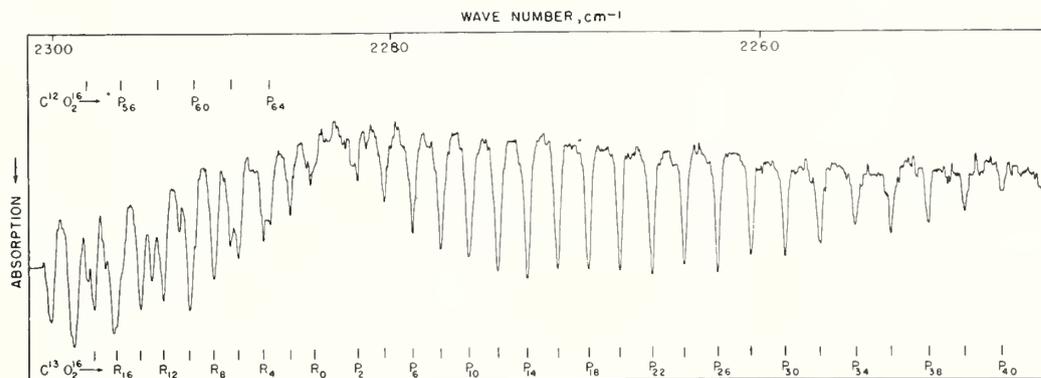


FIGURE 7-A. The ν_3 fundamental of C^{13}O_2 ¹⁶ recorded in the first order of a 7,500 lines/in. grating under atmospheric conditions and 6-m path.

Spectral slit width about 0.3 cm^{-1} .

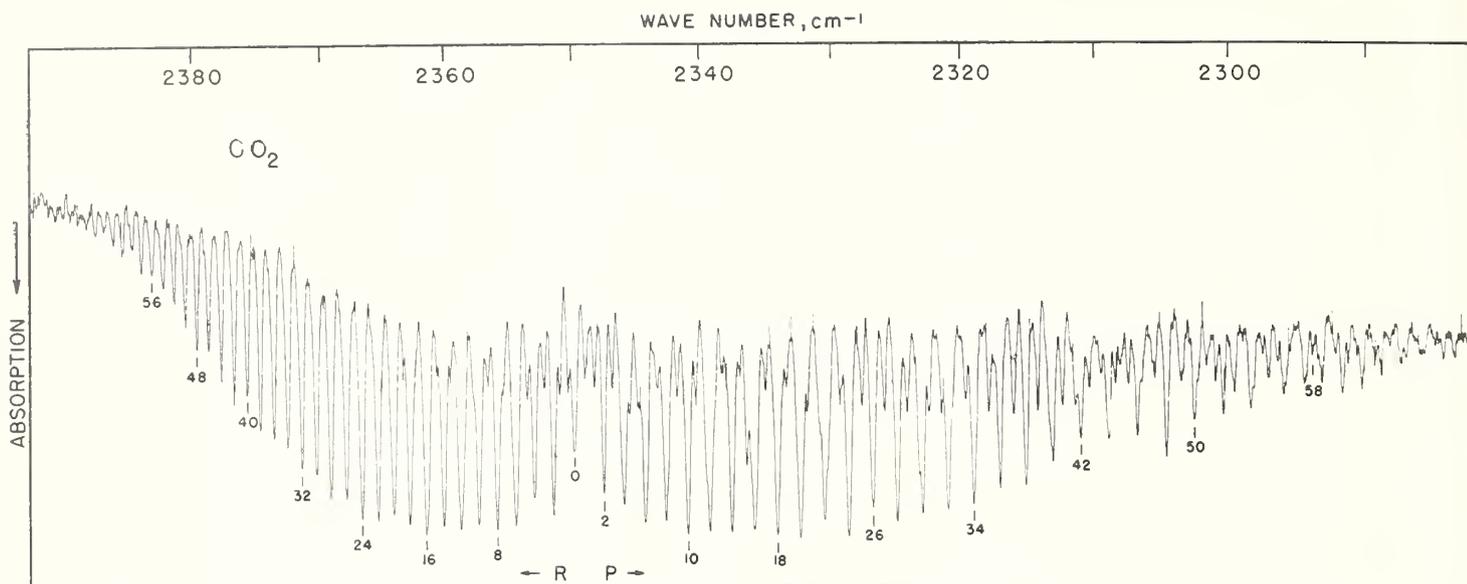


FIGURE 7-B. The ν_3 fundamental of $C^{12}O_2^{16}$ recorded in the third order of a 70 lines/mm grating with a small grating instrument. The spectrometer housing was purged of atmospheric CO_2 with dry nitrogen and the spectrum recorded with 30-mm CO_2 in a 10-cm cell. Spectral slit width about 0.25 cm^{-1} .

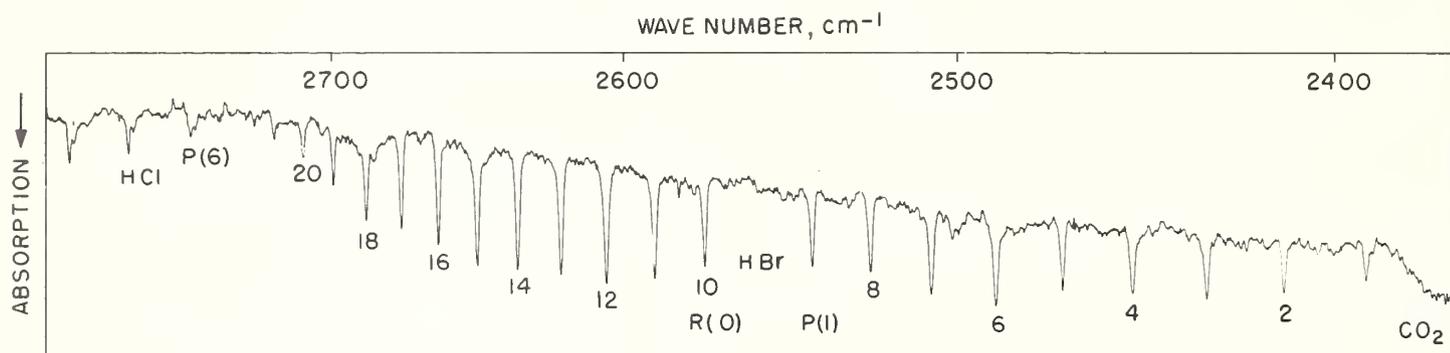


FIGURE 8-A. Absorption band of HBr recorded in the second order of a 5,000 lines/in. grating with 40-cm pressure and 5-cm path. Spectral slit width about 0.8 cm^{-1} . The CO_2 band just appears at the low frequency end and the HCl band at the high frequency side. HCl was present in the HBr as an impurity.

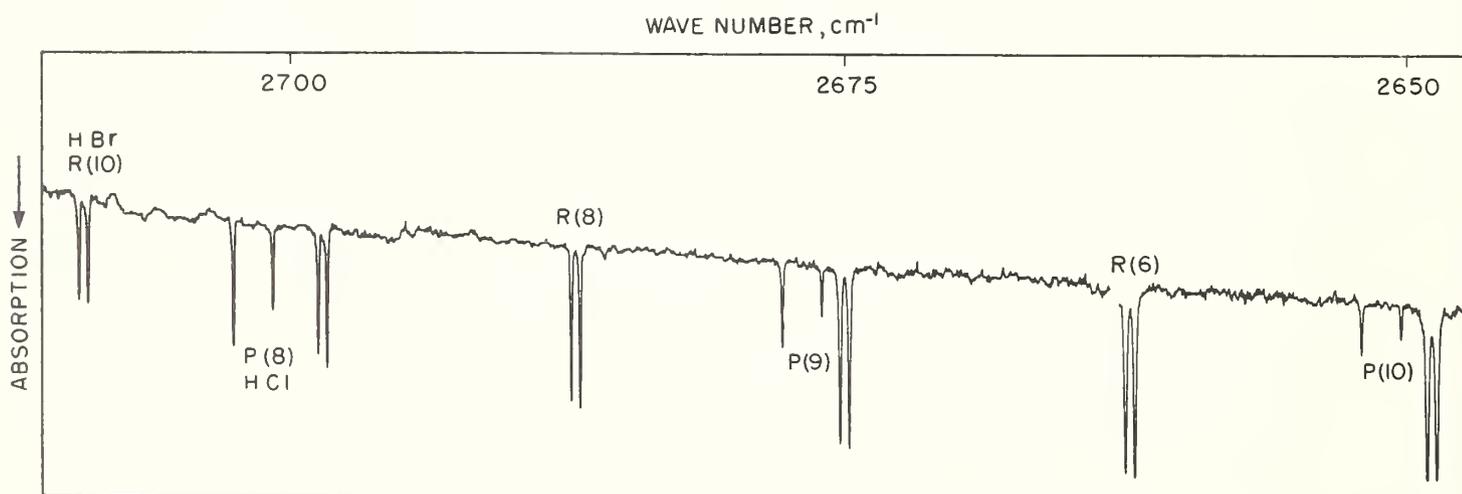


FIGURE 8-B. A section of the HBr $79, 81$ bands with HCl $35, 37$ overlapping. Recorded with a 10,000 lines/in. grating singly passed with 2.5-mm pressure of each acid in a 6-m cell. Spectral slit width about 0.06 cm^{-1} .

TABLE 7-B. Absorption lines of ν_3 fundamental of $C^{12}O_2^{16}$ from 2,280 cm^{-1} to 2,390 cm^{-1}

Line number	This work ν cm^{-1} (vac.) observed	Previous work this laboratory ν cm^{-1} (vac.) observed	Line number	This work ν cm^{-1} (vac.) observed	Previous work this laboratory ν cm^{-1} (vac.) observed
P 68	2,282.21	-----	R 48	-----	2,379.80
66	-----	-----	50	-----	80.74
64	86.93	-----	52	-----	81.65
62	89.23	-----	54	-----	82.51
60	-----	-----	56	2,383.39	83.38
58	93.81	2,293.81	58	84.22	84.21
56	96.03	96.03	60	85.01	85.01
54	98.30	-----	62	85.80	85.79
52	2,300.50	-----	64	86.55	86.55
50	-----	2,302.70	66	-----	87.26
48	-----	04.84	68	-----	87.97
46	-----	06.96	70	-----	88.64
R 30	-----	2,370.36	72	-----	89.32
32	-----	71.48	74	-----	89.91
34	-----	72.65	76	-----	90.49
36	-----	73.71	78	-----	91.10
38	-----	74.79	80	-----	91.61
40	-----	75.83	82	-----	92.16
42	-----	76.87	84	-----	-----
44	-----	77.88	86	-----	93.09
46	-----	78.86	88	-----	93.55
			90	-----	93.96

Calculated values for lines near band center

P 44	2,309.05	R 0	2,349.97
42	11.13	2	51.50
40	13.19	4	53.01
38	15.22	6	54.49
36	17.23	8	55.95
34	19.21	10	57.38
32	21.17	12	58.78
30	23.11	14	60.16
28	25.02	16	61.52
26	26.91	18	62.86
24	28.77	20	64.16
22	30.60	22	65.44
20	32.41	24	66.70
18	34.20	26	67.93
16	35.97	28	69.13
14	37.71		
12	39.43		
10	41.12		
8	42.78		
6	44.42		
4	46.04		
2	47.63		

^a E. K. Plyler and L. R. Blaine, unpublished work.

observed and calculated values should be good to about ± 0.02 - cm^{-1} absolute. Since the band consists of a collection of no less than four overlapped bands, results are subject to being extremely resolution sensitive and workers should not use lines exhibiting pronounced overlap. (The *R* branch is comparatively free of such difficulties.)

5.9. HBr Fundamental

As shown in figures 8-A and 8-B, this band was measured under considerably different resolution. With the conditions describing figure 8-A, there was no evidence to the effect that the components arising from HBr^{79} and HBr^{81} were being resolved out. This was done intentionally to see whether, when measured as a single peak, the frequency agreed with the average determined for each component under

resolution approaching 0.05 cm^{-1} . The low resolution single-peak frequency agreed with the high resolution average value to within 0.05 cm^{-1} on the average (See table 8). This relatively good agreement does indicate that the high resolution results are correct in the absolute sense and that apart from greater inability to determine the center of the single

TABLE 8. Absorption lines of HBr^{79} and HBr^{81} together with single unresolved peaks at lower resolution (2,390-2,750 cm^{-1})

Serial number	This work high resolution isotopic components ν cm^{-1} (vac.) observed	High resolution average	This work low resolution single peak ν cm^{-1} (vac.) observed
1	Br ⁸¹ 2,392.61	2,392.78	-----
	Br ⁷⁹ 92.94		
2	Br ⁸¹ 2,412.73	2,412.89	2,412.93
	Br ⁷⁹ 13.06		
3	Br ⁸¹ 32.43	32.60	32.65
	Br ⁷⁹ 32.77		
4	Br ⁸¹ 51.73	51.91	51.94
	Br ⁷⁹ 52.08		
5	Br ⁸¹ 70.61	70.79	70.82
	Br ⁷⁹ 70.97		
6	Br ⁸¹ 89.08	89.26	89.30
	Br ⁷⁹ 89.43		
7	Br ⁸¹ 2,507.11	2,507.30	2,507.24
	Br ⁷⁹ 07.48		
8	Br ⁸¹ 24.71	24.90	24.83
	Br ⁷⁹ 25.08		
9	Br ⁸¹ 41.87	42.06	41.99
	Br ⁷⁹ 42.25		
10	Br ⁸¹ 74.80	74.99	74.98
	Br ⁷⁹ 75.19		
11	Br ⁸¹ 90.56	90.76	90.67
	Br ⁷⁹ 90.95		
12	Br ⁸¹ 2,605.82	2,606.02	2,605.95
	Br ⁷⁹ 06.22		
13	Br ⁸¹ 20.63	20.83	20.81
	Br ⁷⁹ 21.03		
14	Br ⁸¹ 34.92	35.13	35.03
	Br ⁷⁹ 35.33		
15	Br ⁸¹ 48.71	48.92	48.85
	Br ⁷⁹ 49.13		
16	Br ⁸¹ 61.99	62.19	62.13
	Br ⁷⁹ 62.39		
17	Br ⁸¹ 74.76	74.97	-----
	Br ⁷⁹ 75.19		
18	Br ⁸¹ 87.00	87.20	-----
	Br ⁷⁹ 87.41		
19	Br ⁸¹ 98.68	98.90	-----
	Br ⁷⁹ 99.11		
20	Br ⁸¹ 2,709.86	2,710.07	-----
	Br ⁷⁹ 10.29		
21	Br ⁸¹ 20.48	20.68	-----
	Br ⁷⁹ 20.89		
22	Br ⁸¹ 30.51	30.73	-----
	Br ⁷⁹ 30.95		
23	Br ⁸¹ 40.02	40.24	-----
	Br ⁷⁹ 40.46		
24	Br ⁸¹ 48.93	49.16	-----
	Br ⁷⁹ 49.39		

peak (near 1-cm^{-1} half-width), its frequency is given well enough by the component average. In table 8, the high resolution average of components is the recommended value for single peak use. Calculated values were also determined for the high resolution results and the values given in the first column of table 8 are believed to be accurate to within $\pm 0.02\text{-cm}^{-1}$ absolute.

5.10. HCl Fundamental

The spectrum illustrated in figure 9 was recorded under low resolution and the entire band shown in this way for the sake of compression. Figure 8-B shows 3 lines of the band under high resolution. The band was measured on both instruments in this laboratory and results are compared with values reported by Mills, Thompson, and Williams [28] (see table 9). Molecular constants were obtained from the high resolution observations and calculated line frequencies agree well with the observed. In general, the high resolution results of this work fall between the low resolution values and those of Mills, Thompson, and Williams. The high resolution work does not, however, represent precision measurement, since the lines were measured at medium-fast scan in order to appropriately cover this extremely wide band in reasonable time. The observed values given in the table for the high resolution work are to be preferred and represent the best possible set. These values should be good to about $\pm 0.02\text{-cm}^{-1}$ absolute. (Future precision measurements are contemplated for this band.)

5.11. Methane, ν_3 Band

The *R* branch of this band and some of the lower-*J* lines of the *P* branch are fairly suitable for providing calibration points in this region. However, care should be exercised to the extent of staying within the bounds of the resolution illustrated in figure 10.

TABLE 9. Absorption lines of HCl from 2,650 to 3,050

Serial number	This work high resolution ν cm^{-1} (vac.) observed	This work high resolution ν cm^{-1} (vac.) calculated	This work low resolution ν cm^{-1} (vac.) observed	M., T., and W. ^a ν cm^{-1} (vac.) observed
HCl ³⁵				
0	2,651.98	2,651.98	-----	.94
1	77.74	.75	.86	.77
2	2,703.02	2,703.02	.03	2.95
3	27.79	.79	.76	.75
4	52.05	.05	.15	.01
5	75.77	.77	.85	.77
6	98.95	.95	.96	9.00
7	2,821.58	2,821.57	.56	.59
8	43.63	.63	.60	.63
9	65.10	.10	.14	.14
10	2,906.25	2,906.25	.24	.25
11	25.91	.90	.89	.92
12	44.92	.92	-----	.99
13	63.30	.30	.20	.35
14	81.02	.02	-----	.05
15	98.07	.07	-----	.05
16	3,014.44	3,014.44	-----	.50
17	30.10	.11	.12	.12
18	45.07	.08	.02	.15
HCl ³⁷				
0	2,650.23	2,650.24	-----	.17
1	75.96	.96	-----	.98
2	2,701.20	2,701.19	.28	.15
3	25.93	.93	.95	.90
4	50.14	.14	-----	.13
5	73.84	.83	.89	.82
6	96.98	.98	-----	7.01
7	2,819.57	2,819.57	-----	.56
8	41.59	.59	-----	.56
9	63.02	.03	-----	.06
10	2,904.12	2,904.12	.14	.07
11	23.74	.74	.79	.74
12	42.74	.73	.66	.79
13	61.08	.08	.06	.13
14	78.77	.77	-----	.80
15	95.79	.79	-----	.78
16	3,012.15	3,012.14	.14	.23
17	27.80	.79	.72	.84
18	42.74	.74	.68	.80

^a Mills, Thompson and Williams [28].

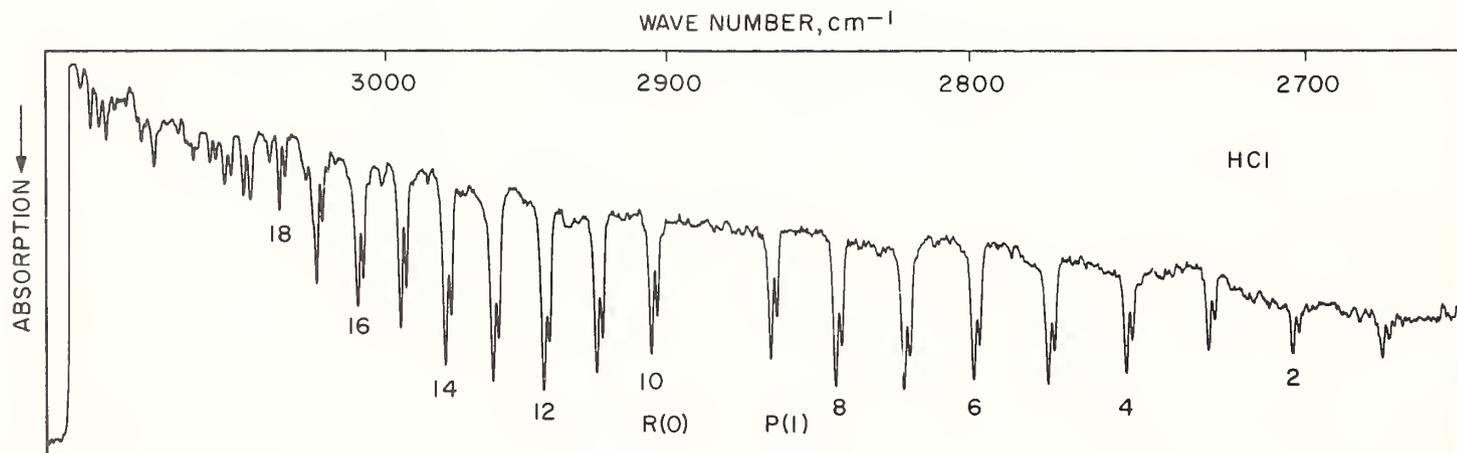


FIGURE 9. Absorption band of HCl recorded in the second order of a 5,000 lines/in. grating with 20-cm pressure and 5-cm path. Spectral slit width about 1.0-cm^{-1} . Weak atmospheric water lines of the $2\nu_2$ band begin to appear at the high frequency end.

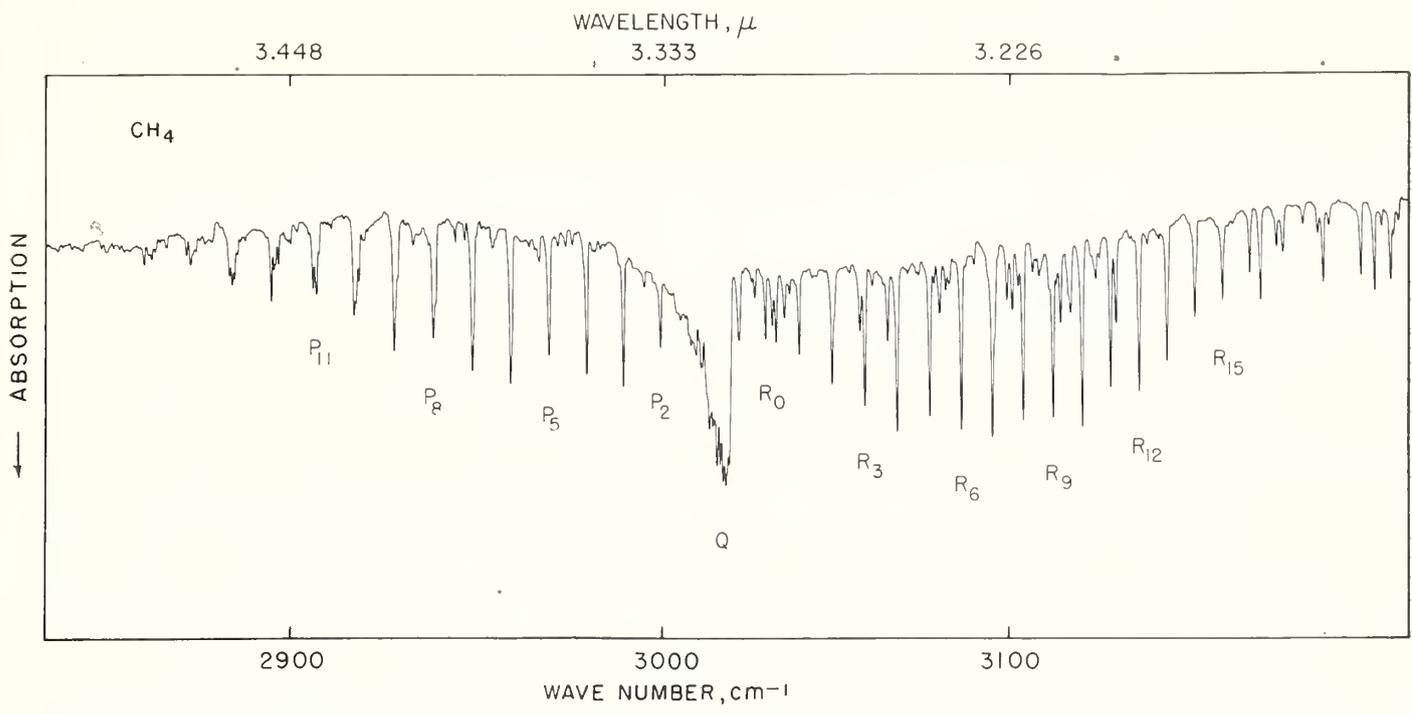


FIGURE 10. The ν_2 fundamental of methane recorded with a 7,500 lines/in. grating with 10-cm pressure and 5-cm path. Spectral slit width about 0.9 cm^{-1} , certain weak lines occurring between methane lines are part of the $2\nu_2$ band of atmospheric water vapor.

Under higher resolution the lines begin to break up, P_4 , for example, shows four components within 0.37 cm^{-1} and frequency allocation becomes difficult. The values are listed in table 10 and strongest components should be used where indicated.

5.12. Acetylene Bands Near $3,300 \text{ cm}^{-1}$

The spectrogram of figure 11 gives a rather interesting comparison of lines and peaks under considerably differing resolution. The scale on the lower

TABLE 10. Absorption lines of methane from 2,900 to 3,170 cm^{-1}

Line number	Plyler, Blaine, and Nowak ^a $\nu \text{ cm}^{-1}$ (vac.) observed	Line number	Plyler, Blaine, and Nowak ^a $\nu \text{ cm}^{-1}$ (vac.) observed
P_{12}	^b 2,895.18	R_0	3,028.84
11	^b 2,906.72	1	38.58
10	^b 16.36	2	48.25
9	26.86	3	57.79
8	37.34	4	67.30
7	47.92	5	76.74
6	58.20	6	86.02
5	68.67	7	95.22
4	79.00	8	3,104.36
3	88.27	9	13.42
2	99.10	10	22.46
		11	31.39
		12	40.20
		13	48.95
		14	57.61
		15	66.20

^a Plyler, Blaine, and Nowak [11].
^b Frequency of strongest component.

panel, though close to that of the upper one differs somewhat and is indicated by the tie lines. The values listed in table 11 indicate the rather remarkable feature that even under considerably lower resolution single or symmetrical lines can still be accurately measured. The high resolution measurements were made with fringes and the grating circle was used for the lower resolution measurements. In two cases (lines 6 and 7; 20 and 21), two closely spaced and nearly equal intensity components were measured as a single peak and this value compared with the average of the two components. As in the case of HBr, the agreement is quite satisfactory. The results of this work are compared with values reported by Christensen, Eaton, Green, and Thompson (see ref. [23]).

5.13. Water and Carbon Dioxide Bands Near $3,700 \text{ cm}^{-1}$

The spectrogram (fig. 12-A), illustrating the atmospheric absorption in this region, was recorded with the low resolution instrument so that results would approximate those attainable with small grating instruments. The indicated lines were measured under these conditions and in table 12-A the results are compared with the high resolution and high precision work of Plyler and Tidwell [29]. The agreement is satisfactory and indicates that even with lower resolution, good measurements in the order of 1 part in 100,000 can still be made. The higher resolution numbers are the recommended ones and if more frequent calibrating points are required, workers should make use of reference [29].

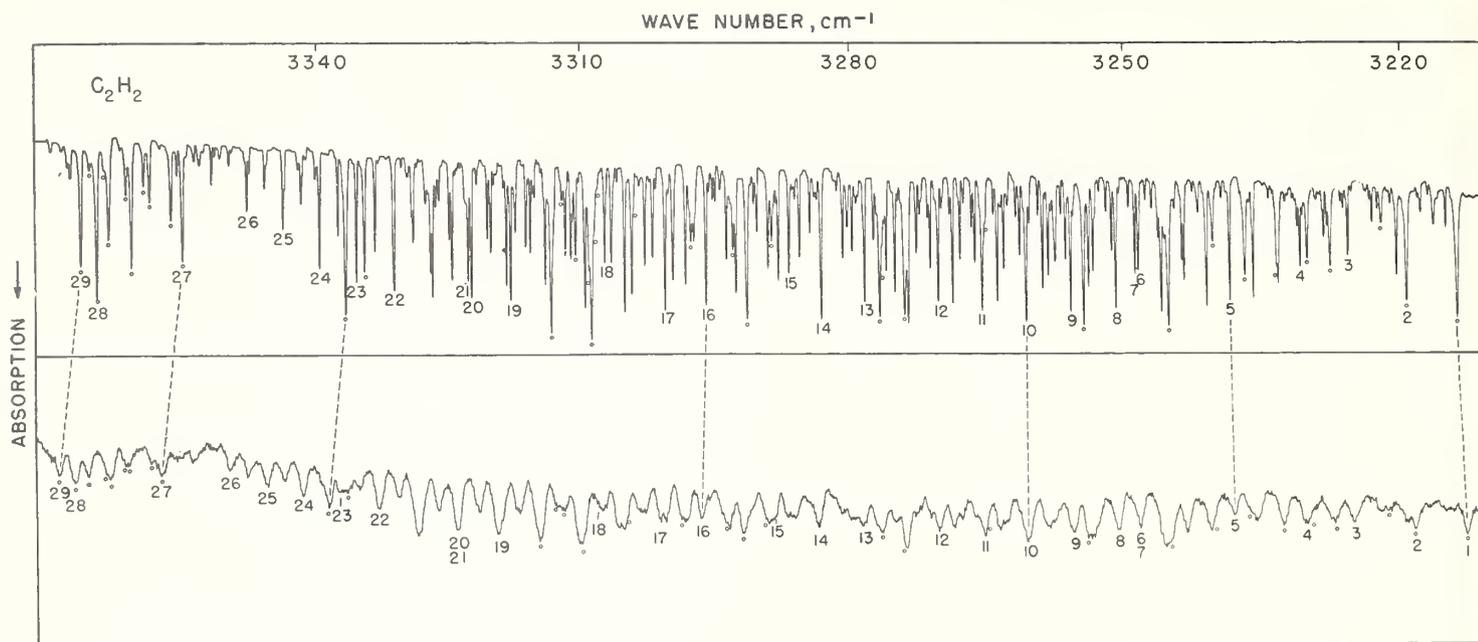


FIGURE 11. The two absorption bands of acetylene near $3,300\text{ cm}^{-1}$ compared under high and lower resolution.

The upper spectrogram was recorded with a 10,000 lines/in. grating singly passed with 1.5-cm pressure and 5-cm path. The lower spectrogram was recorded in the second order of a 5,000 lines/in. grating with 4-cm pressure and 5-cm path. Spectral slit widths about 0.10 and 0.8 cm^{-1} , respectively. Atmospheric water lines of the $2\nu_2$ band are denoted by open circles.

TABLE 11. Absorption bands of acetylene (2 bands) from $3,200$ to $3,380\text{ cm}^{-1}$

Serial number	This work medium-high resolution $\nu\text{ cm}^{-1}$ (vac.) observed	This work low resolution $\nu\text{ cm}^{-1}$ (vac.) observed	C., E., G., and T ^b medium resolution $\nu\text{ cm}^{-1}$ (vac.) observed
2	3,219.42 (H ₂ O)	-----	-----
3	25.63	-----	.64
4	30.72	-----	.72
5	38.31	.44	.29
6	48.19 ^a 48.38	48.41	.34 ^a 48.40
7	48.56	-----	.47
8	50.71	.82 (slightly blended)	.65
9	55.59	-----	.56
10	60.46	.60 (slightly blended)	.40
11	65.29	.32	.26
12	70.10	-----	.04
13	78.22	-----	.20
14	83.04	.14	2.93
15	86.63	-----	.48
16	95.88	.80	.83
17	3,300.46	-----	.35
18	07.27	-----	.14
19	17.92	-----	.99
20	22.41 ^a 22.59	22.62	22.44
21	22.77	-----	-----
22	31.29	.31	.18
23	35.59	-----	.49
24	39.83	.98	.72
25	44.08	.01 (slightly blended)	3.93
26	48.23	.21	.08
27	55.74 (H ₂ O)	-----	-----
28	65.78 (H ₂ O)	.86	-----
29	67.67 (H ₂ O)	-----	-----

^a Average of two components of nearly equal intensity.

^b Christensen, Eaton, Green, and Thompson [23].

5.14. CO₂, O₂1, and 101 Bands

Plyler and Tidwell (see ref. [29]) report the precise measurement of many of these lines. Certain others, which could not be measured due to overlapping water lines, are not listed in table 12-B. Most of these lines are good to about $\pm 0.02\text{ cm}^{-1}$. The two bands are illustrated in figure 12-B as recorded with a nitrogen-purged small grating instrument with CO₂ in a cell. (The spectrogram was recorded by Dr. Norman Jones, National Research Council, Ottawa, Canada.)

5.15. C₂H₂, $\nu_1 + \nu_5$ Band

From the end of the *P* branch of the CO harmonic to the beginning of the μ_3 water vapor band there is a gap of about 100 cm^{-1} . There is a band of acetylene of medium intensity that falls in this region ($4,039$ to $4,130\text{ cm}^{-1}$). This band is overlapped by four weaker bands and when measured with low pressure and high resolution many lines are observed [30]. When a pressure of 1 atm. is used in a 10-cm cell, the smaller bands are not observed and the *P* and *R* branches of the band stand out clearly. A number of the strong lines were measured under both high and low pressures and the values agreed closely. The numbers listed in table 13 are the results of the low pressure measurements. The spectrum is shown in figure 13.

WAVE NUMBER, cm^{-1}

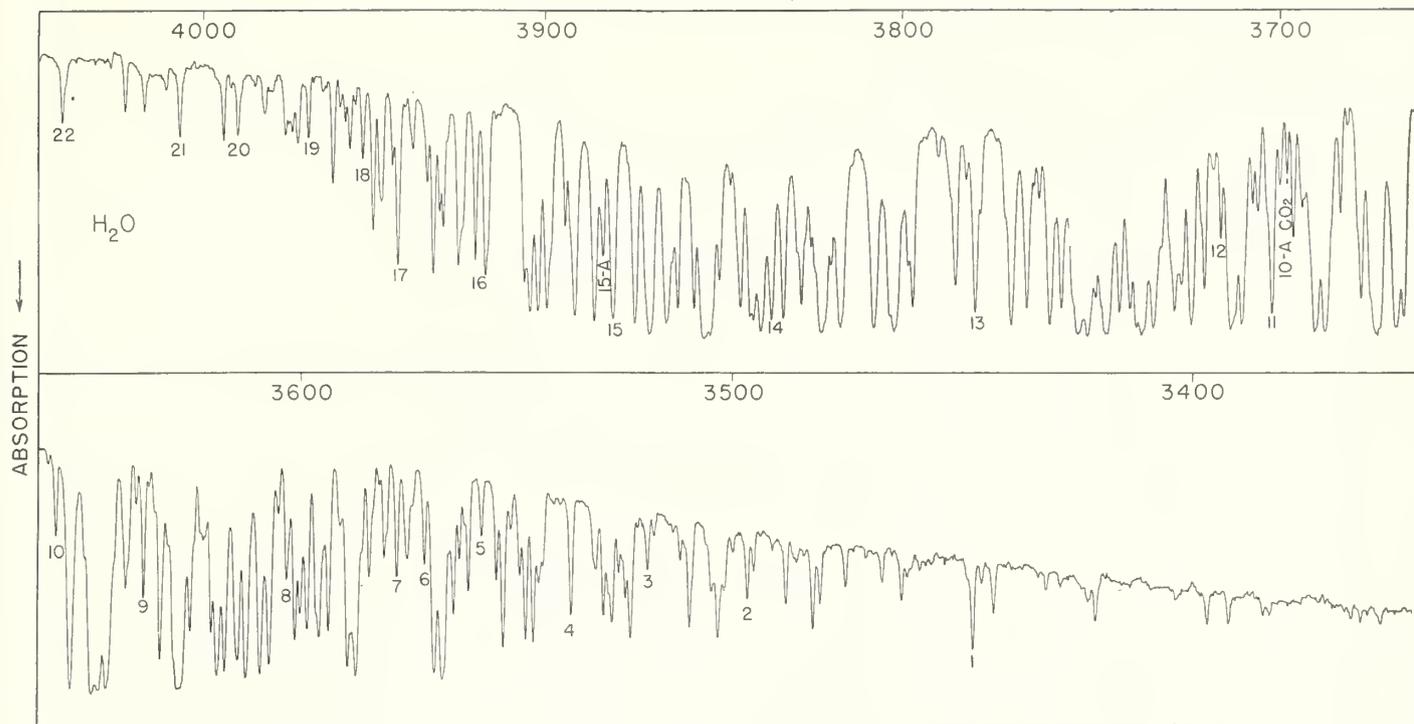


FIGURE 12-A. Atmospheric absorption bands of H_2O and CO_2 in the $3,700\text{-cm}^{-1}$ region recorded under intermediate resolution in the second order of a $5,000$ lines/in. grating.

The path was 7 m with 48 percent humidity at 23°C . Spectral slit width about 0.7 cm^{-1} .

TABLE 12-A. Absorption lines of water from $3,400$ to $4,000\text{ cm}^{-1}$

Line serial number	High resolution Plyler and Tidwell ^a $\nu\text{ cm}^{-1}$ (vac.) observed	Intermediate resolution this work $\nu\text{ cm}^{-1}$ (vac.) observed
1	3,397.20	3,447.21
2	3,447.20 (7.03)	96.62
3	96.63	3,519.05
4	3,518.97	36.44 avg
5	(2 components)	57.21
6	70.54	70.48
7	76.89	77.02 avg
	77.06 avg	
(8)	77.23	3,602.98 (complex)
9	3,603.08	38.15
10	59.94	59.94
10-A (CO_2)	98.00	98.08
11		3,701.90
12	3,714.81	3,714.82
13		79.38
14		3,835.06
15		80.52
15-A	3,883.26	83.32
16	3,920.10	3,920.28
17		42.86
18	53.11	53.22
19	69.15	69.16
20	90.72	90.74
21	4,008.59	4,008.59
22	44.90	

^a Plyler and Tidwell [29].

TABLE 12-B. Some precisely measured CO_2 lines of the 021 and 101 bands at $3,609$ and $3,716\text{ cm}^{-1}$

Line number	Plyler and Tidwell ^a $\nu\text{ cm}^{-1}$ (vac.) observed	Line number	Plyler and Tidwell ^a $\nu\text{ cm}^{-1}$ (vac.) observed
021 Band			
P 38	3,579.34	R 2	(3,615.25)
36	81.31	10	(21.17)
32	85.21	12	22.50
26	90.78	14	23.93
24	92.65	16	25.27
22	94.42	26	31.85
20	(96.22)	40	40.00
16	99.74		
14	3,601.40		
10	04.81		
8	06.48		
6	08.07		
101 Band			
P 36	3,682.78	R 6	3,720.09
32	86.74	10	22.90
26	92.46	12	24.35
24	94.34	14	(25.72)
20	98.00	18	28.42
18	99.79	20	29.73
14	3,703.29	22	31.01
12	05.02	26	33.48
10	06.70		
8	08.38		

^a Plyler and Tidwell [29].

Values in parentheses indicate lines which may be overlapped by water lines.

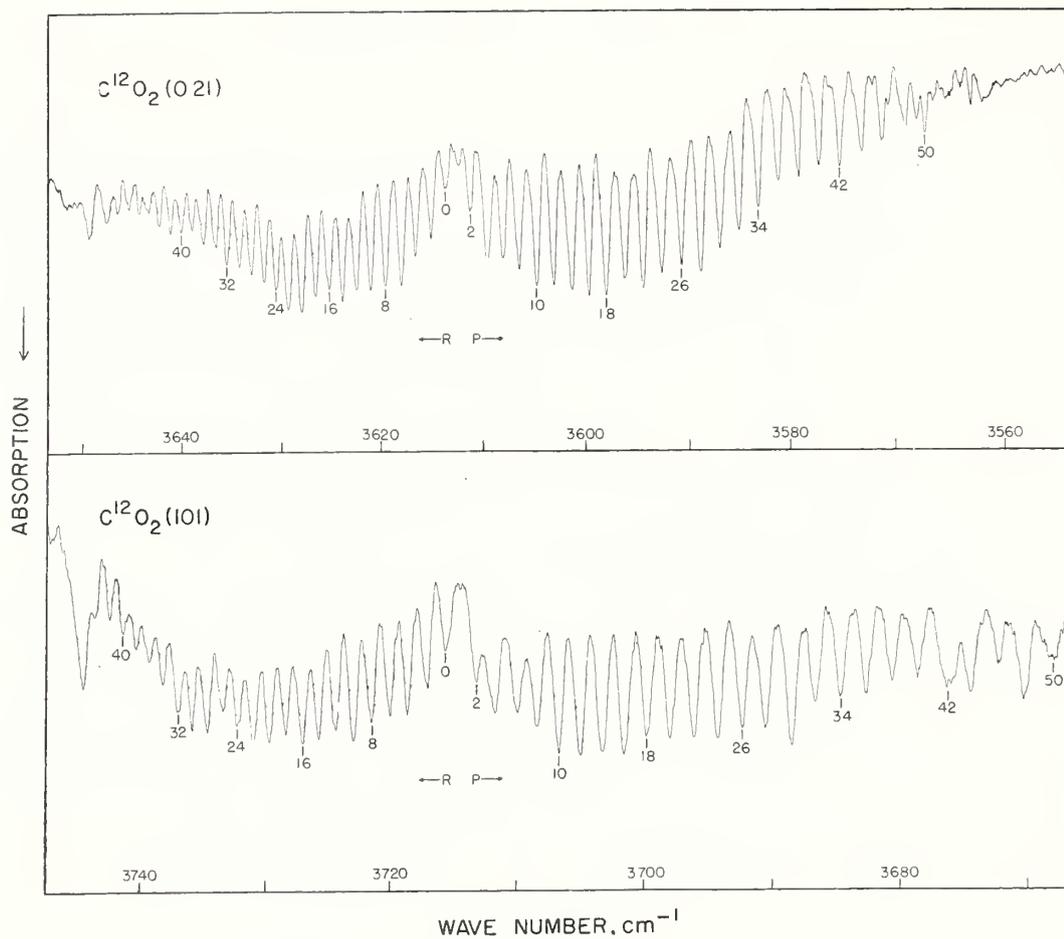


FIGURE 12-B. The 021 and 101 bands of CO_2 recorded with a purged small grating instrument. The CO_2 was contained in a 10-cm cell at 25-cm pressure for both bands. Spectral slit width about 0.30 cm^{-1} .

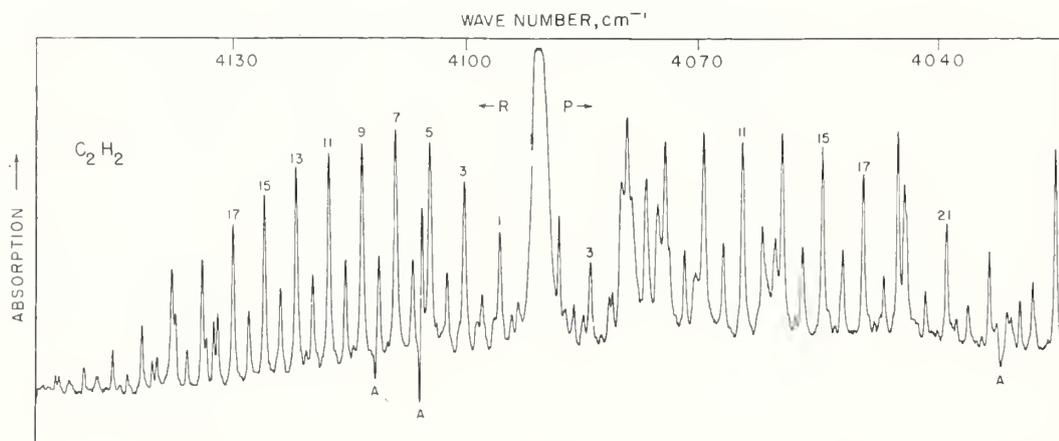


FIGURE 13. The $\nu_1 + \nu_5$ band of acetylene recorded with a 15,000 lines/in. grating singly passed with 1-atm pressure and 10-cm path. Spectral slit width about 0.08 cm^{-1} .

TABLE 13. Absorption lines of acetylene from 4,040 to 4,130 cm^{-1}

Line number	Previous work this laboratory ν cm^{-1} (vac.) observed	Line number	Previous work this laboratory ν cm^{-1} (vac.) observed
P 3	4,084.14	R 17	4,130.10
11	64.58	15	26.91
15	54.52	13	22.63
17	49.36	11	18.30
21	38.95	9	13.94
		7	09.47
		5	04.98
		3	4,100.47
		1	4,095.84

5.16. CO Harmonic Band

The CO harmonic band at 2.34μ has been measured very accurately by Rank and his colleagues (see ref. [14]) and also by Plyler, Allen, and Tidwell (see ref. [15]). This band is one of the few which have been measured in the infrared to such a precision that the reported frequencies are accurate to a few thousandths of a cm^{-1} . This spectrum was measured with a 60-cm cell and 20-cm pressure, but a 10-cm cell with 50-cm pressure is sufficient for observing forty lines of this band. The spectrum is shown in figure 14 and the frequencies are listed in table 14.

TABLE 14. Absorption lines of the 2-0 harmonic band of CO from 4,100 to 4,340 cm^{-1}

Line number	Plyler, Allen, and Tidwell ^a ν cm^{-1} (vac.) observed	Rank et al. ^b ν cm^{-1} (vac.) observed	Line number	Plyler, Allen, and Tidwell ^a ν cm^{-1} (vac.) observed	Rank et al. ^b ν cm^{-1} (vac.) observed
P 27	4,132.19	-----	R 0	4,263.842	.838
26	37.79	770	1	67.548	.542
25	43.33	-----	2	71.182	.179
24	48.817	-----	3	74.750	.743
23	54.234	-----	4	78.235	.234
			5	81.655	.661
22	59.564	-----	6	85.013	.010
21	64.852	-----	7	88.298	.290
20	70.066	.058	8	91.512	.500
19	75.212	.204	9	94.639	-----
18	80.286	.285			
			10	97.703	-----
17	85.302	.298	11	4,300.712	-----
16	90.234	.242	12	03.614	-----
15	95.120	-----	13	06.482	-----
14	99.931	-----	14	09.262	.257
13	4,204.673	-----			
			15	11.969	.962
12	09.356	-----	16	14.606	.597
11	13.962	-----	17	17.173	.161
10	18.504	-----	18	19.665	.650
9	22.974	954	19	22.085	.065
8	27.371	-----			
			20	24.425	-----
7	31.694	.685	21	26.699	-----
6	35.952	.949	22	28.878	-----
5	40.150	-----	23	31.022	-----
4	44.278	.267	24	33.057	.054
3	48.330	-----			
			25	-----	4,335.030
2	52.308	.307	26	-----	36.934
1	56.226	-----	27	-----	38.763
			28	-----	40.517
			29	-----	42.202

^a Plyler, Allen, and Tidwell [15].
^b Rank et al. [14].

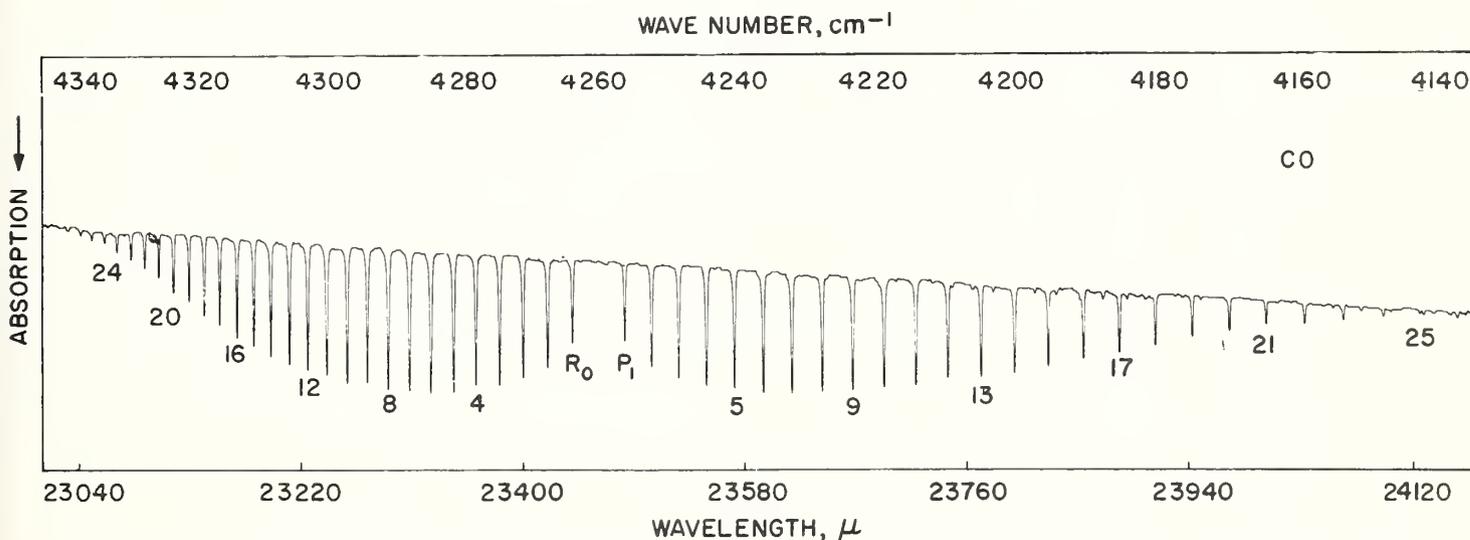


FIGURE 14. The 2-0 band of CO recorded with a 15,000 lines/in. grating with 20-cm pressure and 60-cm path. Spectral slit width about 0.15 cm^{-1} .

5.17. N₂O, 2 ν , Band and Hot Band

The $2\nu_1$ band of N₂O has been measured in the region from 2,520 to 2,580 cm^{-1} and the results are shown in figure 15 and table 15. Thompson and Williams [31] had previously measured this band and their values are used to compare with the results obtained in this work. The lines which have been measured are noted by a dot on the spectrogram.

There is an overlapping structure of weak lines and only those lines were measured which appeared in the open. This band can be resolved by a small grating instrument if a photoconducting cell is used as the detector and could be used as an alternate for HBr in this region. Also there are several regions where lines are closely grouped and the band can be used to check instrumental performance and resolution. The measurements of Thompson and Williams agree, on the average, to within $\pm 0.04 \text{ cm}^{-1}$ of the

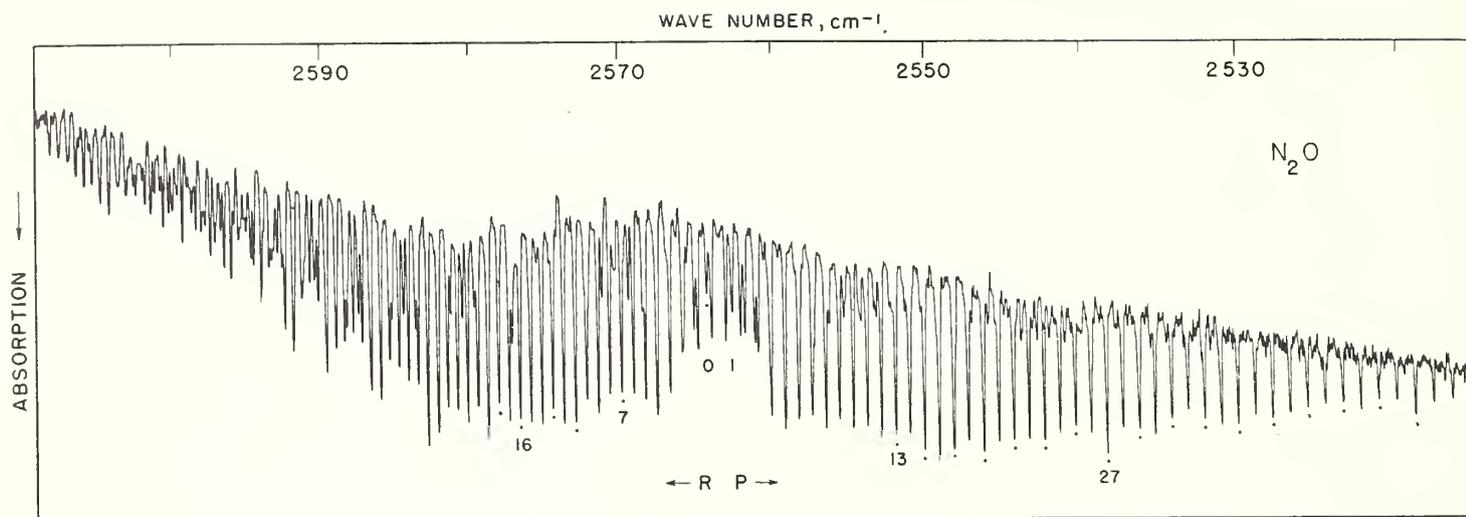


FIGURE 15. The $2\nu_1$ band (2564 cm^{-1}) of N_2O together with the hot band ($2\nu_1 + \nu_2 - \nu_2$, 2577 cm^{-1}) recorded with a 10,000 lines/in. grating with 2-mm pressure and 6-m path.

Spectral slit width about 0.08 cm^{-1} . The dots denote the measured and tabulated lines.

TABLE 15. Absorption lines of N_2O from $2,520$ to $2,580\text{ cm}^{-1}$

J	This work $\nu\text{ cm}^{-1}$ (vac.) observed	Thompson and Williams ^a $\nu\text{ cm}^{-1}$ (vac.) obs. laboratory
P 45	2,519.01	.05
43	21.27	.29
41	23.50	.52
39	25.71	.72
37	27.87	.88
35	30.03	.03
33	32.15	.17
31	34.25	.27
29	36.31	.35
27	38.37	.41
25	40.38	.42
23	42.38	.42
21	44.34	.38
19	46.27	.32
17	48.19	.23
15	50.07	.13
13	51.92	2.01 (1.97 calc.)
R 0	2,564.18	.24
7	69.82	.86
11	72.87	.94
13	74.38	.45
16	76.56	.59
18	77.99	8.04

^a Thompson and Williams [31].

values in this work. There is a small shift in the direction of lower frequency of all the values of the present measurements. An average of the two sets of values would probably be correct to $\pm 0.03\text{ cm}^{-1}$, which should be entirely adequate for calibrating medium resolution instruments.

5.18. NH_3 , ν_4 Fundamental

This band is being included as an alternate to the $6\text{-}\mu$ water band and should prove of some use to workers who wish to flush their instruments or use double-beam operation to study samples contained in cells. Dr. W. S. Benedict, of the Johns Hopkins

University, has picked out of the band (in accordance with the resolution illustrated in fig. 16) some 19 single or not-too-complex absorption peaks and has determined the best possible frequencies from the higher resolution results of Garing and Nielsen (see ref. [24]) and unpublished results from the National Bureau of Standards Laboratory. The results of these two works agree to within about $\pm 0.02\text{ cm}^{-1}$. The frequency assigned to certain absorption peaks which appear single in figure 16 but which actually consist of several components is, of course, no longer applicable if the peak is broken up into its components under slightly higher resolution. (The letter S in figure 16 indicates a single line.) No table accompanies this spectrogram but instead the acceptable values which are probably good to $\pm 0.02\text{ cm}^{-1}$ for single lines and off by as much as $\pm 0.2\text{ cm}^{-1}$ for some complex lines, have been printed on the illustration. For calibration purposes, the μ_2 water lines (see table 5) are considerably better than the lines of this band. (The spectrogram was recorded by Dr. Norman Jones, National Research Council, Ottawa, Canada.)

6. Polystyrene Film

In 1950 Plyler and Peters (see ref. [7]) measured the infrared absorption spectrum of polystyrene on a grating instrument and certain bands were suggested for use in calibrating prism instruments. Since that time, films of polystyrene have been widely used in many laboratories and it has been found very useful in checking the calibration of an instrument in certain regions to ascertain if there are any changes in the reading of the wavelength or frequency scale of the instrument. There is a question as to the suitability of polystyrene for a calibrating material as it is not completely stable and has a different absorption spectrum when it

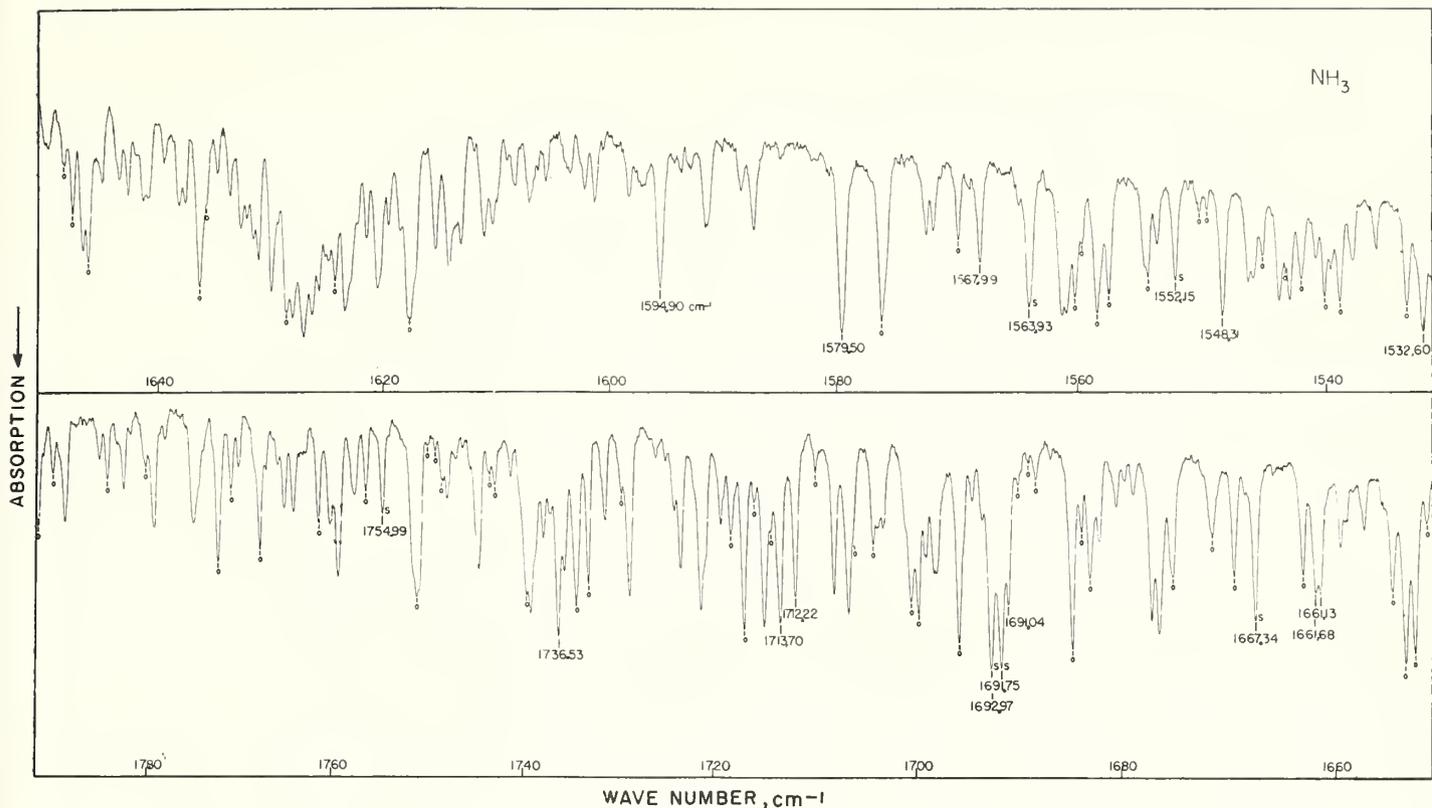


FIGURE 16. The ν_3 band of NH_3 at 1628 cm^{-1} recorded with a nitrogen-purged small grating instrument. Certain strong lines of the ν_2 water band still appear. A gas pressure of 7.5 cm in a 10-cm cell was used. Spectral slit width about 0.50 cm^{-1} .

ages. Also different batches of the material may show differences in absorption. The variation in the absorption spectrum of different films can easily be demonstrated, but the strong bands are not appreciably changed in wavelength by age or the origin of the film. In this work, we checked a film which had been made ten years ago and it showed no appreciable shifts in the absorption bands. Eight bands from 6 to $14.4\ \mu$ have been remeasured and the results are compared with the previous measurements of Plyler, Blaine, and Nowak (see ref. [11]) in 1957. The spectrum is shown in figure 17 and the wavelengths and wave numbers in vacuum are listed in table 16. When the faces of the film are parallel, interference fringes appear but these usually occur

TABLE 16. *Polystyrene absorption bands*

Previous measurement wavelength (air) μ	New measurement this work wavelength (air)	This work cm^{-1} (vac.)
3.3026	-----	-----
3.422	-----	-----
3.507	-----	-----
5.138	-----	-----
5.343	-----	-----
5.549	-----	-----
6.238	6.2427	1,601.45
-----	6.3151	1,583.08
6.692	-----	-----
-----	8.4615	1,181.50
8.662	8.6626	1,154.08
-----	9.3524	1,068.96
9.724	9.7226	1,028.25
11.035	11.028	906.50
-----	14.316	698.33

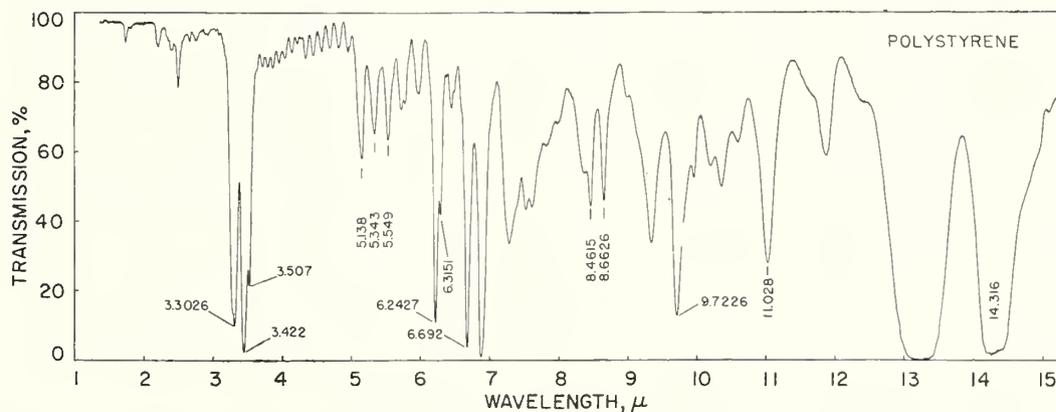


FIGURE 17. Infrared absorption spectrum, from 2 to $15\ \mu$, of a $50\text{-}\mu$ film of polystyrene recorded with a rock-salt prism instrument.

between 4 and 6 μ for a 50- μ film and do not change the position of the bands beyond 6 μ . The small band at 6.3151 μ , which is on the long wavelength side of the strong band at 6.24 μ , is completely resolved by a low resolution grating instrument and is not overlapped by atmospheric water lines. It should be useful for calibration in this region. The strong band at 14.316 μ was measured with a film of 8- μ thickness and with this film the maximum of absorption could be accurately determined. It is estimated that the bands listed in table 16 are accurate to $\pm 0.3\text{cm}^{-1}$ and should be useful in calibrating low resolution instruments, but should not be considered as being of the same order of accuracy as the molecular vapor bands. In measuring the polystyrene bands, the center was determined near the $\frac{3}{4}$ -absorption point for symmetrical bands and reference should be made to this point when making use of the numbers in table 16.

7. Summary

The rather "popular" ammonia band at 3 μ was not used in this work because of its resolution sensitiveness and also because of temperature and pressure effects. For example, there are 12 components in the P 8 "line" extending over 1.5 cm^{-1} . Under low resolution (see ref. [11]) the Q branch, which extends over 5.5 cm^{-1} and consists of hundreds of lines is probably good to 0.5 cm^{-1} .

The 16- μ coverage may be appropriately extended by making use of frequency values reported by Lakshmi, Rao, and Nielsen [32] for N_2O . In this work, we have checked Rao's measurements on the 15- μ CO_2 band and feel that if the same techniques were used on the N_2O measurements, then these numbers may be used. Considering the lower resolution used by the Japanese workers (see ref. [12]) on this band, the agreement with values reported in [32] above is quite good.

At the high frequency end, we have already given reference to the work of Rank and coworkers on HCN (see ref. [16]) and wish to include also the grating work on H_2O vapor at 1.9 μ and on CH_4 at 2.2 μ as given in reference [8].

The calibrating values given in this report should make it easier for infrared spectroscopists to obtain accurate measurements in the region from 2 to 16 μ . As further measurements are carried out in other laboratories; there will be more values for inter-comparison and it should be possible to obtain calculated values for certain bands which would be more accurate than the observed values from any one laboratory. No recommended frequencies representing properly weighted averages of the work of several laboratories have been given, but this may be done at a later date when more data are available. It is hoped that spectroscopists will send new data of this type to the authors for wavelength standards so that it may be incorporated in a future report. It is also requested that any corrections or suggested changes be communicated to the authors.

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Wavelengths From Thorium-Halide Lamps

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The present system of international secondary standards of wavelength for spectroscopic measurements is based on interferometric determinations of wavelengths emitted at atmospheric pressure by an electric arc between iron electrodes. Because of the poor quality and uneven distribution of these iron standards they are not suitable for accurate measurement of wavelengths in the spectra of heavier elements, most of which are more complex and consist of much sharper lines than the standards. Quartz-tube lamps containing a small quantity of a thorium halide, when excited by microwaves, emit thousands of uniformly sharp and evenly distributed lines whose wavelengths, or positions in a spectrum, can be determined with about one-tenth the error of locating iron-arc lines. Preliminary values of 222 vacuum wavelengths emitted by a thorium-iodide lamp have been measured relative to 5462.2705 and 4047.7144 Å emitted by a similar lamp containing mercury-198. Fabry-Perot interferometers with plate separations of 25, 40, or 50 millimeters were used with a stigmatic grating spectrograph in making these measurements. The thorium wavelengths range from 3288.7356 to 6991.5839 Å in vacuum and from 3287.7885 to 6989.6562 Å in standard air. The accuracy in relative value of 27 classified thorium lines is tested by means of the combination principle, which indicates that the average error is less than 1 part in 20 million.

1. Introduction

During the past half-century the wavelengths of hundreds of radiations from the iron arc at atmospheric pressure have been measured with interferometers to serve as international standards of wavelength for spectroscopic measurements. The history and results of this activity were summarized in *Wavelengths From Iron-Halide Lamps* [1]¹, in which it was shown that the accuracy of measurement could be increased by a factor of 2 or 3 if the iron lines were emitted by simple lamps containing a trace of iron halide excited, at low pressure and moderate temperature, by microwaves. But the iron arc in air is still the main source of internationally adopted standard wavelengths.

The actual application of the adopted iron standards to wavelength measurements in various spectra is handicapped by two serious defects in the iron-arc spectrum. First, because of the high temperature (6,300° K) of the iron arc and the relatively small atomic number ($Z=26$) and mass ($A=56$) of the iron atom its spectral lines are excessively broadened on account of Doppler-Fizeau effect. Moreover, an electric arc between iron electrodes at atmospheric pressure produces lines of various kinds: strongly self-reversed lines involving low energy levels, diffuse lines involving high energy levels, and unstable lines of variable wavelength due to "pole effect." Second, the distribution of strong or similar lines in the arc spectrum of iron is such that no uniform spacing of wavelength standards is possible, and unduly large gaps between usable standards are unavoidable in certain spectral regions. Consequently many spectroscopists have expressed dissatisfaction with the international standards of wavelength emitted by the iron arc. Thus, Harrison [2] states that the wavelength precision of measurements in the MIT Wavelength Tables has been limited primarily by

"the insufficiency of adequate wavelength standards in some spectral regions, particularly of standard lines of suitable intensities." Likewise, Bovey [3] in giving a preliminary list of 6-figure wavelengths of plutonium excited in a furnace complains that "A serious difficulty for accurate measurement has been the width of the iron-arc lines, many of which are several times wider than the emission lines from a furnace." Also Fred and Tomkins [4] in describing the first two spectra of americium to 7 figures, announce that "Most of the uncertainty in the wavelengths is due to the poor quality of the standards, since the agreement in the lfs intervals shows that the line positions can be measured to another decimal place."

Logically the iron-arc standards should have been used only for measuring atmospheric arc and spark spectra of metals lighter than iron, because, other things equal, the heavier atoms emit sharper lines which cannot be measured accurately relative to standards that are broader and badly spaced. When the arc spectrum of cerium ($Z=58$, $A=140$) is photographed beside that of the standard iron arc it is readily seen that all cerium lines are narrower than iron lines, and a comparison with thorium ($Z=90$, $A=232$) or with uranium ($Z=92$, $A=238$) is even more embarrassing for iron. This was observed by Harrison [5], who remarked that "In the extensive series of wavelength measurements made with the automatic comparator, which resulted in the publication of the *Massachusetts Institute of Technology Wavelength Tables*, the most serious limitation on precision was found to arise from the lack of satisfactory standard lines. Measurements on lines in such complex spectra as cerium and thorium, for example, showed much better internal consistency than did those on the iron standards themselves." To assist in remedying this situation, Harrison [5] cleverly contrived a complicated machine for the rapid direct determination of wavelengths from Fabry-Perot interferometer patterns. He called this machine the WINMAC

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¹ Figures in brackets indicate the literature references at the end of this paper.

(Wavelength interferometric measurement and computation), and in 1950 he reported [6] that "the WINMAC is now being readied for what is hoped will be an extensive series of wavelength determinations of intermediate wavelength standards in cerium, thorium, and other elements." Unfortunately this hope did not materialize. However, this is not seriously regretted now because in the meantime great improvements over the traditional arcs and sparks at atmospheric pressure have been made in spectroscopic light sources.

The most simple, convenient, efficient, and economical type of spectroscopic light source is an evacuated tube of fused quartz enclosing a trace of gas, metal vapor, or volatile metallic compound excited by microwaves generated by vacuum-tube oscillators or by magnetrons. The prototype was the low pressure, water-cooled Hg^{198} lamp described by Meggers and Westfall [7], who also discovered that "clean-up" in such discharges varied inversely with frequency. In most laboratories magnetrons producing 2,450 Mc, or more, soon replaced vacuum-tube oscillators producing 100 to 300 Mc for the excitation of these lamps. The preparation of similar lamps with other volatile metals was described by Zelikoff, Wyckoff, Aschenbrand, and Loomis [8], while Corliss, Bozman, and Westfall [9] demonstrated that such lamps can be made to emit the spectra of all metals that form volatile compounds. A precise procedure for the preparation of electrodeless discharge tubes containing very pure rare earths and highly radioactive elements has been described by Tomkins and Fred [10]. Compared with conventional arcs and sparks, these lamps require a minimum amount of sample, which is enclosed and conserved. Also, they operate at relatively low temperature and pressure, and consequently emit spectral lines of greatly reduced width usually free of self-reversal. When operated with continuous wave power and moderate gas or vapor pressure, these lamps strongly favor the spectra of neutral atoms, but pulsed discharges and/or reduced pressures enhance the second and third spectra. Lamps viewed end-on when excited by a 100-watt magnetron emit spectra comparable in intensity to a 10-ampere 220-volt d-c arc, but with line widths much less than half as great. Bright, sharp lines are especially needed for the resolution of complex Zeeman patterns, or for the measurement of isotopic-spectroscopic effects. The development of these electrodeless lamps has thus greatly increased the range and precision of spectroscopic observations.

Now the accurate description and quantum interpretation of the spectra of rare-earth types of atoms and ions is still in a very unsatisfactory state, because of their extreme complexity and because pure samples have not been available in the past. The latter difficulty has recently been overcome either by efficient separation or by artificial preparation, but extreme complexity of the spectra remains as an obstacle to successful analysis. It is primarily for this reason that wavelengths must be measured with greater accuracy, and for this purpose reference standards of greater precision and better distribution than those from an iron arc are absolutely necessary.

Fortunately the simple electrodeless lamps referred to provide the means for obtaining a greatly improved system of standard wavelengths [11]. Lamps containing a trace of a thorium halide excited by microwaves emit approximately 20,000 lines of uniformly sharp character and even distribution between 2000 and 12000 Å. Since these lines are characteristic of heavy atoms and ions of even mass ($Z=90$, $A=232$) they have relatively small Doppler-Fizeau widths, and are entirely free from hyperfine structure or isotope shifts. By imaging a thorium-halide lamp inside a similar one containing Hg^{198} (or vice versa) both sources can be caused simultaneously to illuminate a Fabry-Perot interferometer and stigmatic spectrograph. From the interference patterns the wavelengths of thorium lines can then be determined to 8 figures relative to the previously determined values of the mercury lines. The purpose of this paper is to present preliminary results of such wavelength measurements for 222 radiations from thorium, covering slightly more than one octave of spectrum.

2. Light Sources

The thorium source used in this investigation was an electrodeless discharge tube containing a few milligrams of thorium iodide and pure helium gas at a pressure of 5 mm of Hg or less. The tube was prepared by C. H. Corliss in a manner previously described [9]. The tube blank is made of Vycor glass; it has a bore of approximately 5 mm and a length of 11.3 cm. The discharge was excited at a frequency of 2,450 Mc obtained from a commercial diathermy generator.

The thorium tube used throughout the investigation was chosen from among several that were made at the same time. After a warmup period of 30 to 60 sec, the tube would glow very brightly for approximately 3 min. At the end of this time the thorium spectrum would weaken, and the discharge would be confined to a narrow filament near the center of the tube, indicating that the pressure in the tube had increased. The period of high intensity mentioned above was accompanied by a gradual movement of the thorium iodide deposit from the top to the bottom of the tube. As soon as the thorium spectrum began to weaken, the discharge tube was removed from its holder, cooled in water, and replaced in an inverted position with the thorium iodide deposit again at the top of the tube. For many of the photographs, this 3-min running time was sufficient. For longer exposures it was necessary to cool and invert the tube several times. This change could be made within 30 sec.

The primary standard on which the thorium measurements were based was a NBS-Meggers Hg^{198} lamp [7], that contained 1 mg of Hg^{198} and argon at a pressure of approximately 3 mm of Hg. The mercury tube was excited by a second diathermy generator at the same frequency of 2,450 Mc. The Hg^{198} source was cooled by an air stream from a small blower.

3. Experimental Method

The optical arrangement was the same as that used by the authors in the previous investigation of iron wavelengths from an electrodeless tube [1]. For this reason, only a brief description will be given here. The Fabry-Perot interferometer consisted of aluminized quartz plates separated by 3 sets of invar pins either 25, 40, or 50 mm in length. Good interference fringes were also obtained with plate separation of 67.5 mm, but lack of time did not permit the exploitation of the higher orders of interference.

The evacuated ($<10^{-4}$ mm Hg) interferometer was mounted externally in parallel light. Both thorium and Hg^{198} lamps were placed on the optical axis but at right angles to it and parallel to the spectrograph slit, so that both sources were observed side-on. The Hg^{198} lamp was farther from the slit so that it could be imaged inside the thorium tube. From this point on, light from both lamps traveled the same path through condensing lens, interferometer, projection lens, and thence through the slit and spectrograph. The quartz-fluorite achromatic projection lens had the proper focal length (50 cm or 100 cm) to form six or more circular fringes on the spectrograph slit, which was 25 mm long and either 0.15 or 0.30 mm wide. Accuracy of measurement was thus favored by the large scale of the interference patterns and by a slit width that made the fringes approximate rectangles.

Dispersion in the horizontal direction was produced by a 15,000-line-per-inch concave grating in the Wadsworth mounting. The curvature of the plate holder was decreased so as to favor the horizontal focus and thus produce sharp images of the Fabry-Perot fringes from both lamps, simultaneously over as large a range as possible. This instrument was used to photograph diametral sections of interference patterns for waves between 4400 and 7000 Å, with a slit width of 0.30 mm and a reciprocal dispersion of 5 Å/mm in the first-order grating spectrum. Because of the higher density of lines in the ultraviolet spectrum of thorium, the region from 3275 to 4500 Å was photographed in the second-order grating spectrum with a reciprocal dispersion of 2.45 Å/mm and slit width of 0.15 mm. Under these conditions most of the measured interference patterns were free from overlaps of neighboring lines, but if many more thorium wavelengths are to be measured larger grating dispersion or smaller slit width will be necessary. In each wavelength range several spectrograms were made with different exposure times, and with slight mechanical-pressure changes in the interferometer spacing to alter the interference configurations or fractional orders for all spectral lines.

The interferometer plates were the same ones used previously for measurements of iron and mercury wavelengths [1, 12, 13]. The last series of measurements [13] suggested that the dispersion of phase change in the aluminum films was very small between 3300 and 6000 Å, so no corrections on account of phase change were applied to these thorium wave-

lengths. No significant differences were found between results from the 25-, 40-, and 50-mm etalons.

All mercury lines that appeared with sufficient intensity were measured along with the thorium lines. The green (5462.2705 Å) and violet (4047.7144 Å) lines of Hg^{198} were chosen as standards respectively for the long- and short-wave interference spectrograms described above. The values of other Hg^{198} lines measured on these spectrograms agreed quite well with unpublished wavelengths [13], the greatest difference being 0.0002 Å. This supports our assumption that any possible corrections to these thorium preliminary wavelengths on account of dispersion of phase-change in the interferometer films are negligible.

The interferograms were measured at the National Bureau of Standards with the interference comparator designed by K. Burus [14], and at Purdue University with a comparator made by Carl Leiss. In order to measure as many thorium wavelengths as possible within a given time, it was found desirable to measure just 5 diameters of each interference pattern. As mentioned before [7], by measuring the diameters of 5 interference rings, the least-squares evaluation of the fractional order at the center may be made with a minimum of arithmetical computation because some of the steps are easily performed mentally. Even with this conservation of time and labor the thorium wavelengths presented in this paper required the bisection of more than 12,000 interference fringes and calculations for more than 1,200 individual patterns.

Another way to reduce the time and labor involved in interferometric comparisons of wavelengths is to begin with the largest orders of interference consistent with finding the correct integral order for each line. If grating measurements of wavelength are to be refined and replaced by interferometric values, the initial orders of interference should not be so great that errors in the grating values lead to wrong integral orders. For example, if a thorium wavelength near 5000 Å is to be measured relative to 5462.2705 Å of Hg^{198} , the whole order of the thorium wavelength with a 25-mm etalon will be about 100,000, and the error in the grating value must be less than ± 0.02 Å if it is to yield the correct integral order. We assumed that the available grating values of thorium wavelengths justified beginning the interferometric observations with a 25-mm etalon and then testing these values with a 40-mm etalon. If the integral orders of interference are in error because of too large errors in the grating values, the 25- and 40-mm etalon values will differ radically, but if the integral orders are correct, the results will agree within a very small possible error of observation. This bold procedure saved much time and labor by eliminating interferometric observations with all smaller etalons which do not contribute anything to the accuracy attainable with the larger etalons.

Two independent sets of grating values of thorium wavelengths were available, one in the *MIT Wavelength Tables* [2] representing wavelengths emitted by a thorium arc, and the second representing unpublished wavelengths from thorium halide lamps measured by R. Zalubas in the National Bureau of

TABLE 1. Thorium wavelengths: grating and interferometer values

	λ_{air}	λ_{vac}	λ_{vac} (25 mm)	Change in order	λ_{vac} (40 mm)
MIT.....	4165. 813	4166. 987	4166. 9750	----	4166. 9832
NBS.....	4165. 782	4166. 956	4166. 9403	+1	4166. 9403
MIT.....	3869. 639	3870. 736	3870. 7308	----	3870. 7416
NBS.....	3869. 665	3870. 762	3870. 7606	-1	3870. 7604
MIT.....	3854. 547	3855. 640	3855. 6333	----	3855. 6222
NBS.....	3854. 512	3855. 605	3855. 6036	+1	3855. 6035
MIT.....	3421. 189	3422. 170	3422. 1673	----	3422. 1618
NBS.....	3421. 215	3422. 196	3422. 1908	-1	3422. 1910

Standards. Both sets were referred to the adopted secondary standards of wavelength obtained from the iron arc at atmospheric pressure. We gave preference to the MIT grating values because these were objectively measured and calculated by automatic machines [2], and therefore entirely free from human errors. This was a mistake; the only errors in integral orders of 25-mm etalons occurred because of errors larger than 0.02 Å in the MIT grating values as shown in table 1. The first line shows in succession the MIT wavelength in air, its value in vacuum [15], and discordant results from 25-mm etalons and from 40-mm etalons. The second line gives similar data for NBS grating wavelengths, but the results from 25- and 40-mm etalons are in perfect agreement because the integral orders were corrected as indicated by the NBS grating values. The interferometric values for these four lines show that the MIT grating values have errors of +0.047, -0.025, +0.036, and -0.021 Å, respectively, whereas the NBS errors are +0.016, +0.001, +0.001, and +0.005 Å. No explanation of the large and erratic errors in the MIT grating values is offered, but this experience should be a warning that some 7-figure MIT wavelengths may not be trustworthy beyond 5 figures.

4. Results

The thorium wavelengths obtained as a result of this investigation are presented in table 2. Since the interferometer was evacuated and the separation of the interferometer plates was always expressed in terms of vacuum wavelengths of Hg¹⁹⁸ standards, all the measured wavelengths of thorium are valid only in vacuum. Column 1 displays only that part of the vacuum wavelength that can be expressed in whole angstroms, the fraction that must be added to this is given in columns 2, 3, and 4, depending on whether it was measured respectively with 25-, 40-, or 50-mm etalons. A figure in parentheses following each fraction shows the number of individual determinations. Column 5 contains the average preliminary vacuum wavelength resulting from all the observations. In column 6 the vacuum wavelengths of column 5 are changed to values in standard air with the aid of Edlén's conversion table [15]. Column 7 quotes unpublished estimated relative intensities of thorium lines as given by Zalubas, and

column 8 indicates the particular spectrum to which each wavelength belongs.

It is a common characteristic of all complex spectra that neutral atoms dominate the visible, and singly-ionized atoms predominate in the ultraviolet. In these preliminary measurements of thorium wavelengths with Fabry-Perot etalons no difference in character could be observed between the Th I and Th II lines, and there is no reason to suspect that one category is more accurately measured than the other. Unfortunately none of the thorium wavelengths has been measured a sufficient number of times with any given etalon to compute a meaningful probable error or standard deviation. A considerable number of the wavelengths from a given etalon are limited to a single observation and a comparison of these with the over-all averages may give an impression of the uncertainty in individual observations.

Because of the lack of sufficient observational material to calculate probable errors of the thorium wavelengths, an attempt was made to test their relative values by means of the combination principle. Although the Th II spectrum has been extensively analyzed [16] no recurring wavenumber differences were found between Th II lines in table 2. Somewhat better fortune was encountered with Th I lines. Even though the published analysis of Th I is extremely fragmentary [17] it is now being extended by Zalubas [18] who has reported that 27 of our interferometric values of Th I lines test the constancy of 4 wavenumber differences, viz., 2869.261, 3687.990, 5563.143, and 6362.396 K (=cm⁻¹) between low-energy levels established for neutral thorium atoms. These are presented in table 3 as 14 pairs of vacuum wavelengths in Å, and their reciprocals, or vacuum wavenumbers in K. The mean deviation of the 14 differences from their arithmetical averages is 0.001₂ K, that is 1 part in about 20 million. The pairs numbered 2 and 3 account for two-thirds of the summed deviations; if these are ignored the remaining 12 exhibit an average departure of only 0.0005 K, or 1 part in about 40 million, but this may be discounted as accidental.

The 222 wavelengths (3288.7356 to 6991.5839 Å) of thorium radiations in table 2 are presented as a preliminary set of improved standard wavelengths for the measurement of highly-dispersed complex spectra. The average interval between these stand-

TABLE 2. Wavelengths in thorium spectra

1	2	3	4	5	6	7	8
Whole angstrom	Fractional angstrom			Vacuum	Air	Relative intensity	Spectrum
	25 mm(obs.)	40 mm(obs.)	50 mm(obs.)				
6991	5840 (2)	5839 (3)	5839 (1)	6991. 5839	6989. 6562	900	I
6945	5265 (3)	5266 (3)	5263 (1)	6945. 5265	6943. 6112	600	I
6913	1334 (3)	1338 (3)	1333 (1)	6913. 1336	6911. 2264	400	I
6836	8110 (4)	8114 (2)	8108 (1)	6836. 8110	6834. 9249	75	I
6830	9196 (2)	9205 (1)	9201 (1)	6830. 9200	6829. 0355	150	I
6758	3179 (3)	3178 (2)	3176 (1)	6758. 3178	6756. 4528	250	I
6729	3153 (2)	3161 (1)	3162 (1)	6729. 3157	6727. 4585	200	I
6680	-----	5511 (1)	5522 (1)	6680. 5516	6678. 7076	30	I
6664	1088 (4)	1089 (3)	1094 (1)	6664. 1090	6662. 2694	250	I
6660	-----	5161 (1)	5161 (1)	6660. 5161	6658. 6774	50	I
6595	7610 (4)	7610 (3)	7608 (1)	6595. 7610	6593. 9397	200	I
6593	-----	3059 (1)	3051 (1)	6593. 3055	6591. 4849	100	I
6590	3595 (4)	3600 (2)	3596 (1)	6590. 3596	6588. 5398	200	I
6585	7248 (4)	7254 (3)	7251 (1)	6585. 7251	6583. 9065	200	I
6555	9715 (2)	9710 (4)	9707 (1)	6555. 9711	6554. 1605	100	I
6533	1468 (5)	1465 (4)	1466 (1)	6533. 1467	6531. 3423	400	I
6492	5317 (2)	5312 (4)	5313 (1)	6492. 5313	6490. 7378	120	I
6459	0675 (4)	0678 (3)	0680 (1)	6459. 0677	6457. 2834	500	I
6415	-----	3881 (3)	3876 (1)	6415. 3880	6413. 6152	200	I
6413	6717 (4)	6721 (4)	6719 (1)	6413. 6719	6411. 8996	250	I
6378	6939 (3)	6940 (4)	6933 (1)	6378. 6939	6376. 9310	350	I
6344	6134 (4)	6143 (4)	6135 (1)	6344. 6138	6342. 8600	300	I
6329	0286 (2)	0285 (4)	0270 (1)	6329. 0284	6327. 2788	180	I
6263	1499 (4)	1493 (4)	1504 (1)	6263. 1496	6261. 4177	180	I
6259	1546 (3)	1546 (3)	1548 (1)	6259. 1546	6257. 4237	100	I
6226	2497 (2)	2494 (4)	2494 (1)	6226. 2495	6224. 5275	100	I
6208	9379 (5)	9380 (4)	9378 (1)	6208. 9379	6207. 2205	160	I
6193	-----	6188 (1)	6186 (1)	6193. 6187	6191. 9054	100	I
6184	3324 (4)	3331 (4)	3326 (1)	6184. 3327	6182. 6219	400	I
6153	6954 (5)	6962 (3)	6959 (1)	6153. 6958	6151. 9932	120	I
6104	2838 (2)	2837 (2)	2849 (1)	6104. 2839	6102. 5946	90	I
6089	7161 (2)	7159 (2)	7160 (1)	6089. 7160	6088. 0306	125	I
6087	0592 (5)	0596 (2)	0586 (1)	6087. 0592	6085. 3745	100	I
6050	7258 (2)	7259 (2)	7259 (1)	6050. 7259	6049. 0510	100	I
6039	3696 (5)	3700 (4)	3696 (1)	6039. 3697	6037. 6978	140	I
6022	7042 (1)	7038 (2)	-----	6022. 7040	6021. 0362	140	I
6008	7362 (4)	7361 (4)	7364 (1)	6008. 7362	6007. 0725	180	I
5976	7202 (2)	7208 (4)	7213 (1)	5976. 7207	5975. 0656	250	I
5975	3202 (5)	3196 (4)	3193 (1)	5975. 3199	5973. 6651	250	I
5940	4710 (4)	4708 (4)	4708 (1)	5940. 4709	5938. 8255	140	I
5887	3325 (1)	3331 (1)	3329 (1)	5887. 3329	5885. 7017	120	I
5854	3043 (4)	3033 (2)	3053 (1)	5854. 3040	5852. 6817	200	I
5805	7508 (5)	7507 (4)	7509 (1)	5805. 7508	5804. 1414	300	I
5792	2686 (5)	2686 (3)	2683 (1)	5792. 2686	5790. 6629	-----	Hg ¹⁹⁸
5791	2494 (3)	2495 (2)	2492 (1)	5791. 2494	5789. 6439	200	I
5771	1983 (5)	1983 (4)	1980 (1)	5771. 1983	5769. 5982	-----	Hg ¹⁹⁸
5762	1484 (4)	1489 (4)	1489 (1)	5762. 1487	5760. 5510	600	I
5726	9766 (1)	9771 (3)	-----	5726. 9770	5725. 3887	250	I
5708	6864 (4)	6870 (4)	6872 (1)	5708. 6867	5707. 1033	200	II
5659	-----	4963 (1)	4958 (1)	5659. 4960	5657. 9258	100	I
5641	3109 (3)	3122 (3)	3120 (1)	5641. 3115	5639. 7461	250	II
5616	8789 (2)	8790 (4)	-----	5616. 8790	5615. 3202	350	I
5588	5775 (5)	5780 (5)	5781 (1)	5588. 5778	5587. 0265	500	I
5580	9073 (3)	9080 (4)	9080 (1)	5580. 9077	5579. 3585	300	I
5574	9014 (4)	9015 (4)	9010 (1)	5574. 9014	5573. 3538	350	I
5559	8862 (3)	8862 (4)	8859 (1)	5559. 8862	5558. 3426	400	I
5549	7171 (4)	7169 (3)	7172 (1)	5549. 7170	5548. 1761	300	I
5540	8001 (5)	8000 (5)	7999 (1)	5540. 8000	5539. 2615	400	I
5511	5243 (4)	5247 (4)	5238 (1)	5511. 5244	5509. 9937	300	I
5500	7828 (5)	7831 (5)	7831 (1)	5500. 7830	5499. 2552	250	I

TABLE 2. Wavelengths in thorium spectra—Continued

1	2	3	4	5	6	7	8
Whole angstrom	Fractional angstrom			Vacuum	Air	Relative intensity	Spectrum
	25 mm(obs.)	40 mm(obs.)	50 mm(obs.)				
5462	2705 (5)	2705 (5)	2705 (1)	5462. 2705	5460. 7530	-----	Hg ¹⁹⁸
5453	7338 (1)	7343 (2)	-----	5453. 7341	5452. 2188	250	I
5432	6213 (4)	6211 (2)	6216 (1)	5432. 6212	5431. 1116	300	I
5427	1859 (2)	1865 (3)	-----	5427. 1863	5425. 6781	250	II
5418	9915 (5)	9916 (5)	9916 (1)	5418. 9916	5417. 4856	200	I
5409	1568 (4)	1570 (3)	1573 (1)	5409. 1569	5407. 6535	260	I
5388	1093 (1)	1083 (1)	1087 (1)	5388. 1087	5386. 6109	300	I
5345	0672 (3)	0678 (3)	0681 (1)	5345. 0676	5343. 5813	500	I
5328	4573 (3)	4575 (3)	4572 (1)	5328. 4574	5326. 9755	400	I
5278	9691 (2)	9687 (2)	-----	5278. 9689	5277. 5002	400	II
5259	8245 (3)	8245 (3)	-----	5259. 8245	5258. 3609	300	I
5232	6158 (3)	6160 (3)	-----	5232. 6159	5231. 1596	900	I
5178	4028 (3)	4023 (3)	-----	5178. 4025	5176. 9606	400	I
5160	0408 (3)	0413 (3)	-----	5160. 0411	5158. 6041	700	I
5155	6781 (1)	6789 (3)	-----	5155. 6787	5154. 2429	400	I
5116	4694 (2)	4700 (2)	-----	5116. 4697	5115. 0443	250	I
5069	3865 (2)	3870 (2)	-----	5069. 3868	5067. 9739	900	I
5051	2039 (2)	2039 (2)	-----	5051. 2039	5049. 7959	400	II
5030	0581 (2)	0594 (2)	-----	5030. 0588	5028. 6564	400	II
5018	6537 (3)	6542 (2)	-----	5018. 6539	5017. 2545	500	II
5003	4922 (3)	4922 (2)	-----	5003. 4922	5002. 0968	400	I
4941	0205 (4)	0204 (2)	-----	4941. 0205	4939. 6417	350	I
4921	1894 (3)	1886 (2)	-----	4921. 1890	4919. 8155	600	II
4896	3214 (4)	3216 (2)	-----	4896. 3215	4894. 9546	350	I
4880	0950 (2)	0965 (1)	-----	4880. 096	4878. 733	200	I
4866	8360 (2)	8360 (1)	-----	4866. 8360	4865. 4769	350	I
4864	5306 (3)	5309 (1)	-----	4864. 5307	4863. 1722	1000	II
4842	1949 (2)	1953 (2)	-----	4842. 1951	4840. 8426	400	I
4809	4775 (3)	4770 (2)	-----	4809. 4773	4808. 1334	350	I
4790	7254 (3)	7259 (2)	-----	4790. 7256	4789. 3867	300	I
4767	9328 (2)	9334 (1)	-----	4767. 9330	4766. 6001	200	I
4753	7427 (2)	7432 (2)	-----	4753. 7430	4752. 4139	500	II
4705	3059 (3)	3060 (2)	-----	4705. 3060	4703. 9897	500	I
4687	5062 (2)	5055 (1)	-----	4687. 5060	4686. 1944	1200	I
4674	9691 (2)	9689 (2)	-----	4674. 9690	4673. 6608	600	I
4669	4787 (3)	4789 (2)	-----	4669. 4788	4668. 1720	700	I
4664	5076 (1)	5076 (2)	-----	4664. 5076	4663. 2021	200	I
4633	0584 (3)	0583 (2)	-----	4633. 0583	4631. 7611	1100	I
4596	7072 (3)	7085 (1)	-----	4596. 7074	4595. 4198	600	I
4589	7122 (2)	7124 (1)	-----	4589. 7123	4588. 4266	400	I
4572	2522 (2)	2533 (1)	-----	4572. 2526	4570. 9715	500	I
4557	0912 (2)	0890 (1)	-----	4557. 090	4555. 813	500	I
4536	5251 (2)	5266 (1)	-----	4536. 526	4535. 254	300	I
4511	7906 (3)	7910 (2)	-----	4511. 7908	4510. 5257	800	II
4494	5941 (3)	5942 (2)	-----	4494. 5941	4493. 3335	1200	I
4483	4270 (3)	4269 (2)	-----	4483. 4270	4482. 1694	300	I
4466	5936 (3)	5940 (3)	-----	4466. 5938	4465. 3406	300	II
4459	2531 (5)	2530 (3)	-----	4459. 2531	4458. 0018	600	I
4446	5562 (2)	5561 (2)	-----	4446. 5561	4445. 3082	300	I
4434	2074 (4)	2076 (2)	-----	4434. 2075	4432. 9628	600	II
4410	1211 (5)	1210 (3)	-----	4410. 1211	4408. 8828	600	I
4404	1635 (5)	1641 (3)	-----	4404. 1637	4402. 9270	400	I
4402	8179 (1)	8183 (1)	-----	4402. 8181	4401. 5817	400	I
4392	3440 (7)	3440 (2)	-----	4392. 3440	4391. 1104	3000	II
4383	0917 (5)	0915 (2)	-----	4383. 0916	4381. 8604	900	I

TABLE 2. Wavelengths in thorium spectra—Continued

1	2	3	4	5	6	7	8
Whole angstrom	Fractional angstrom			Vacuum	Air	Relative intensity	Spectrum
	25 mm(obs.)	40 mm(obs.)	50 mm(obs.)				
4379	4069 (5)	4072 (2)	-----	4379. 4070	4378. 1768	500	I
4375	3535 (2)	3538 (1)	-----	4375. 3536	4374. 1244	600	I
4367	1572 (5)	1575 (2)	-----	4367. 1573	4365. 9303	600	I
4359	5624 (4)	5624 (3)	-----	4359. 5624	4358. 3374	-----	Hg ¹⁹⁸
4343	4765 (4)	4760 (2)	-----	4343. 4763	4342. 2555	300	I
4332	0620 (3)	0617 (1)	-----	4332. 0619	4330. 8441	300	I
4319	6302 (1)	6307 (1)	-----	4319. 6305	4318. 4160	700	I
4316	4681 (3)	4681 (2)	-----	4316. 4681	4315. 2544	400	I
4308	3878 (3)	3878 (2)	-----	4308. 3878	4307. 1763	700	I
4301	0490 (3)	0486 (1)	-----	4301. 0489	4299. 8393	600	I
4293	0176 (3)	0178 (2)	-----	4293. 0177	4291. 8102	400	I
4278	5178 (3)	5181 (2)	-----	4278. 5179	4277. 3142	1200	II
4274	5599 (3)	5603 (2)	-----	4274. 5600	4273. 3574	1000	II
4258	6946 (3)	6941 (2)	-----	4258. 6944	4257. 4959	700	I
4236	6563 (3)	6560 (1)	-----	4236. 6562	4235. 4635	600	I
4231	6183 (3)	6180 (1)	-----	4231. 6182	4230. 4268	600	I
4216	0152 (2)	0161 (1)	-----	4216. 0156	4214. 8283	200	I
4210	0766 (3)	0762 (2)	-----	4210. 0764	4208. 8907	3000	II
4194	1981 (3)	1980 (2)	-----	4194. 1980	4193. 0165	900	I
4179	2375 (3)	2374 (2)	-----	4179. 2374	4178. 0598	3000	II
4166	9403 (3)	9403 (2)	-----	4166. 9403	4165. 7659	1000	I
4159	7078 (3)	7074 (2)	-----	4159. 7076	4158. 5351	800	I
4151	1566 (3)	1570 (2)	-----	4151. 1568	4149. 9865	800	II
4133	9191 (3)	9191 (1)	-----	4133. 9191	4132. 7534	600	II
4128	5761 (3)	5759 (2)	-----	4128. 5760	4127. 4117	1400	I
4116	9199 (3)	9201 (2)	-----	4116. 9200	4115. 7587	800	I
4109	5788 (3)	5791 (1)	-----	4109. 5789	4108. 4195	800	II
4101	4985 (3)	4984 (2)	-----	4101. 4984	4100. 3412	1100	I
4095	9025 (3)	9031 (2)	-----	4095. 9028	4094. 7470	1600	II
4087	6743 (2)	6739 (2)	-----	4087. 6741	4086. 5205	1600	II
4068	5993 (3)	5992 (2)	-----	4068. 5993	4067. 4507	400	I
4060	3990 (3)	3991 (2)	-----	4060. 3990	4059. 2525	1000	I
4047	7144 (3)	7144 (2)	-----	4047. 7144	4046. 5712	-----	Hg ¹⁹⁸
4044	5366 (3)	5371 (2)	-----	4044. 5368	4043. 3945	800	I
4037	1880 (3)	1878 (2)	-----	4037. 1879	4036. 0475	1800	I
4020	2649 (4)	2649 (2)	-----	4020. 2649	4019. 1289	4000	II
4013	6295 (3)	6292 (2)	-----	4013. 6293	4012. 4950	2000	I
4009	3433 (3)	3438 (2)	-----	4009. 3435	4008. 2104	1600	I
3995	6786 (3)	6787 (2)	-----	3995. 6786	3994. 5490	1200	I
3981	2147 (3)	2153 (2)	-----	3981. 2150	3980. 0892	1100	I
3968	5145 (3)	5142 (2)	-----	3968. 5144	3967. 3919	2000	I
3950	0814 (2)	0810 (1)	-----	3950. 0813	3948. 9636	1000	I
3934	0243 (3)	0243 (1)	-----	3934. 0243	3932. 9108	1400	I
3924	9103 (3)	9106 (1)	-----	3924. 9104	3923. 7993	400	I
3906	2992 (2)	2926 (1)	-----	3906. 2924	3905. 1861	1500	II
3870	7606 (2)	7604 (2)	-----	3870. 7605	3869. 6635	600	I
3864	5008 (3)	5011 (2)	-----	3864. 5009	3863. 4055	1200	II
3855	6036 (3)	6035 (2)	-----	3855. 6036	3854. 5105	1200	II
3843	0497 (3)	0499 (2)	-----	3843. 0498	3841. 9600	1200	II
3840	7829 (3)	7837 (2)	-----	3840. 7833	3839. 6941	2500	I
3829	4707 (3)	4709 (2)	-----	3829. 4708	3828. 3845	3200	I
3819	7691 (1)	7692 (1)	-----	3819. 7692	3818. 6855	500	I
3814	1497 (3)	1497 (2)	-----	3814. 1497	3813. 0674	1200	II
3804	1545 (3)	1550 (2)	-----	3804. 1547	3803. 0750	4000	I
3786	6748 (1)	6749 (1)	-----	3786. 6749	3785. 5997	1000	II

TABLE 2. Wavelengths in thorium spectra—Continued

1 Whole angstrom	2 Fractional angstrom			5 Vacuum	6 Air	7 Relative intensity	8 Spectrum
	3						
	25 mm(obs.)	40 mm(obs.)	50 mm(obs.)				
3782	0398 (1)	0405 (1)	-----	3782. 0402	3780. 9663	350	I
3772	4418 (3)	4418 (2)	-----	3772. 4418	3771. 3703	1500	I
3764	0037 (3)	0027 (2)	-----	3764. 0032	3762. 9345	1200	I
3753	6349 (3)	6353 (2)	-----	3753. 6351	3752. 5685	3500	II
3743	9871 (3)	9873 (2)	-----	3743. 9872	3742. 9231	1100	I
3728	9626 (3)	9622 (1)	-----	3728. 9624	3727. 9022	800	I
3720	4924 (3)	4926 (1)	-----	3720. 4925	3719. 4345	3000	I
3712	3595 (2)	3597 (2)	-----	3712. 3596	3711. 3037	600	II
3702	0310 (3)	0313 (2)	-----	3702. 0312	3700. 9780	300	I
3693	6171 (3)	6171 (2)	-----	3693. 6171	3692. 5661	1200	I
3683	5343 (3)	5347 (2)	-----	3683. 5345	3682. 4861	1000	I
3671	0137 (1)	0141 (1)	-----	3671. 0139	3669. 9687	750	I
3669	1842 (3)	1844 (2)	-----	3669. 1843	3668. 1396	1000	I
3657	7352 (3)	7354 (2)	-----	3657. 7353	3656. 6936	1000	I
3651	1962 (2)	1971 (1)	-----	3651. 1967	3650. 1566	-----	Hg ¹⁹⁸
3643	2866 (3)	2867 (2)	-----	3643. 2867	3642. 2487	2200	I
3633	8656 (3)	8654 (2)	-----	3633. 8655	3632. 8299	1000	I
3623	8282 (3)	8280 (2)	-----	3623. 8281	3622. 7951	800	I
3616	1633 (1)	1634 (1)	-----	3616. 1634	3615. 1324	2000	II
3613	4575 (3)	4573 (2)	-----	3613. 4574	3612. 4271	1400	I
3599	1459 (3)	1464 (2)	-----	3599. 1462	3598. 1196	2000	I
3593	8040 (3)	8042 (2)	-----	3593. 8041	3592. 7788	2000	I
3585	1985 (2)	1981 (2)	-----	3585. 1983	3584. 1753	800	I
3577	5786 (3)	5782 (2)	-----	3577. 5784	3576. 5573	1000	II
3568	2818 (3)	2825 (2)	-----	3568. 2822	3567. 2635	1200	I
3560	4658 (1)	4657 (1)	-----	3560. 4657	3559. 4490	2500	II
3552	4161 (3)	4158 (2)	-----	3552. 4159	3551. 4013	1000	I
3545	0306 (3)	0300 (2)	-----	3545. 0303	3544. 0176	1500	I
3540	5980 (3)	5983 (2)	-----	3540. 5982	3539. 5867	4000	II
3519	4094 (3)	4095 (2)	-----	3519. 4094	3518. 4033	1000	I
3512	1610 (3)	1613 (2)	-----	3512. 1612	3511. 1570	1000	I
3504	7880 (1)	7870 (1)	-----	3504. 7875	3503. 7852	500	I
3499	6215 (3)	6216 (2)	-----	3499. 6216	3498. 6206	900	I
3494	5174 (2)	5174 (1)	-----	3494. 5174	3493. 5177	2000	II
3480	1680 (3)	1688 (1)	-----	3480. 1683	3479. 1723	800	II
3469	2126 (3)	2125 (2)	-----	3469. 2125	3468. 2193	2000	II
3463	8419 (2)	8417 (2)	-----	3463. 8418	3462. 8500	2000	II
3452	6910 (3)	6908 (2)	-----	3452. 6909	3451. 7019	900	I
3443	5652 (3)	5651 (2)	-----	3443. 5651	3442. 5785	800	I
3434	9827 (3)	9831 (2)	-----	3434. 9829	3433. 9985	3000	II
3422	1908 (3)	1910 (2)	-----	3422. 1909	3421. 2097	2500	I
3413	9918 (3)	9918 (2)	-----	3413. 9918	3413. 0127	1800	I
3406	5346 (3)	5348 (2)	-----	3406. 5347	3405. 5575	1400	I
3397	7021 (3)	7022 (2)	-----	3397. 7022	3396. 7273	1400	I
3393	0083 (1)	0087 (1)	-----	3393. 0085	3392. 0348	6000	II
3386	5033 (3)	5030 (2)	-----	3386. 5032	3385. 5312	800	II
3381	8300 (3)	8305 (2)	-----	3381. 8303	3380. 8595	900	I
3375	9438 (3)	9440 (2)	-----	3375. 9439	3374. 9746	1600	I
3359	5663 (3)	5668 (3)	-----	3359. 5666	3358. 6014	2000	II
3352	1910 (3)	1913 (2)	-----	3352. 1911	3351. 2278	2500	II
3338	8296 (3)	8297 (2)	-----	3338. 8296	3337. 8697	2000	II
3331	4347 (2)	4343 (1)	-----	3331. 4345	3330. 4765	1800	I
3326	0765 (3)	0768 (2)	-----	3326. 0767	3325. 1201	3000	II
3325	7088 (3)	7088 (2)	-----	3325. 7088	3324. 7523	2000	II
3310	3174 (3)	3180 (2)	-----	3310. 3176	3309. 3650	800	I
3305	1894 (3)	-----	-----	3305. 1894	3304. 2381	3000	I
3293	4685 (3)	4686 (1)	-----	3293. 4685	3292. 5202	3000	II
3288	7355 (3)	7357 (2)	-----	3288. 7356	3287. 7885	1600	II

TABLE 3. Test of Th I wavelengths

Line pair	Wavelength	Wavenumber	Wavenumber difference
	λ	K	K
1	4494. 5941	22248. 950	2869. 260
	5160. 0414	19379. 690	
2	4375. 3536	22855. 296	2869. 255
	5003. 4922	19986. 041	
3	3840. 7833	26036. 356	2869. 267
	4316. 4681	23167. 089	
4	3643. 2867	27447. 744	2869. 261
	4068. 5993	24578. 483	
5	3599. 1462	27784. 367	2869. 261
	4013. 6293	24915. 106	
6	3331. 4345	30017. 099	2869. 261
	3683. 5345	27147. 838	
7	3310. 3176	30208. 582	2869. 261
	3657. 7353	27339. 321	
8	4896. 3215	20423. 495	3687. 989
	5975. 3199	16735. 566	
9	3829. 4708	26113. 269	3687. 989
	4459. 2531	22425. 280	
10	3305. 1894	30255. 452	3687. 993
	3764. 0032	26567. 459	
11	4896. 3215	20423. 495	5563. 143
	6729. 3157	14860. 352	
12	4258. 6944	23481. 375	5563. 143
	5580. 9077	17918. 232	
13	4104. 4981	24381. 333	6362. 396
	5549. 7170	18918. 937	
14	4037. 1879	24769. 716	6362. 396
	5432. 6212	18407. 320	

ards is 10 Å from 3300 to 4300 Å, 17 Å from 4300 to 5300 Å, 23 Å from 5300 to 6300 Å, and 30 Å from 6300 to 7000 Å. This distribution according to wavelength corresponds closely to an average spacing of standards at intervals of 72 K on a wavenumber scale throughout the range 14300 to 30400 K. Since spectra are always measured in wavelength units, but interpreted in wavenumber units (proportional to energy) this is suggested as the ideal distribution of secondary standards of wavelength. It is emphasized again that the values in table 2 are only a preliminary list which should be refined by comparing still higher orders of interference, and perhaps measuring still more wavelengths to decrease the intervals between standards.

The present list of 222 thorium wavelengths in table 2 may serve for immediate direct measurement of complex spectra from 3300 to 7000 Å, and by photographing these standards in a grating's second-order spectrum and doubling the values, they may serve also for the measurement of spectra from 6600 to 14000 Å photographed in the grating's first order.

Finally, the most important next step is to extend the measurement of wavelengths from thorium-halide lamps to shorter waves, that is from 3300 to 2000 Å or less. When this is done the iron arc at atmospheric pressure may be abandoned as a source of standard wavelengths (except for rough measurements) but still retained as an aid in recognizing the thorium standards. Because of the highly complex character of thorium spectra, and the lack of striking groups of lines or useful maps, it will be convenient (and perhaps necessary) to juxtapose the simpler and more familiar spectrum of the iron arc for purposes of orientation.

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UNITED STATES DEPARTMENT OF COMMERCE • Sinclair Weeks, *Secretary*
NATIONAL BUREAU OF STANDARDS • A. V. Astin, *Director*

Design of Free-Air Ionization Chambers

H. O. Wyckoff and F. H. Attix



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Preface

In the past, free-air standard chambers have been used in a few of the national standards laboratories. This was sufficient for the need at that time, for these chambers are principally used for the calibration of the smaller clinical and field instruments. With the increase in radiation therapy, more and more of the national laboratories have had to become interested in the problem. Also many industries have found it worthwhile to provide such calibration facilities.

In the past several years, some of the larger national standards laboratories have intercompared their free-air chambers. Differences between the chambers after proper corrections are applied amount to the order of 0.5 percent. In order to disseminate the information obtained during these calibrations and during the evaluations of the corrections which are to be applied to their readings, it seemed worth while to incorporate it into a single publication. The present report depends largely upon information obtained at the National Bureau of Standards, but includes also such information from published work of other laboratories as is pertinent to the problem.

This is a problem that is of interest to both the National Committee on Radiation Protection and Measurements, and the International Commission on Radiological Units and Measurements. The authors would like to express their gratitude to members of these two groups who have read the manuscript and have given them the benefit of their criticisms. In this regard, the authors wish especially to thank C. Garrett of the National Research Council, Ottawa, Canada, who suggested several additional topics to be included in the report.

A. V. ASTIN, *Director.*

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Design of Free-Air Ionization Chambers

H. O. Wyckoff and F. H. Attix

General design characteristics are given for standard free-air type ionization chambers for X-rays from 50 to 500 kilovolts. Included are the plate-separation requirements, elimination of electric-field distortion, data on air-attenuation and scattering effects, current measurement techniques, and other relevant features.

1. Introduction

The roentgen has been used for many years as a unit of X-ray dose. The free-air ionization chamber is the instrument employed in the experimental realization of this unit. Until recently, free-air-chamber installations have been confined principally to some of the national standards laboratories. However, a number of other laboratories have become interested in setting up their own primary standards for measurement in roentgens. In addition, there have been several papers [1 to 7]* pointing up small errors in the existing instruments.

Generally, such primary standards are used only for calibration of secondary instruments which have greater flexibility for field use. For some applications, such secondary instruments should have an accuracy of perhaps ± 1 percent. Thus, it is desirable that calibration of such secondary standard instruments be reproducible to within 1 percent when measured by the various primary standard instruments of the world. As the calibration of secondary instruments in terms of the primary is not itself without some experimental error, it is necessary to know the over-all errors of the primary instrument to, say, ± 0.5 percent. Thus all of the factors influencing this accuracy must be known to a few tenths of 1 percent. A review of the design criteria and of their absolute accuracies therefore seems desirable.

The International Commission on Radiological Units and Measurements has defined [8] the unit of X-ray dose as follows: "One roentgen is an exposure dose of X- or gamma radiation such that the associated corpuscular emission per 0.001293 gram of air produces, in air, ions carrying 1 electrostatic unit of quantity of electricity of either sign." According to this definition, a measurement of the number of roentgens requires that one measure all of the ionization produced in air by the high-speed electrons that are themselves produced within the defined mass of air. Real difficulties arise in the measurement of the ionization as required by the definition, so the principle of electronic equilibrium is always used in practice.

According to the principle of electronic equilibrium, within a medium under uniform irradiation the ionization produced outside of a specified mass, m , by high-speed electrons generated inside m is compensated by ionization in m produced by high-speed electrons generated outside of m [9]. Elec-

trons produced out to a distance from m equal to the electron range thus contribute to the ionization in m . In order to conform to the definition of the roentgen, it is obvious that the ionization produced by the scattered radiation from this region must be eliminated in the measurement.

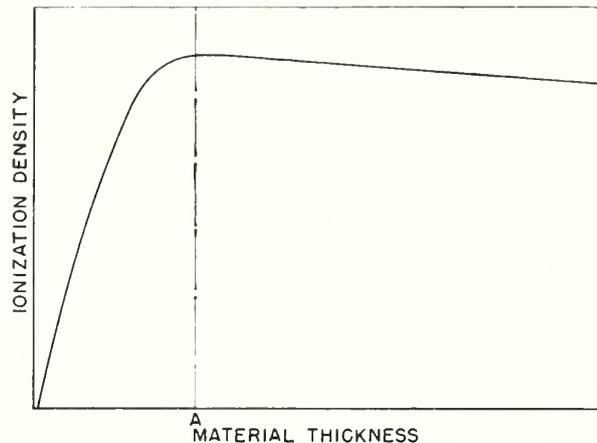


FIGURE 1. Growth of ionization density in a small volume of gas from an initially electron-free beam of photon radiation as the gas is surrounded by more and more material.

Electronic equilibrium is said to exist for a thickness, A , of material.

Figure 1 assists in picturing this electronic equilibrium. This figure indicates the ionization density in a small volume of gas due to an initially electron-free beam of X- or gamma radiation as the gas is surrounded by more and more material. The photon beam interacts with the material to produce high-speed electrons, but in the process is itself reduced in intensity. These two competing mechanisms cause an initial increase in the curve (due principally to the increase in the number of high-speed electrons), a plateau, and finally a decrease (principally due to the decreased intensity of the photon beam). A thickness of material corresponding to A in figure 1 is approximately equal to the electron range in the material.

Special considerations involved in applying the idea of electronic equilibrium to free-air chambers will be brought out in the next section.

1.1. Characteristics of Free-Air Chambers

While free-air chambers of different detailed construction have been made, they all have the general characteristics indicated in figure 2. This is a plan view of a parallel-plate ionization chamber

* Figures in brackets indicate the literature references at the end of this Handbook.

like the ones used at the National Bureau of Standards and at the National Physical Laboratory in Great Britain. Chambers having other electrode configurations are also used as standards in other laboratories.¹ The plate system is contained in a radiation-shielded box. A known area of the horizontal X-ray beam is defined by the diaphragm, D, so that a beam of photons passes centrally between the plates. A high potential (field strength of the order of 100 v/cm) on plate H, with respect to the other plates, sweeps out the ionization produced in the air between the plates. The ionization is measured for a length, L, determined by the limiting lines of force to the edges of the collector, C. These lines are made straight and perpendicular to the collector by the guard plates, G, and surrounding guard wires or strips, W. The latter are connected to a resistance-dividing network to grade the potential uniformly across the gap between C or G, and H. Generally, guard plates like G are not considered necessary above and below the collector because there the ionization density is low.

Ionization is collected throughout the region enclosed by the dashed lines, F. Some electrons, such as e_1 , having paths predominantly perpendicular to the X-ray beam, will expend their entire energies within the ion-collecting region, assuming the plate separation to be sufficiently large. (Actually, of course, the electron paths are not straight as indicated.) Others, like e_2 , will pass out of that region and produce ionization where it will be collected on one of the guard plates and hence will not be measured. Under the electronic equilibrium condition, this ionization loss is just compensated by the contribution of other electrons such as e_3 , assuming air attenuation of the primary beam to be small. Such a condition can exist only if the two ends of the box housing the chamber are at a distance (from the ion-collecting region) greater than the range of the electrons present, or about half the distance required for plate separation.

Electrons, like e_4 , resulting from scattered photons, produce ionization which is not included in the definition of the roentgen, as these electrons do not originate directly from photons collimated by D. Their ionization contribution is usually assumed to be negligible. In large chambers, however, that assumption may not be justified. On the other hand, the presence of such unwanted ionization tends to compensate for ionization losses due to insufficient plate separation.

In the calibration of a secondary radiation instrument by a standard chamber, a substitution method is used. The dose rate is determined from the measurements of the ionization in the standard chamber, this chamber is removed, and the secondary instrument is placed at the position formerly occupied by the aperture of the standard

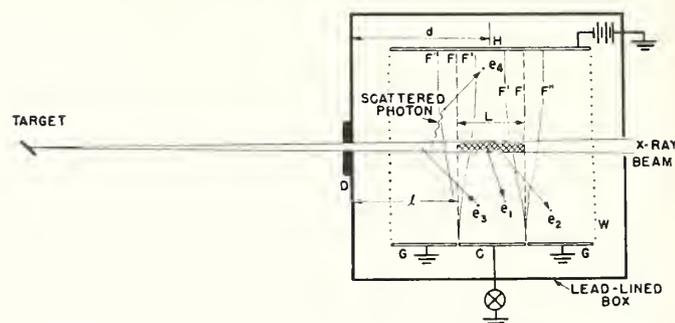


FIGURE 2. Schematic plan view of a parallel-plate free-air chamber.

chamber. Therefore it is necessary to compute the dose rate at the aperture of the standard chamber. This computation requires a knowledge of the absorption of X-rays in the air between the aperture and the weighted positions of electron production. Thus, for the X-rays producing electrons such as e_3 (fig. 2), the absorption is less than for those producing electrons like e_1 . However, it has been shown [5] that even for 500-kv X-rays the contribution of electrons like e_3 is so small that the effective center for electron production is essentially the center of the collector. Therefore the air-absorption correction should be made for the distance between the center of the collector and the aperture.

In the Swedish type of free-air chamber [10], the high-voltage plate is a cylinder, and the second electrode is also cylindrical and coaxial with it. A central portion of this inner electrode serves as collector. The diaphragmed beam passes through the chamber parallel to its axis and between the center and outer electrodes. The Kustner-type chamber [11] also uses an outer cylindrical high-voltage electrode. However, the beam passes along the axis of the chamber and the collector is placed between this radiation beam and the outer electrode.

The factors that must be investigated for the proper design of one of these chambers include (1) the diaphragm system, (2) the effective length of the region of ion collection as it is distorted by variations in the electric field, (3) the spacing between the beam and the high-voltage electrode, and between the beam and the collector electrode, (4) the distance between the diaphragm and the collector, (5) the collecting potential required in order to achieve saturation (i. e., so that negligible recombination of the ionization is permitted), (6) the air absorption between the center of the collector and the aperture, and (7) radiation shielding of the box.

In the present report information is collected on the above design factors for X-rays of 60 to 500 kv. As an example of the use of these data, an estimate is made of the corrections to be applied to the measurements by two free-air chambers. This is compared with the data obtained in an experimental intercomparison between the chambers.

¹ The advantages of the various kinds of configurations are discussed by Rajewsky, et al. [32].

2. Details of Chamber Design

2.1. The Defined Mass of Air

In a free-air chamber the aperture area and the length of the collecting electrode (along the beam direction) define a *volume* of air. The definition of the roentgen, on the other hand, requires measurement of the ionization per *mass* of 0.001293 g of (dry) air (which numerically equals the density of dry air, in grams per cubic centimeter, at 0° C and 760 mm of mercury). Thus one must apply suitable corrections to the ionization measured under other conditions than these, to reduce the results to the standard conditions. For ordinary operating temperatures and pressures, the perfect-gas law may be used. If a unit mass of air is defined as 0.001293 g, the number of unit masses of air in a given volume V (cm³) equals

$$\frac{273.2}{T} \frac{PV}{760} \quad (1)$$

where T (in degrees Kelvin) is the air temperature,² and P (in millimeters of mercury) is the pressure. However, air usually contains water vapor. As the electron densities of air and water vapor are different, the density must be corrected for this difference if absolute determinations to better than the order of 1 percent are desired. Furthermore, the energy W , needed to produce an ion pair in water vapor, is somewhat less than that in air.³ For humid air, the effective number of unit masses of air [12] within volume V equals⁴

$$\frac{273.2}{T} \frac{(P - 0.238P_1)V}{760} \quad (2)$$

where P_1 is the vapor pressure of the moisture in the air (in mm of mercury). The volume, V , is determined for a free-air chamber by the product of the area of the diaphragm aperture and the effective length of the collecting region [13] (see section 2.3.).

2.2. Diaphragm

Usually the diaphragm consists of a disk of dense metal attached to the front of the box in such a way that no X-rays can leak past its outer edge.

² Because the air in the chamber may be heated slightly by the resistors used for collector-field uniformity and because drafts may cause temperature gradients, the air temperature should be measured inside the box itself.

³ According to a private communication from G. Failla, $W_{H_2O} \approx 0.9W_{air}$. This value has been assumed here in weighting the effectiveness of the water-vapor electrons.

⁴ In addition, for low-energy photons, water vapor is less effective in producing ionization than are those of air because the energy absorption coefficient for air is greater. Thus, for example, at 50 keV, the energy absorption coefficient for air is about 8 percent greater (per electron) than that for H₂O vapor. At room temperatures with 100 percent humidity, the water vapor electrons constitute ≈ 2 percent of the electrons present. Therefore the energy absorbed (and thus the ionization) is about 0.2 percent less than if the same number of electrons of dry air were present. At keV's down to 10 keV, this remains in the neighborhood of 0.2 percent (see table 1 in ref [8]). At 80 keV, it has dropped to ≈ 1 percent, and at 100 keV is nearly zero. Note that this is separate from the effect of electron density which we have taken into account already. However, the relative humidity is usually 50 percent or less in order to secure low insulator leakage. Therefore this effect will not be important in most cases.

The hole in the middle of this disk, through which the X-rays pass into the box, is usually cylindrical in shape because of the ease of construction and measurement (although the Kustner-type chamber sometimes uses a crescent-shaped aperture). The size of this hole is arbitrary, although certain factors should be considered in making a choice.

Too small a hole may result in:

- Difficulty in machining.
- Difficulty in accurately measuring the area.
- Criticality of alinement with the X-ray beam.
- Excessive secondary radiation from the surface.⁵

Too large a hole may cause:

- Excessive ion recombination at a given collecting potential.
- Attenuation of the X-rays in the guard wires or strips on the front of the plate system. (Removal of the wires or strips increases field distortion.)
- Increased ionization losses due to inadequate plate separation.
- Nonuniformity of the X-ray beam over the area of the hole.

A hole of the order of 1 cm in diameter has been found to be adequate for the energy region under consideration. Holes of this diameter have been produced in tungsten alloy, by reasonably careful shop practices, to be circular and of uniform diameter within 1 μ , over a length of 1 cm.

The thickness of the peripheral region of the diaphragm disk should produce an X-ray attenuation as great as that of the shielding on the front of the box (which is discussed later). The attenuation near the edge of the hole need not be so great, however, because the thickness necessary to attain this attenuation would cause the alinement of the diaphragm aperture with the X-ray beam to become unnecessarily critical. It is preferable to taper a portion of the aperture (on the side away from the X-ray source) in the manner indicated in figure 3. While the angle of taper, θ , is not critical, it should be chosen small enough to

⁵ König [15] has shown that the fractional contribution of secondary radiation from the cylindrical surface of the aperture is inversely proportional to the aperture diameter.

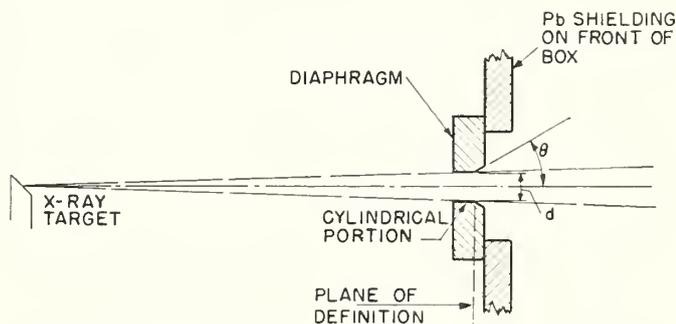


FIGURE 3. Defining diaphragm with an aperture diameter, d , and taper, θ .

preserve the full attenuation over most of the diaphragm area and to make negligible the penetration of X-rays through the edges of the aperture at the plane of definition (i. e., the plane where the aperture limits the solid angle of X-rays passing through). θ must be large enough, however, so that the plane of definition does not shift to the rear surface of the diaphragm for the smallest target-to-diaphragm distance to be employed. The experience at the Bureau has shown that $\theta=45^\circ$ is adequate for the energy range under consideration.

Selection of the depth of the cylindrical part of the aperture, of course, depends on the material used in constructing the diaphragm. Gold, lead, and tungsten alloy ($\sim 89\%$ W, 7% Ni, 4% Cu) have all been used at the Bureau. Aperture areas in diaphragms of gold and lead have been found stable to within 0.1 percent over a period of 25 years. However, these materials are not readily machinable with high precision. Tungsten alloy machines very well and is therefore probably preferable. Although the authors' experience with this material covers only a 3-year period, it has been found to maintain very closely its dimensions.

Temperature coefficients of linear expansion lie between 5 and $20 \times 10^{-6}/^\circ\text{C}$ for these three materials; thus the alteration of aperture dimensions by changing temperature is negligible for temperature ranges usually encountered.

Table 1 gives the narrow-beam attenuation coefficients for the above tungsten alloy, for monochromatic irradiation. Diaphragms should be designed to reduce the X-ray penetration (integrated over the diaphragm surface) to a negligible fraction (less than 0.1 percent) of the X-rays entering the box through the aperture.

TABLE 1. *Monochromatic attenuation coefficients for narrow-beam conditions in tungsten alloy ($\sim 89\%$ W + 7% Ni + 4% Cu)*

kev	$\mu(\text{cm}^{-1})$
50	71.2
100	66.8
150	23.1
200	11.6
300	4.90
400	2.96
500	2.14

A second diaphragm located some distance behind the defining diaphragm (in the direction away from the X-ray source) has sometimes been used. This is intended to stop X-rays scattered from the first diaphragm. Measurements at the Bureau have shown that this extra diaphragm is (a) unnecessary because the diaphragm scatter and fluorescence amounts to only a few hundredths of 1 percent of the radiation passing through the aperture,⁶ and (b) undesirable because the presence of a scatter diaphragm increases the criticality of the beam alinement.

⁶ König [15] indicates that the scatter from a 1-cm-diameter aperture is about 0.3 percent of that passing through the aperture. However, much smaller amounts are observed at the Bureau.

These corrections were measured by a method described elsewhere [3] which includes any wide-angle scatter from the diaphragm. Measurements of G. H. Aston [17] also confirm that the diaphragm scatter does not contribute more than 0.1 percent to the ionization for several different types of aperture construction with an aperture diameter of approximately 1 cm. The alinement of the defining diaphragm with the X-ray beam remains somewhat critical, even with no scatter diaphragm present. For an aperture of 1 cm in both diameter and depth, the passage of X-rays from a target 62 cm away was experimentally found to decrease by about 2 percent when the angle between the aperture axis and the beam direction was changed from 0 to 1.4 degrees. Thus this angle must be reduced to a few minutes of arc if the error is not to exceed 0.1 percent.

The procedure for alining the diaphragm with the X-ray beam consists of two steps:

(a) Placing the chamber so that the diaphragm is located approximately at the center of the X-ray beam.

(b) Orienting the chamber so that the axis of the diaphragm aperture is parallel with the rays passing through.

Step (a) can be conveniently done visually with the aid of a fluorescent screen. Step (b) can be accomplished in several ways. If the chamber enclosure has been constructed with sufficient care the rear exit hole will be concentric with the axis of the diaphragm aperture. In that case one can place crossed wires of dense material (e. g., tungsten) over the exit hole and observe their X-ray shadow on a fluorescent screen. If the geometrical precision of the enclosing box is not sufficient to allow this procedure, a substitute diaphragm of brass or steel can be constructed for use in alinement. This should be made with a very long aperture of small diameter, thus being much more critical to aline than the regular diaphragm. (This need not be the same diameter throughout, but can be made as a tube with a small aperture at each end.) Passing X-rays through this diaphragm and observing their image on a fluorescent screen will allow the chamber to be properly alined with the beam.

2.3. Field Distortion

The length of the collection region is usually fixed by the effective⁷ collector electrode length and by assuring that the limiting lines of force between the collector and the high-voltage electrode are perpendicular to the collector. A similar problem has been solved in standard capacitor design by the use of guard plates. However, the dimensions of the electrode system for an air chamber may be orders of magnitude

⁷ The gaps between the collector and guard electrodes are made very small ($<4\%$) [18] compared to the collector length, and the effective collector length is the collector length plus one-half of the sum of the two gaps. Note that the entire parallel-plate system must be alined parallel with the X-ray beam, to avoid cosine errors amounting to 0.1 percent for 2.5 degrees of angular misalinement.

larger than for a capacitor. The proximity of a grounded box around the free-air chamber electrodes tends to distort the electric field; however, it is important to make the box as small as possible to minimize its weight. A more efficient system than simple guard electrodes alone is therefore desirable. Aluminum guard wires [19], guard rods [20], or guard strips [5, 21, 22] have been employed. For parallel-plate chambers these are located, as shown in figure 4, at the periphery of the plate system, uniformly spaced between the ground and high-voltage plates, and parallel to them. Their potentials are fixed by a voltage divider to give a linear gradient between the plates. The current through the voltage divider should be large compared with the ionization current, but not so large that the power dissipated within the box is excessive. Power dissipation of the order of a few tenths of 1 watt does not cause the temperature of the air in the box to rise appreciably.

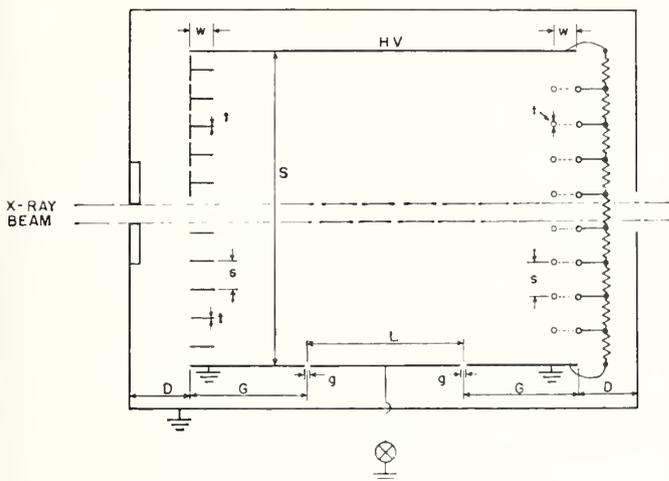


FIGURE 4. Three types of peripheral electrode systems for guarding the electric field, in a parallel-plate chamber, from the influence of the grounded enclosures.

At the right is shown the double-wire system. Dashed lines indicate the pairs of wires at the same electrical potential, fixed by the linear voltage divider. At left are shown parallel strips and T-section electrodes. An opening must be left for the X-ray beam to pass through, to avoid attenuation and scattering. Important dimensions are lettered for reference in the text (t is the diameter of wire or thickness of the strips).

The adequacy of the guarding is sometimes determined from symmetry considerations. Figure 2 assists in picturing this symmetry for parallel-plate geometry. The lines, F , show the proper position for the defining lines of force. Distortion of a grounded box would produce distortion shown schematically by lines F' . If the box is placed at the potential of the high-voltage electrode, the defining lines become F'' . For symmetrical conditions (i. e., if the guard electrodes are of the same length and the box is the same distance from each), and for small distortions, the reduction in effective collection length for the first condition should be equal to the increase from the second. Thus the average of the two should correspond to a measurement made with an undistorted field.

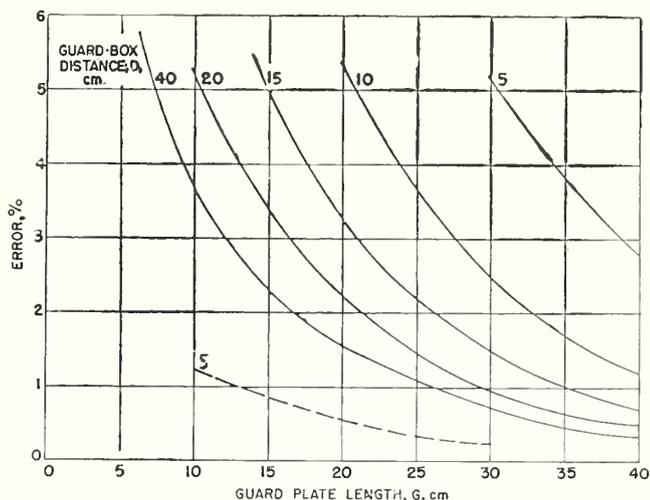


FIGURE 5. Error in collecting length caused by field distortion in a two-dimensional analog of a parallel-plate free-air chamber.

L (fig. 4) = 10 cm, S = 40 cm, D and G variable. The data are for double guard wires (solid lines) and guard strips (dotted line), with s = 2 cm, w = 3 cm, and t = 0.5 mm.

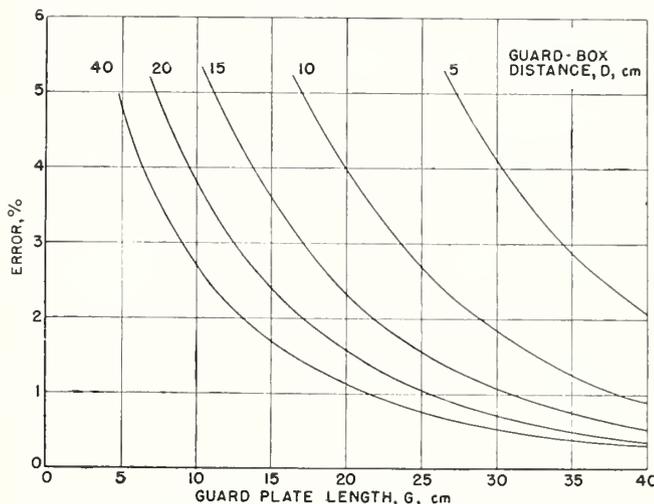


FIGURE 6. Error in collecting length caused by field distortion in a two-dimensional analog of a parallel-plate free-air chamber.

L (fig. 4) = 20 cm, S = 40 cm, D and G variable. The data are for double guard wires with s = 2 cm, w = 3 cm, and t = 0.5 mm.

These considerations have been used to determine and correct for field distortion. This has been done with actual chambers by measuring ionization under the two conditions of box potential [23] and has also been used in a resistance analog method [4] for determining the distortion in two dimensions for a parallel-plate system. Unfortunately, the two-dimensional results overestimate the three-dimensional distortion,⁸ but they are the only extensive data available on the subject. Figures 5 and 6 show the distortion for

⁸ In one case considered, Miller and Kennedy [4] predicted a distortion of about 1 percent whereas the ionization chamber actually indicated an error of 0.3 percent. However, when the third-dimensional effect was eliminated in the ionization chamber, the measured distortion error was 0.7 percent—in fair agreement with the predicted 1 percent.

two-dimensional conditions with double wires or strips. The two wires of a given set are in the same plane and connected electrically; the strips have their large dimension in the plane parallel to the electrodes. These data were obtained with a plate separation of 40 cm (S , fig. 4), with 2 cm (s) between adjacent guard wires in the same plane, with 3 cm (w) between the two guard wire layers, and with guard wires of approximately 0.5-mm diameter (t). The strips were $t=3$ mm by $w=2$ cm in cross section. Figure 7 shows the advantage of using larger-diameter wires. The distortion, of course, is unchanged if all of the dimensions are scaled equally. Guard wires may be necessary to reduce absorption in the collimated beam, but the more efficient guard strips should be used outside of this region.

In the design of a parallel-plate free-air chamber, it is recommended that the field distortion be estimated from figures 5, 6, and 7. A design should be chosen so that the two-dimensional distortion is less than, say, 0.5 percent. The actual distortion should then be determined experimentally on the completed chamber from symmetry considerations. Generally the experimentally determined distortion will be lower than the two-dimensional estimate and independent of photon energy.

In addition to the field distortion caused by the proximity of the grounded box, there are at least four other ways in which field distortion can arise.

a. *Distortion near guard wires or strips.*

The guard wires or strips must be sufficiently far from the collecting region so as not to individually distort the field there. In one particular arrangement [24], a distance of about 10 times the guard-strip separation seems to be adequate. This means that ($G-W$) (fig. 4) should be at least 10 times s .

b. *Misalignment of the collector plate and adjacent guard plates.*

For a parallel-plate chamber, these plates should be coplanar. If they are not, the collector will receive too much ionization if it projects toward the X-ray beam, or too little if it is recessed behind the plane of the guard plates. For $L=10$ cm and $g=0.9$ mm, the error in ionization current collected is approximately 0.1 percent per 0.025 mm of misalignment. This was found to be independent of S over the observed range of 6 to 10 cm, and hence may be assumed to be caused by a local field disturbance in the immediate region of the junction of the collector and guard plates. The magnitude of this error should be inversely proportional to L for a constant value of g , and independent of the potential on the high-voltage plate.

c. *Contact potential difference between guard plates and collector electrodes.*

If the work functions of the surfaces of the collector and guard plates are appreciably different, a potential difference sufficient to distort the field will result across the gap between them. The

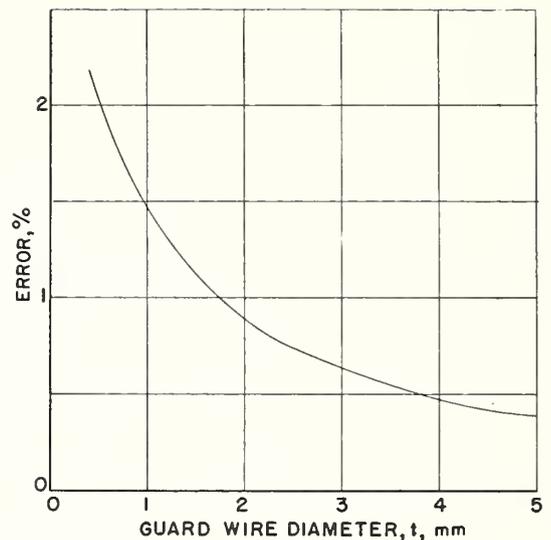


FIGURE 7. Effect of guard-wire diameter, t , on the errors shown in figures 5 and 6.

S (fig. 4)=40 cm, $L=10$ cm, $G=40$ cm. single guard wires with $s=2$ cm.

collector will receive more or less than the correct ionization current, depending on the polarity of the potential on the high-voltage plate. This effect has been observed at the Bureau even when the collector and guard plates of an experimental parallel-plate chamber were of the same material (aluminum). Coating these plates with colloidal graphite removed the effect, which was causing an error of several tenths of 1 percent. Most of the effect disappeared when only a 1-cm-wide strip on each plate, adjacent to the gap, was coated, which tends to confirm further the nature of the effect as being contact potential difference between the plate surfaces near the gap. If this error is present in a chamber, it shows up as a difference in the measured ionization when the polarity of the potential on the high-voltage plate is reversed. The magnitude of the error is reduced by increasing the high-voltage potential. Thus for sufficiently high potential to satisfy saturation requirements, the contact potential error becomes small and the ionization currents measured with the two polarities approach their average value [25].

d. *Externally imposed potential difference between collector and guard electrodes.*

This causes distortion similar to that of contact potential difference (see section 3).

2.4. X-ray Scattering and Electron Losses

The collimated beam of X-rays within the chamber interacts with the air to produce high-speed electrons and secondary photons (Compton and fluorescent). These secondary photons may in turn produce high-speed electrons within the measuring volume. However, a measurement in roentgens requires a determination of the ionization from the high-speed electrons produced by the collimated photons alone. Thus it is well to consider separately the ionization from the two

mechanisms (electron and secondary photon). Both ionization densities are functions only of the radial distance from the photon beam.⁹ However, the gain of ionization from the secondary photons tends to compensate for the loss of ionization from the high-speed electrons due to insufficient plate spacing.

Figures 8, 9, and 10 show the percentage of electron ionization loss, E_r , beyond a radius, r , from the photon beam, computed for a zero diameter of beam (air at 26° C and 760 mm Hg).¹⁰ Figures 11, 12, and 13 show the electron contribution per radius increment, $\Delta E/\Delta r$, at different inner radii. Table 2 shows the unwanted secondary photon contribution, S_r (within a radius, r), in percentage of the total electron contribution (i. e., ionization resulting from complete utilization of the electrons produced by primary photons).

⁹ It is assumed that electronic equilibrium exists. See section 2.5 for distance between aperture and collector for electronic equilibrium.
¹⁰ For other pressures and temperatures, the distances given should be scaled to the same mass of air.

As this contribution varies very slowly with the radius, 5-cm increments have been taken. The secondary radiation contribution is also for a zero-diameter beam. The use of these data to compute the ionization excess or deficiency for a particular design is illustrated later.

TABLE 2. Secondary photon contribution

Radius	* Secondary photon contribution, S_r								
	60	75	100	150	200	250	300	400	500
<i>cm</i>	<i>kv</i>	<i>kv</i>	<i>kv</i>	<i>kv</i>	<i>kv</i>	<i>kv</i>	<i>kv</i>	<i>kv</i>	<i>kv</i>
5	0.14	0.14	0.13	0.11	0.10	0.09	0.08	0.06	0.06
10	.32	.32	.30	.25	.23	.21	.19	.17	.17
15	.55	.55	.51	.44	.40	.36	.33	.29	.29
20	.79	.79	.74	.63	.58	.53	.47	.42	.42
25	1.02	1.02	.95	.81	.75	.68	.61	.54	.54
30	1.23	1.23	1.15	.98	.90	.82	.74	.66	.66
35	1.43	1.43	1.33	1.14	1.05	.95	.86	.76	.76
40	1.62	1.62	1.51	1.30	1.19	1.08	.97	.86	.86

* Percentage of total electron contribution for filtrations listed in figures 8 and 10.

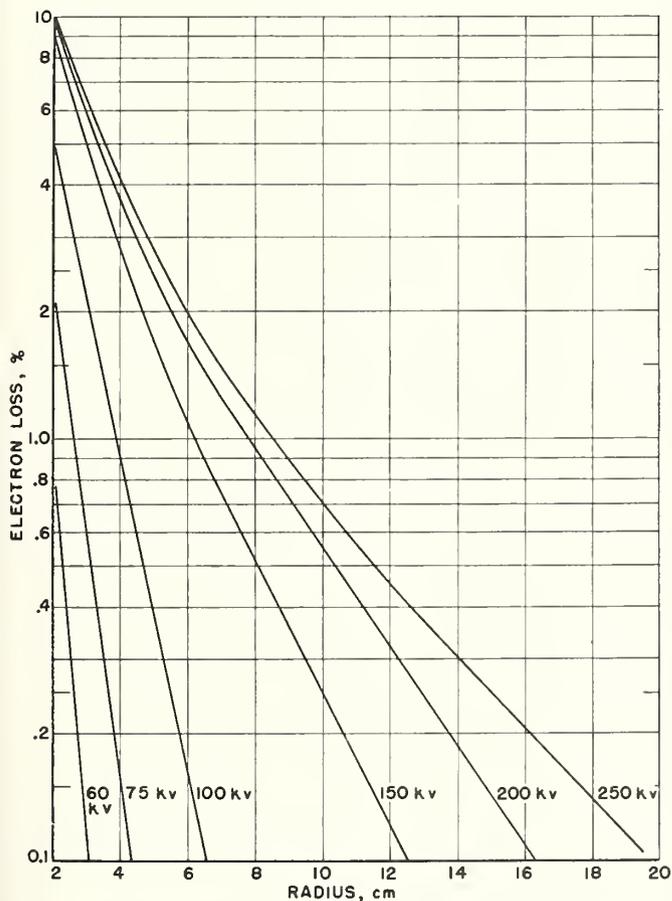


FIGURE 8. Loss of electron-produced ionization (in percentage of total electron ionization) beyond different radii from a zero-diameter beam of constant potential X-rays.

The X-ray potentials were 60 and 75 kv with zero added filter, 100 kv with 1 mm Al added filter, 150 kv with 0.23 mm Cu+1 mm Al, 200 kv with 0.5 mm Cu+1 mm Al, and 250 kv with 1 mm Cu+1 mm Al. The X-ray tube had an inherent filtration of approximately 3 mm Al.

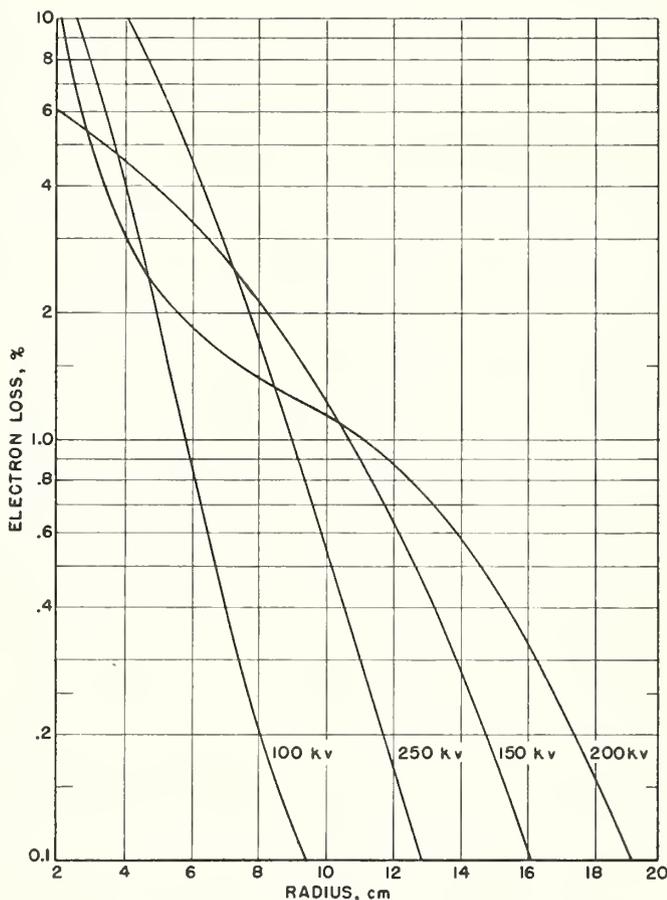


FIGURE 9. Loss of electron-produced ionization (in percentage of total electron ionization) beyond different radii from a zero-diameter beam of constant potential X-rays.

The X-ray potentials were 100 kv with 0.53 mm Pb added filter, 150 kv with 1.53 mm Sn+4.0 mm Cu, 200 kv with 0.7 mm Pb+4.0 mm Sn+0.59 mm Cu, and 250 kv with 2.7 mm Pb+1.0 mm Sn+0.59 mm Cu.

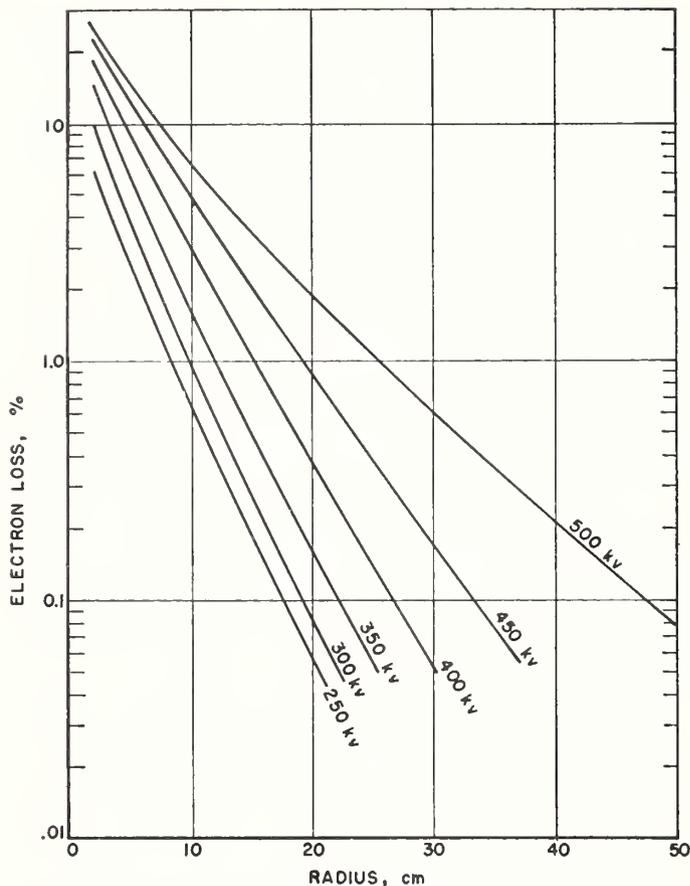


FIGURE 10. Loss of electron-produced ionization (in percentage of total electron ionization) beyond different radii from zero-diameter X-ray beams of the constant potentials indicated.

The inherent filtration of the beam was approximately 3 mm Cu.

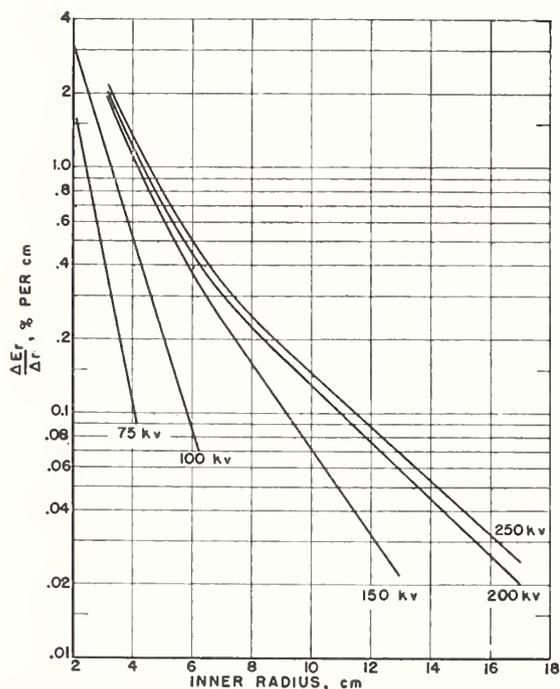


FIGURE 11. Loss of electron-produced ionization per centimeter for the indicated constant potential X-ray beams and the filtrations given in figure 8.

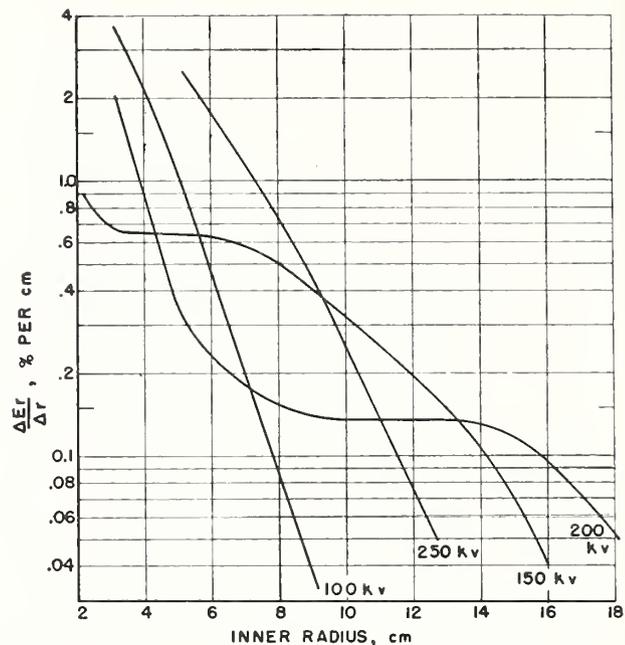


FIGURE 12. Loss of electron-produced ionization per centimeter for the indicated constant potential X-ray beams and the filtrations given in figure 9.

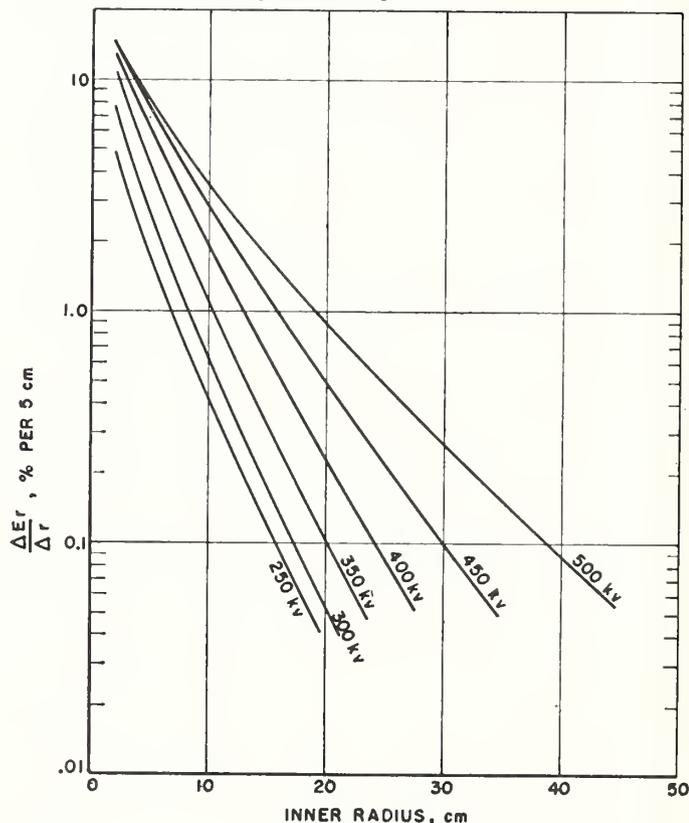


FIGURE 13. Loss of electron-produced ionization per 5 cm for the indicated constant potential X-ray beams and an inherent filtration of approximately 3 mm Cu.

2.5. Aperture-to-Collector Distance

The distance, l (fig. 2), between the aperture and the front edge of the collector, must be sufficient for electronic equilibrium. Figure 14 shows the minimum distance required for electronic equilibrium at different tube potentials with about 3 mm of copper filtration, as determined experimentally [5]. Also shown is the distance

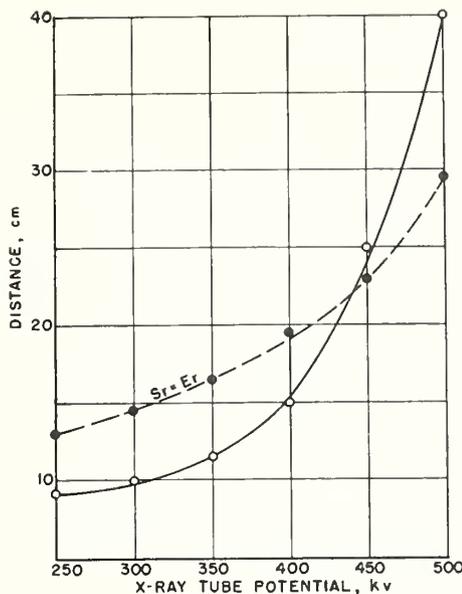


FIGURE 14. Criteria for electronic equilibrium.

Air distance required for electronic equilibrium (solid curve) and radius at which primary electron-produced ionization loss, E_r , is just compensated by secondary photon-produced ionization gain, S_r , (dotted curve) for X-rays having approximately 3 mm Cu filtration.

equal to that radius at which the electron ionization loss is just compensated by the secondary photon contribution, $S_r = E_r$, which has been suggested [5] as a working criterion up to 400 kv. It is evident that this criterion is more than adequate in its kv range, but that l should be at least 25 cm with 450 kv and 40 cm with 500 kv.¹¹

2.6. Losses From Ion Recombination

The definition of the roentgen requires that all of the ionization produced by electrons originating in the defined volume be collected. As the collecting potential is increased from a very low value, the ionization current collected increases rapidly, and then tends to level off. However, it does not become completely independent of voltage but rather approaches the full saturation level asymptotically. Hence, it is necessary to establish a practical working criterion for determining the saturation current without going to extremely high collecting potentials. The criterion most frequently encountered in the literature is simply that the voltage be large enough so that the sensitivity of the electrometer system used is insufficient to detect any voltage dependence of the current as the voltage is increased further. This is somewhat indeterminate unless the electrometer current-sensitivity is further specified and the range of voltages given. For present purposes, one might make use of a criterion requiring that a decrease in potential to one-half the operating potential should not decrease the ionization by more than 0.1 percent. This is a rather stringent requirement, and sometimes cannot be achieved with the maximum voltage available.

There is some evidence [26] that a plot of $1/i$

¹¹ Using a distance of 29.5 cm (where $S_r = E_r$) instead of 40 cm would cause an error of approximately 0.3 percent.

(ionization current, i) versus $1/V$ (collecting potential, V) can be extrapolated to the $1/V=0$ axis to obtain the full saturation current. If the data are carried to large enough potentials, the curve will become nearly linear¹² and the extrapolation therefore straightforward. Of course, for greatest accuracy, the free-air chamber should be operated at sufficiently large potentials so that the extrapolation does not exceed a few tenths of 1 percent in ionization current. A gradient of 250 v/cm has been found adequate for dose rates ordinarily encountered (<50 r/min) and with an X-ray beam diameter (at the collector) of about 1.6 cm. Decreasing the beam diameter lowers the voltage required to achieve a given degree of saturation, but of course also decreases the current output of the chamber for a given dose rate.

It will be useful to recognize that if the applied voltage, say V_1 , is large enough so that $V_1/2$ still lies on the final linear portion of the $1/i$ versus $1/V$ plot, then the decrease in $1/i$ observed in passing from $V_1/2$ to V_1 should equal the decrease obtained by extrapolation from V_1 to $V=\infty$. Thus the alternative criterion (requiring that halving the voltage should not decrease the current by more than 0.1 percent) places one within 0.1 percent of the full saturation current, which can then be immediately obtained by adding the observed decrease.

2.7. Air Attenuation Corrections

As indicated in the discussion of figure 2, the ionization collected is a measure of the intensity of the beam in the collection region. However, it is usually desired to know the exposure dose rate at the diaphragm. For this computation, it is necessary to know the air absorption in distance d , as has been mentioned previously. Figure 15 shows the percentage of absorption per meter experimentally determined for X-rays of various kilovoltages and with various filtrations.

¹² Some experimental evidence obtained at the Bureau, both with a standard free-air chamber and a cavity-type chamber, seems to verify this.

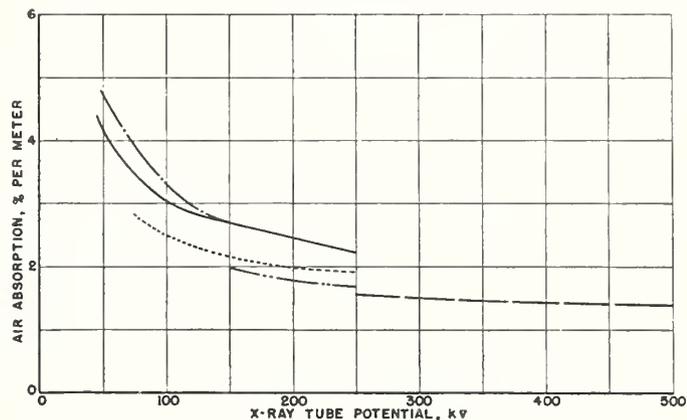


FIGURE 15. Air absorption coefficient for constant potential X-ray beams.

The inherent filtrations are 3 mm Al (dot-dash curve), 1 mm Al (solid curve), 0.25 mm Cu + 1 mm Al (dotted curve), 1 mm Cu + 1 mm Al (dash-dot-dot curve), and a total filtration of approximately 3 mm Cu (dash curve).

2.8. Shielding Against Stray Radiation

A shield (box of fig. 2) around the electrode system is used to reduce stray radiation to a negligible amount compared to the radiation admitted through the aperture. While the radiation beam that strikes the front of this box is generally much smaller than the area of the front face, it is usual to assume that the entire collecting volume (plate spacing times collector height times collector width in a parallel-plate chamber)¹³ can produce ions from this stray radiation. This ionization must be a negligible amount, that is, not more than 0.1 percent of the radiation admitted through the aperture. Thus the transmission allowed by the shielding in the front of the box must not exceed 0.001 times the aperture area divided by the product of the plate height and the plate spacing. Lead, because it readily absorbs this radiation, is usually used for shielding. Figure 16 [27] gives transmission curves for the X-ray potentials of interest here. The sides and back of the box may also require some shielding. It is difficult to say what amount of scattered radiation might strike these surfaces, but if no other data are available, one may assume a thickness requirement equal to one-quarter of that for the front face.

¹³ For a cylindrical chamber, the collecting area is essentially the cross-sectional area enclosed by the outer electrode.

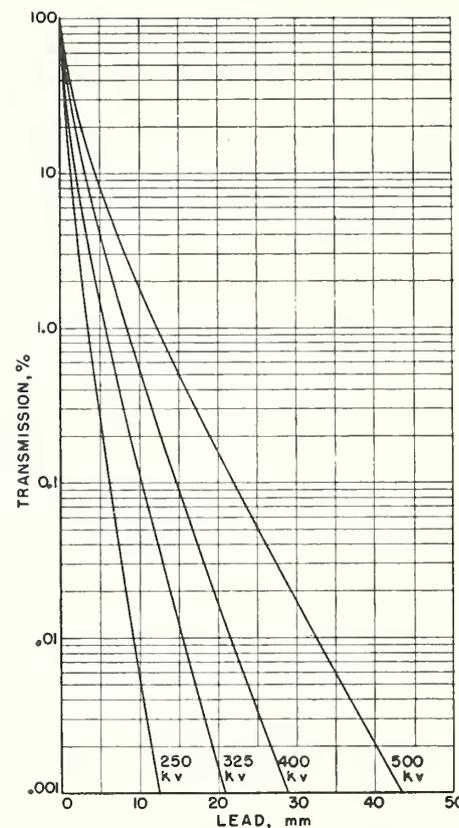


FIGURE 16. Broad-beam transmission curves in lead for constant potential X-ray beams with an inherent filtration of approximately 3 mm Cu.

3. Current or Charge Measurement

Several different types of electrometers have been used to measure the ionization current or charge, but each must be connected and operated in such a way as to maintain the collector electrode at very nearly the same potential¹⁴ (usually ground) as the guard plates [25]. As pointed out earlier, this requirement on the potential is necessary to reduce otherwise excessive field distortion near the gap between the collector and guard plates. Thus the electrometer itself must be sufficiently sensitive to indicate significant differences of potential (~ 0.1 v) between the collector and guards.¹⁵ Most types of electrometers fulfill this requirement.

Figure 17 shows two electrometer circuits used for free-air chamber ionization measurements¹⁶ [31]. Both employ the electrometer as a null detector, making a voltage calibration of its scale unnecessary, and eliminating the collection of charge on the stray capacitance of the system.

¹⁴ Some electrometers, such as the vibrating-reed type made by Applied Physics Corp. of Pasadena, Calif., maintain the potential of the collector plate within a few millivolts of zero potential, regardless of the indication of the meter on the electrometer.

¹⁵ A difference in potential of 0.2 v between collector and guard plates in the present NBS standard chamber has been observed to produce an error in the measured ionization of about 0.1 percent when the collecting field was of the order of 100 v/cm.

¹⁶ The potentiometer connection to ground shown in figures 17a and 17b should be connected instead to the "G" terminal on the electrometer if the vibrating-reed type of the Applied Physics Corporation is used. The internal capacitor C_0 of this electrometer does not enter into the calculation of current for either of these circuit arrangements.

The electrometer need only be sensitive enough to allow the compensating voltage to be adjusted with the required accuracy (< 0.1 percent) and to fulfill the aforementioned field distortion requirement.

The resistor method shown in figure 17a continuously measures the instantaneous current

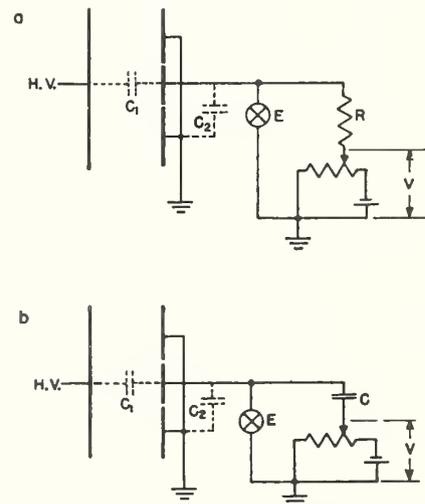


FIGURE 17. Electrical circuits for measuring the ionization current while maintaining the collector electrode near zero potential during X-ray exposure by means of the electrometer, E.

from the chamber. This current produces a potential drop across a known resistance, and this IR drop is balanced by a variable voltage source. In order to obtain the required accuracy, it is convenient to measure the balancing potential with a potentiometer. For low balancing voltages, the potentiometer itself may serve as the voltage source. Note that the ionization current passes through the variable voltage source on its way to ground; hence the voltage source must have a very small resistance compared to that of the calibrated resistor.

Most resistors now commercially available have an appreciable voltage coefficient of resistance [28] and sometimes have erratic behavior for potentials greater than 1 v. Therefore it is wise to use several different resistors and a small

voltage range for each, rather than a single resistor and a large range of voltage to cover the desired ionization current range. Figure 18 shows the design of a convenient resistor "turret" for housing and switching a series of 12 resistors. While this turret was designed for use with a particular electrometer, the design may be adapted readily to other types. With such a turret, it is possible to cover adequately 5 decades of current and still keep the voltage across the resistor to the order of 1 v. Even the best of commercial resistors now available in the 10^8 - to 10^{12} -ohm range sometimes show erratic changes in resistance of as much as 1 percent [28]. While these can be calibrated to the order of 0.1 percent, frequent checking is required to maintain that accuracy. Thus this method of measuring the ionization current leaves something to be desired.

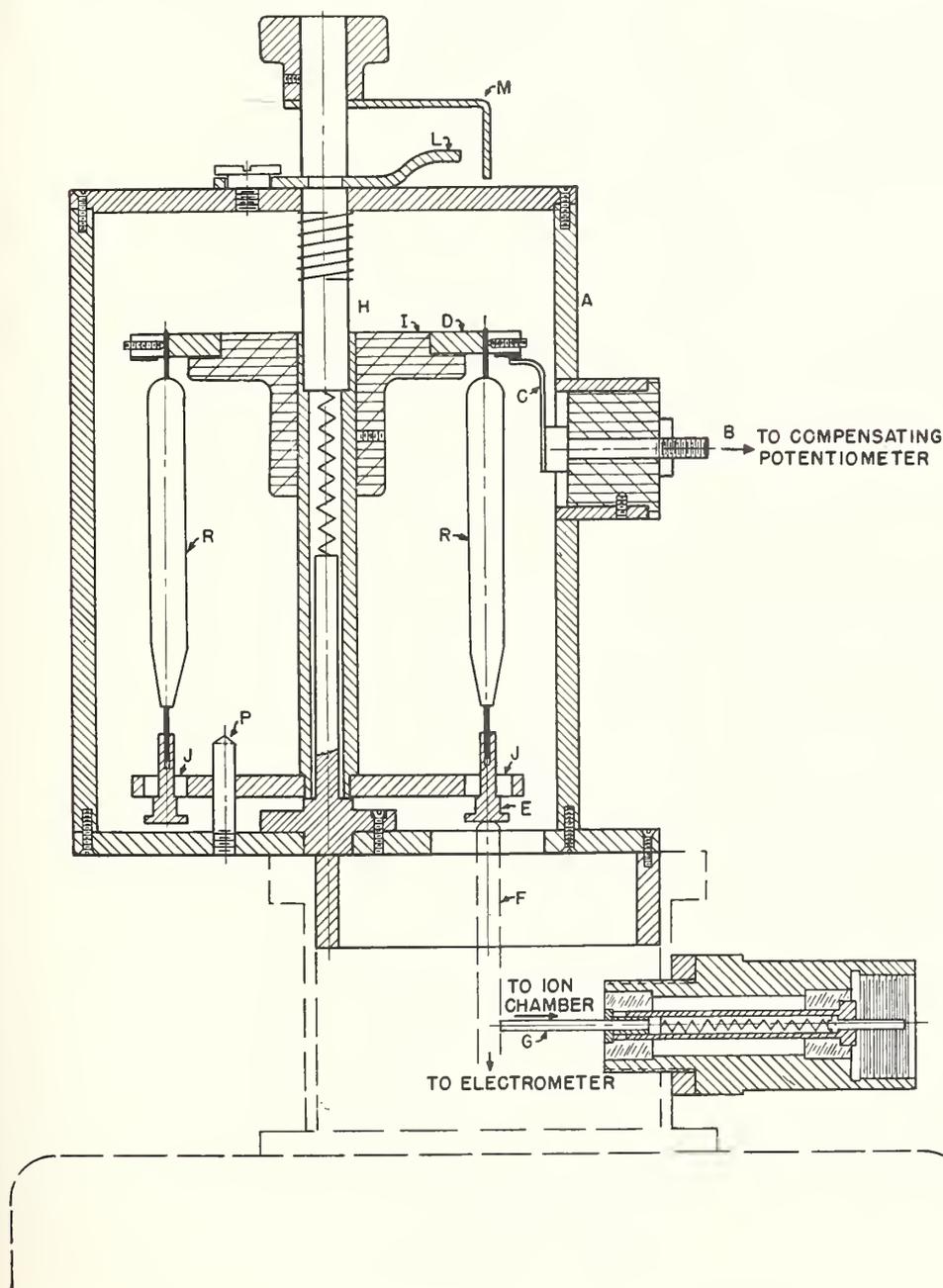


FIGURE 18. Sectional view of resistor turret.

Resistors R are mounted on a rotor which can be lifted and turned to any one of 12 positions. The resistor assembly is housed in a grounded metal case, A. One electrical lead, B, connects to one end of each of the resistors, R, by means of a spring, C, and metal ring, D. The latter is insulated by I from the grounded metal axle, H. The other end of each resistor is insulated by J from the bottom plate. In order to switch to another resistor, the arm, L, is moved aside so that the resistor assembly can move upward in the housing, A, until the bottom plate clears the fixed pin, P. The resistor assembly is then rotated until the arm, M, is over the proper resistor marking on A and the assembly is pushed down so that P goes through the proper hole in the bottom plate. The assembly is locked in place by L. Connection to the resistor is made through the spring-loaded pin, F.

Figure 17b shows a current-measuring circuit incorporating a charge-compensating capacitor for maintaining the collector at ground potential during a measurement. This method, of course, integrates the current over a measured time interval. Figure 19 shows the design of a modified commercial capacitor which has been found to maintain its calibration to better than 0.1 percent over a number of years. The connections can be modified to fit the electrometer and calibration device with which the capacitor is to be used. In this design the dielectric is air, so it is possible to calibrate such capacitors on an a-c bridge (operating at, say, 1,000 cps), which is usually more convenient than a d-c calibration. Such capacitors can be calibrated readily to better than 0.1 percent and are easily removed from the electrometer for recalibration when necessary. Capacitors are not limited to such small voltages as the

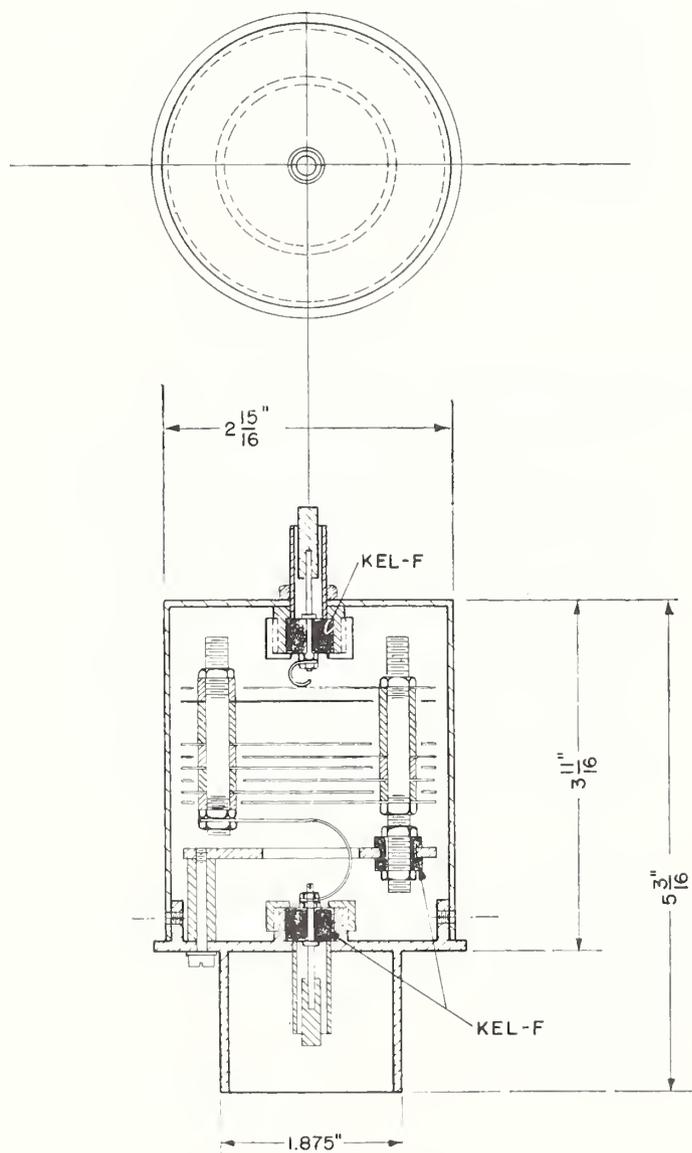


FIGURE 19. Sectional view of capacitor for charge measurement.

The case is to be grounded during use.

resistors. Potentials of over 200 v have been used with impunity.

The high-voltage supply used for the collecting potential must be sufficiently stable to have a negligible effect on the current measurements made with the electrometer. The severity of this requirement can be judged by referring to figure 17. C_1 represents the capacitance between the collector and the high-voltage plate. For a parallel-plate arrangement, if the collecting plate has an area of a cm² and is separated from the high-voltage plate by a distance S cm, the capacitance between the two is approximately $0.0885 (a/S) \mu\mu\text{f}$. Thus for a equal to 200 cm² and S equal to 20 cm, the capacitance C_1 will be of the order of $1 \mu\mu\text{f}$. C_2 represents the stray capacitance from the collector and its connections (including the electrometer) to ground. For purposes of illustration this can be taken as $100 \mu\mu\text{f}$, although its value will depend strongly upon the individual arrangement of conductors. If the collecting potential suddenly fluctuates by an amount ΔV_H , the collecting electrode in figure 17a will vary in potential by an amount

$$\Delta V_c = \frac{C_1}{C_1 + C_2} \Delta V_H \quad (3)$$

or about $0.01 \Delta V_H$ in the example given above. Thus, if ΔV_H is 1 v, the electrometer will indicate a fluctuation of about 0.01 v. For the usual balancing potentials used in the resistor method (≈ 1 v), this amounts to a 1 percent fluctuation. If such fluctuations occur frequently during a measurement, it becomes very difficult to adjust V to the proper value within 0.1 percent, because the null position of the electrometer is not well defined. As the collecting potential usually amounts to several thousand volts, the requirement that it be stable to better than 1 v is very severe.

The charge-balancing method shown in figure 17b is somewhat less restrictive on the collecting potential, because C_2 is increased by the addition of the compensating capacitor C . This usually has a value of several hundred $\mu\mu\text{f}$, so that ΔV_c is of the order of $0.001 \Delta V_H$. This may be considered as another advantage of the capacitor method over the resistor method. Power supplies sufficiently stable to fulfill this requirement are available commercially. An alternative method for compensating for the voltage fluctuations rather than eliminating them has also been described by De Bitetta [29].

Because of the small order of magnitude of the currents involved, only the highest-quality insulating materials are suitable for supporting the collector electrode and insulating the connecting lead to the electrometer. The insulator surfaces must be dust-free and perfectly clean to avoid leakage troubles. Amber, polystyrene, polyethylene, Teflon, and "Kel-F" are all good, although the polyethylene is too flexible for use in rigidly supporting the collector. Polystyrene is usually cleaned by machining but the other materials may be cleaned with absolute alcohol.

4. Examples

In order to test the over-all adequacy of the data just presented and to provide a guide for their use, it is worth while to use these data in determining the corrections to be applied to two chambers, and to compare these corrections with the experimental comparison between them. For such a comparison, it is well to consider the intercomparison of two very dissimilar chambers so that their corrections are very different. The calibration of the Swedish portable chamber with the NBS standard [7] will be used as an example. Figures 20 and 21 show sectional plan views of the Swedish and NBS chambers respectively.

The dose rate of an X-ray beam for saturation conditions may be computed from the equation

$$\frac{r}{\text{sec}} = \left(\frac{3 \times 10^9}{AL} \right) \left(\frac{T}{273.2} \right) \left(\frac{760}{P - 0.238 P_1} \right) \left(\frac{100}{100 + K} \right) \left(\frac{100}{100 + f} \right) \left(C \frac{\Delta V}{\Delta t} \right) e^{+\mu d}, \quad (4)$$

where

A is the area of the defining aperture (in cm^2),

L is the collector length from gap-center to gap-center (in cm),

T is the temperature of the air in the collection region (in $^\circ\text{K}$),

P is the pressure of the air in the collection region (in mm Hg),

P_1 is the pressure of water vapor in the collection region (in mm Hg),

K is the algebraic sum of the deficiency of electron and excess of secondary ionization collection (in percentage of total electron ionization),

μ is the air absorption coefficient (in m^{-1}),

d is the distance from the plane of definition in the aperture to the center of the collecting region (in m),

f is the percentage gain of ionization due to field distortion,

C is the capacitance upon which the charge is collected (farad),

$\Delta V/\Delta t$ is the rate of change of the balancing potential on the charge-measuring instrument (v/sec).

The two chambers were set up on a track perpendicular to the beam so that each could be moved in turn into the fixed X-ray beam. The limiting aperture of each was at the same distance from the target (approximately 1.5 m). Each chamber was aligned visually and fluoroscopically. The temperature of the air in the boxes was measured to the nearest 0.1°C during the intercomparison.¹⁷ The air and vapor pressure were assumed to be the same during the measurement because both of the boxes were open to the room air. Independent comparisons were made of the capacitances, C , by a charging method, and of the

¹⁷ Differences of as much as 0.5°C were observed between the temperatures of the air in the two boxes at the same time.

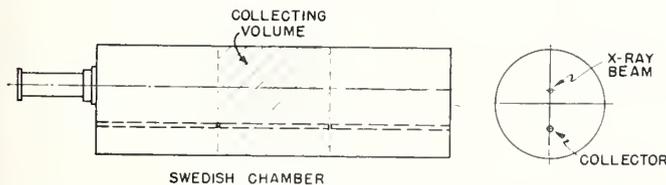


FIGURE 20. Sectional view of Swedish portable free-air chamber.

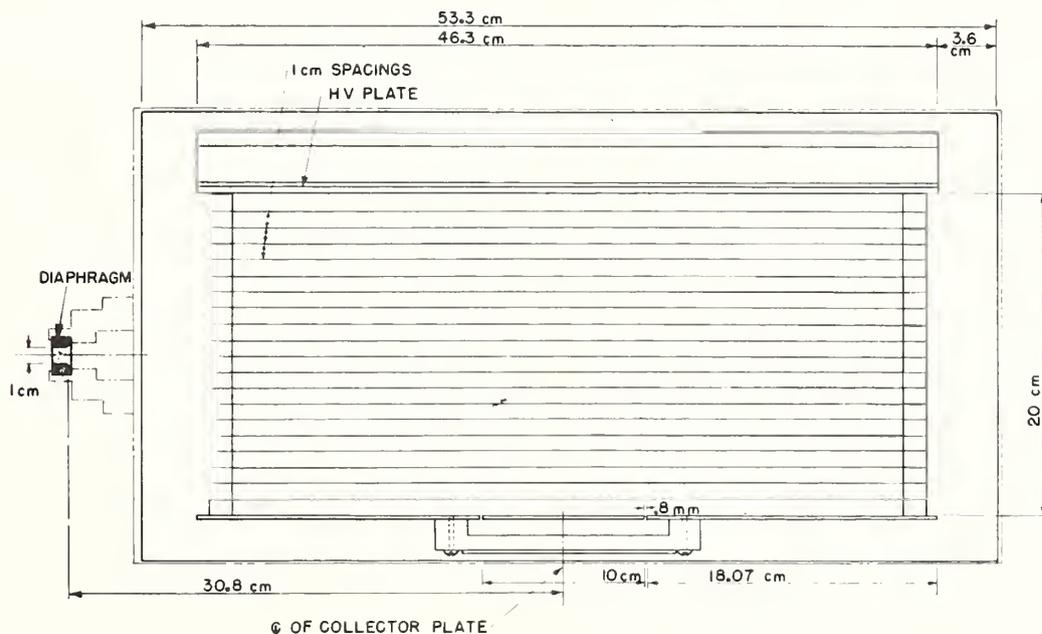


FIGURE 21. Sectional view of NBS free-air chamber.
The collector plate height is 26.8 cm.

diaphragm areas, A , by an ionization method with one of the chambers. The ratio of C 's agreed to within 0.02 percent with the ratio of the value certified by the Swedish laboratory and by the Bureau. The ratio of A 's could not be compared with the areas computed from the measured diameters because of a slight coning of the Swedish diaphragm. Therefore the ionization ratio was accepted. The potentiometers used for measurement of ΔV were intercompared and agreed to within 0.05 percent throughout their range. The values of f for the two chambers were determined independently by ionization means and found to be 0.0 percent for both the NBS and Swedish chambers. The collecting potential for each chamber was sufficient for saturation within 0.1 percent.

The ratio of the calculated dose rates for the two instruments is

$$\frac{(r/\text{sec})_S}{(r/\text{sec})_B} = \left(\frac{A_B L_B}{A_S L_S} \right) \left(\frac{T_S}{T_B} \right) \left(\frac{100 + K_B}{100 + K_S} \right) \left(\frac{100 + f_B}{100 + f_S} \right) \left(\frac{C'_S (\Delta V / \Delta t)_S}{C'_B (\Delta V / \Delta t)_B} \right) e^{\mu(d_S - d_B)} \quad (5)$$

where the subscript S denotes factors for the Swedish chamber and subscript B factors for the NBS chamber. As

$$\frac{A_B}{A_S} = 1.5678,$$

$$\frac{L_B}{L_S} = 0.5040,$$

$$d_S - d_B = 0.447 - 0.308 = 0.139 \text{ m},$$

$$\frac{C'_S}{C'_B} = 0.20894,$$

$$\frac{100 + f_B}{100 + f_S} = 1.000,$$

this ratio becomes

$$\frac{(r/\text{sec})_S}{(r/\text{sec})_B} = 0.16509 \left[\left(\frac{T_S}{T_B} \right) \left(\frac{100 + K_B}{100 + K_S} \right) \left(\frac{(\Delta V / \Delta t)_S}{(\Delta V / \Delta t)_B} \right) e^{0.139\mu} \right]. \quad (6)$$

It is instructive to consider the detailed analysis for the K of one of the chambers. Such an analysis is facilitated by plotting a vertical section of the collecting region on polar graph paper. The outline of the collection region for the Swedish chamber is shown by the full lines of figure 22. The dash lines show the boundary circles for the computation. It is seen that all of the ionization within a 6.3-cm radius is collected, but also that there is a fraction (fraction of 360°), F , of the ionization collected for larger diameter rings.¹⁸

¹⁸ The ionization lost by electrons striking the collector is very small, but can be taken into account by discounting the volume lying behind that electrode.

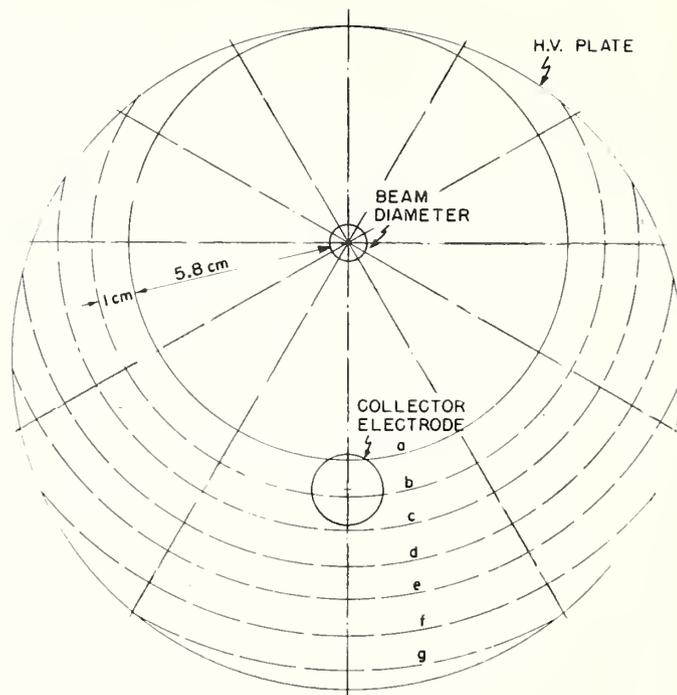


FIGURE 22. Section through collection region of the Swedish chamber.

The loss of electron-produced ionization, E_r , for 5.8 cm is obtained¹⁹ from figure 8, and the gain of secondary photon ionization from table 2. Table 3 shows the numbers obtained for 250-kv X-rays. It is seen that there is an over-all loss of electron ionization of 1.19 percent and a gain of secondary photon ionization of 0.20 percent. Thus the chamber gives about 0.99 percent too low an ionization collection at 250 kv. Table 4 summarizes the computation of K for the two chambers at the different X-ray potentials and filtrations. It is seen that the values vary slowly with potential so an interpolation is feasible. Table 5 shows the values of $(r/\text{sec})_S / (r/\text{sec})_B$ obtained from the experimental data with corrections applied as indicated.

TABLE 3. Analysis of ionization excess in Swedish chamber for 250-kv X-rays

Radii		F	E_r	FE_r	Radii		F	S_r	FS_r
Inner	Outer				Inner	Outer			
cm	cm				cm	cm			
0	5.8	1	-2.15	-2.15	0	5	1	0.09	+0.09
5.8	6.8	0.8	0.55	+0.44	5	10	0.7	.12	+ .08
6.8	7.8	.6	.37	+ .22	10	12.3	.2	.15	+ .03
7.8	8.8	.5	.28	+ .14					
8.8	9.8	.4	.20	+ .08					+0.20
9.8	10.8	.3	.18	+ .05					
10.8	11.8	.2	.12	+ .02					
11.8	12.3	.1	.10	+ .01					
				-1.19					

$K = FE_r + FS_r$
 $K = -1.19 + 0.20$
 $K = -0.99$

¹⁹ The beam radius of 0.5 cm must be subtracted from the radii of figure 22 when determining values of E_r and S_r .

TABLE 4. Values of K

Chamber	60 kv	75 kv	100 kv	150 kv	200 kv	250 kv
Swedish.....	+0.32	+0.32	+0.20	-0.30	-0.74	-0.99
NBS.....	+0.44	+0.44	+0.42	+0.23	-0.04	-0.20

5. Accuracy of Free-Air Chamber Measurements

One can estimate the accuracy with which a measurement with a free-air chamber can be made by estimating the accuracy of each of the factors involved. Table 6 shows the estimates given by the International Commission on Radiological Units and Measurements [8]. If all of these factors act in the same direction, it is possible for an error of as much as ± 1.1 percent to exist in a measurement. This is an unlikely occurrence, however, and the probable limit of the error is about ± 0.5 percent.

The ICRU has also estimated that two free-air chambers when checked against each other should certainly agree to within 0.9 percent, and that when two chambers are compared through an intermediary free-air chamber, one should expect an agreement to within 1.2 percent maximum. The Swedish-NBS calibration just described falls well within the 0.9 percent. The Swedish chamber has also been calibrated at Frankfort [30] and the proper corrections applied [17]. While the NBS Frankfort calibrations were not performed at exactly the same radiation quality, there seems to be an agreement of within 0.5 percent. Thus, the absolute determination of the roentgen can

TABLE 5. Comparison of free-air chambers, $(r/sec)_S/(r/sec)_B$

60 kv	75 kv	100 kv	150 kv	200 kv	250 kv
0.995	0.992	0.995	0.994	0.996	0.997

probably be made to an accuracy somewhat better than that given in table 6.

TABLE 6. Experimental maximum error for quality range of 50 to 500 kv (constant potential), and filtrations of 2 mm Al and 3 mm Cu respectively

Experimental factor	Estimated maximum error
	<i>Percent</i>
Charge (assumed measured by accurate potentiometer and capacitor using electrometer as a null detector).....	± 0.1
Air volume (includes errors in diaphragm area, collecting plate alignment, and field distortion resulting from other causes).....	± 3
Air density (includes pressure and temperature measurements).....	± 1
Humidity of the air (includes measurement of the humidity and the effect of a slightly different value of the average energy per ion pair (W) for water vapor).....	± 1
Saturation of ion collection.....	± 1
Scattering of X-rays by air in the chamber.....	± 1
Inadequacy of plate separation for electron path length.....	± 1
Air attenuation between diaphragm and collector.....	± 2

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WASHINGTON, May 14, 1957.

Design of Free-Air Ionization Chambers for the Soft X-Ray Region (20-100 kv)

VICTOR H. RITZ, B.S.

THE ROENTGEN HAS been recommended (1) by the International Commission on Radiological Units and Measurements as the unit of exposure dose. A measurement with a free-air ionization chamber is the most convenient way of accurately determining exposure dose in the soft x-ray region. Design criteria for standard free-air chambers have been summarized by Wy-

was undertaken to provide design criteria for 20 to 100-kv radiation with filtration ranging from 2 mm. of beryllium to 2 mm. of beryllium plus 4 mm. of aluminum.

A typical free-air ionization chamber is shown schematically in Figure 1. The roentgen is defined in terms of the ionization produced by the interaction of the x-ray beam with a specified mass of air (1).

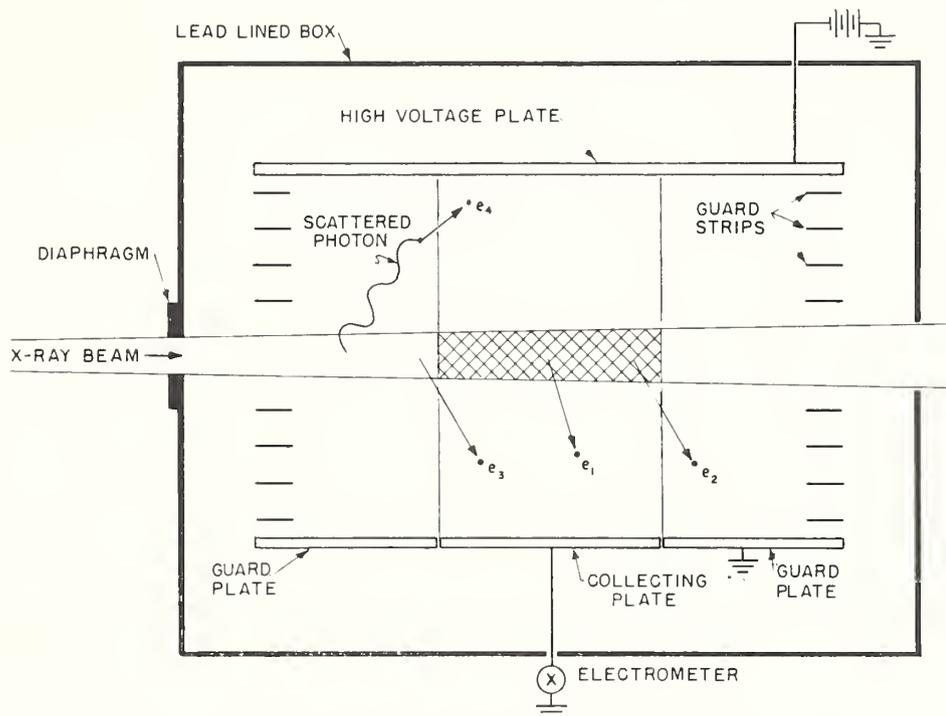


Fig. 1. Schematic top view of a typical free-air ionization chamber.

ckoff and Attix (2) in National Bureau of Standards *Handbook 64* for moderately and heavily filtered x-rays generated at potentials from 50 to 500 kvcp. Agreement to about 0.5 per cent has been reached in international intercomparisons of the roentgen in this energy region. In intercomparisons involving lightly filtered low-energy x-rays, however, differences of 1 per cent or more have been observed (3, 4). The present experiment

In practice, however, one defines a volume represented in Figure 1 by the cross hatched region. The cross-sectional area of the volume is determined by the diaphragm, and the length of the collecting region by the length of the collector and by the electric field between the high-voltage and collecting plates. The presence of the grounded lead box tends to distort the electric field in the collecting region, but guard plates and guard strips can be used

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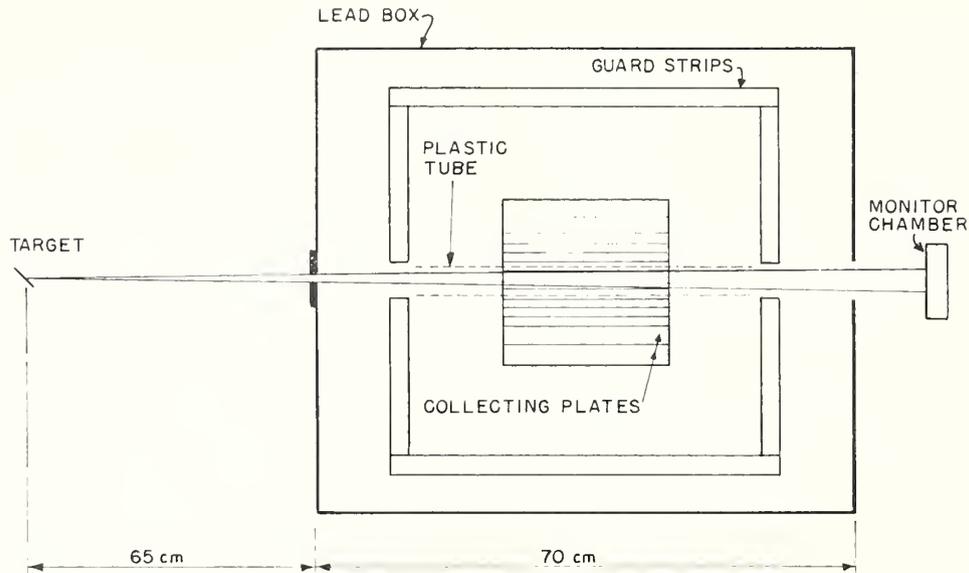


Fig. 2. Side elevation of the experimental set-up.

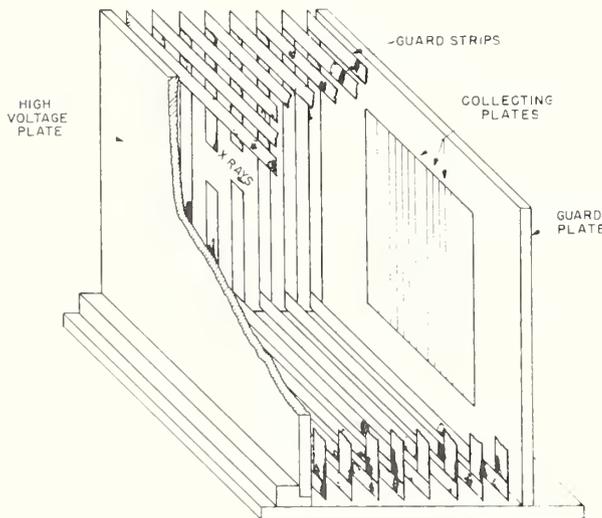


Fig. 3. View of experimental free-air chamber with collectors in position for the field distortion study.

to eliminate such distortion. The guard strips are uniformly spaced between the ground and high-voltage plates and are parallel to them. The potentials of the strips are fixed by a resistor network to give a linear potential gradient between the plates. Distortion can also be caused by the guard strips themselves. This effect becomes important when the strips are close to the edge of the collector or when the centerline-to-centerline spacing between adjacent strips is large.

The x-ray beam interacts with the air to produce electrons like e_1 , which produce

ionization in the collecting region. The precise determination of the roentgen requires that such electrons expend their energy in the air before striking the high-voltage or collecting plates. Thus the proper design of a free-air chamber requires a knowledge of the range of these primary electrons. Electrons like e_2 cause ionization outside the collecting region, but under conditions of electronic equilibrium this loss is compensated by electrons like e_3 . Conditions may also exist in which a photon is scattered out of the primary beam and interacts to produce an electron like e_4 . This represents a contribution that is not included in the definition of the roentgen. This scattered photon contribution must be determined and subtracted from the total ionization measured by the chamber.

Another correction to be applied to a free-air chamber is for the attenuation of the x-ray beam by the air between the diaphragm and the center of the collector. This correction becomes quite large in the soft x-ray region and is, in fact, the largest single correction to be applied to the free-air chamber. The air attenuation correction can be reduced by decreasing the length of the air path between the diaphragm and collector. This, however, increases the distortion caused by the guard-strip system. In practice the reduction

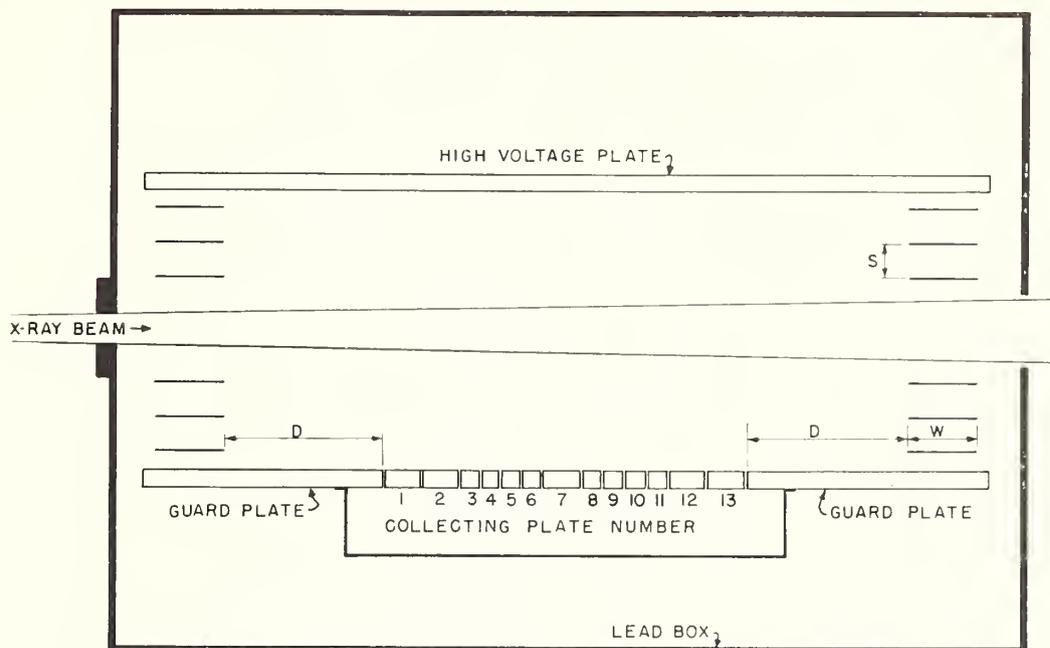


Fig. 4. Section through the chamber, viewed from above, as used in the field-distortion study. The collecting strips number 1, 2, 7, 12, and 13 were 0.5 in. wide, the remaining strips were 0.25 in. wide. D is the distance from the edge of the collecting plate assembly to the guard strip system; S is the centerline-to-centerline spacing of the guard strips. W is the depth of the guard strips.

in the length of air path is limited by the field distortion.

This paper will discuss: (a) the distortion of the electric field in the collecting region by the guard-strip system, (b) the range of primary electrons like e_1 and the scattered photon contribution, and (c) other corrections, such as air attenuation, which are particularly important in the soft x-ray region.

APPARATUS

A side elevation view of the experimental arrangement is shown in Figure 2. A tungsten-target x-ray tube with a beryllium window 2 mm. thick was operated at exciting potentials of 20 to 100 kvcp. The kilovoltage was monitored manually throughout the experiment, and errors because of fluctuations in output were avoided by the use of a monitor chamber placed behind the experimental chamber.

The experimental free-air chamber is shown in Figures 2-4. The thirteen aluminum collecting plates were insulated from each other by narrow air gaps (0.125 to 0.25 mm. wide) and from ground by a polyethylene insulator. They could be grounded or connected to the charge-

measuring system by means of low-noise coaxial cables that passed through the lead box to a terminal box. The collecting plate assembly was machined as a unit until it was flat, to about 0.0025 mm., to avoid field-distortion effects due to the collectors themselves (2). A thin coat of colloidal graphite was put on the collectors to eliminate contact potentials (2). The collecting plates could be turned so that their long axes were vertical for the field-distortion study (Figs. 3 and 4) or horizontal for the electron-range and photon-contribution studies (Fig. 2). Vibrating reed electrometers were used as null detectors (2) to measure the ionization currents in the experimental chamber and monitor.

The guard-strip system shown in Figures 3 and 4 consisted of brass strips separated by Bakelite spacers, surfaces of which were made conducting with colloidal graphite and then scribed with a fine line to insulate adjacent guard strips. This minimized the amount of exposed insulator that could be seen by the collecting plates and eliminated distortion of the electric field by the insulators. The horizontal guard strips were kept fixed in position. The vertical guard strips were movable so that the dis-

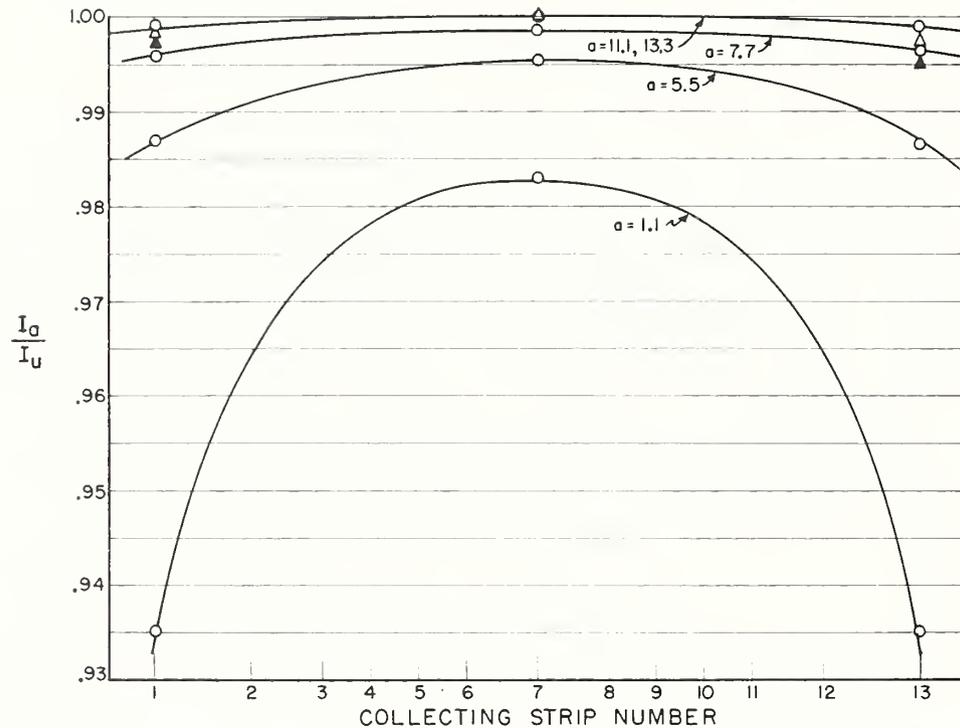


Fig. 5. Field distortion across a collecting plate 11.8 cm. wide. I_a is the ionization measured for a particular value of $a = D/S$. I_u is the ionization measured when the field is undistorted. ○ data of this experiment at 50 kv with inherent filtration and 100 kv with 4 mm. aluminum filtration. ▲ data of Roth (7) at 50 kv, 0.24 mm. Al h.v.l. △ data of Roth at 200 kv, 0.56 mm. Cu h.v.l.

tance between them and the collecting plates could be varied to investigate field distortion.

The ionization distribution of the scattered photon contribution was obtained with the aid of a polystyrene tube, 2 cm. inside diameter, 0.22 mm. thick. This wall thickness was greater than the maximum electron range so that ionization from electrons generated by the primary x-rays could not be produced outside of the tube. The tube was made conducting with a coating of colloidal graphite. Scribed lines through the graphite along the length of the tube insulated portions of it from each other and permitted them to be electrically connected to the guard strips so as to reduce the field distortion caused by the tube itself (8).

FIELD DISTORTION

A section through the experimental free-air chamber, viewed from above, is shown in Figure 4. The thirteen collecting plates

were used to study the ionization at different points along the x-ray beam. The change in ionization for different guard-strip configurations was a measure of the change of field distortion caused by the guard strips. The method used was similar to that described earlier by Failla (5). In general, field distortion can be caused by the grounded lead box and by the guard strips themselves. The guard-strip system used in this experiment completely shielded the collecting region from the box. No change in the ionization current was measurable when the potential of the box was changed from ground to that of the high-voltage plate. The distortion studied was thus due only to the guard strips themselves. Two sets of guard strips were used: one with S , the centerline-to-centerline spacing of the strips, equal to 0.9 cm. and a strip thickness of 0.16 cm.; the other with S equal to 0.45 cm. and a strip thickness of 0.08 cm. The distance between the guard strips and the collecting or high-voltage

plates was $S/2$ for the strips closest to the plates. The potential across these strips was half that applied to the strips with spacing S . W was kept fixed at 1.6 cm. D , the distance between the edge of the collector and the guard strips, was varied from 1 to 12 cm. The distance between the high-voltage and collecting plates was 12.5 cm. There were 10-cm.-wide guard plates above and below the collectors and 14-cm.-wide guard plates at the front and back of the chamber.

Figure 5 shows the results of the field distortion study. The distortion was found to depend on $a = D/S$. I_a is the ionization measured for a particular value of a . I_u , the ionization measured when the field is undistorted, was determined by removing the guard-strip assembly and averaging the ionization measured with the lead box at ground potential and at the potential of the high-voltage plate. The average corresponds to a measurement made with a field undistorted by the box (2). A ratio of I_a/I_u equal to 1 in Figure 5 indicates an undistorted field. This ratio is plotted as a function of the collecting strip number.

Since the total width of collecting strips 1 to 13 was 11.8 cm., Figure 5 represents the field distortion at different points across an 11.8-cm.-wide collector. This figure can be used to average the distortion and obtain ratios of I_a/I_u across an 11.8-cm. collector of 0.966, 0.992, 0.998, and 1.00 for values of a equal to 1.1, 5.5, 7.7, and 11.1 (or greater) respectively. The distortion across collecting plate assemblies that are not 11.8 cm. wide cannot be estimated from Figure 5. A collecting plate assembly consisting of several collectors whose combined width was 11.8 cm. could be designed. A narrow collector at the front of the assembly could be used to measure the low-energy, inherent-filtration x-rays, while the wider collectors could measure the high-energy, heavily filtered x-rays. Figure 5 can be used to estimate the distortion for narrow collecting strips if their width is approximately the same as those used in this experiment. For exam-

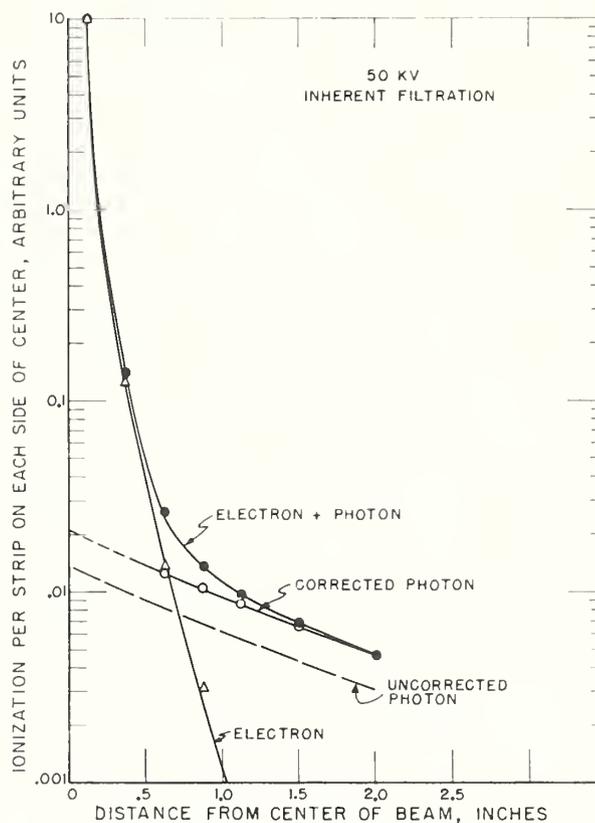


Fig. 6. Method of determining primary electron and photon contributions. ● Electron + photon contribution (tube out). ○ Photon contribution (tube in). △ Primary electron contribution (by subtraction).

ple, a 1.3-cm.-wide collector at the end of the collecting plate assembly would have ratios of I_a/I_u equal to 0.999 and 0.996 for a equal to 11.1 and 7.7.

Data supplied by Roth (6) for a similar experiment, in which a equalled 11, are also shown in Figure 5. Roth found that the field distortion depended upon the energy of the x-ray beam used in the experiment. This effect may have been due to distortion caused by the exit hole for the beam that was cut in the guard-strip system. One would expect exit-hole distortion to be smaller at higher energies since a smaller fraction of the total ionization takes place near the center of the beam. Roth's data at 200 kv are in good agreement with the present experiment, which was done at 50 kv with inherent filtration, and at 100 kv with 4-mm. aluminum filtration. No energy dependence of field distortion was found in this experi-

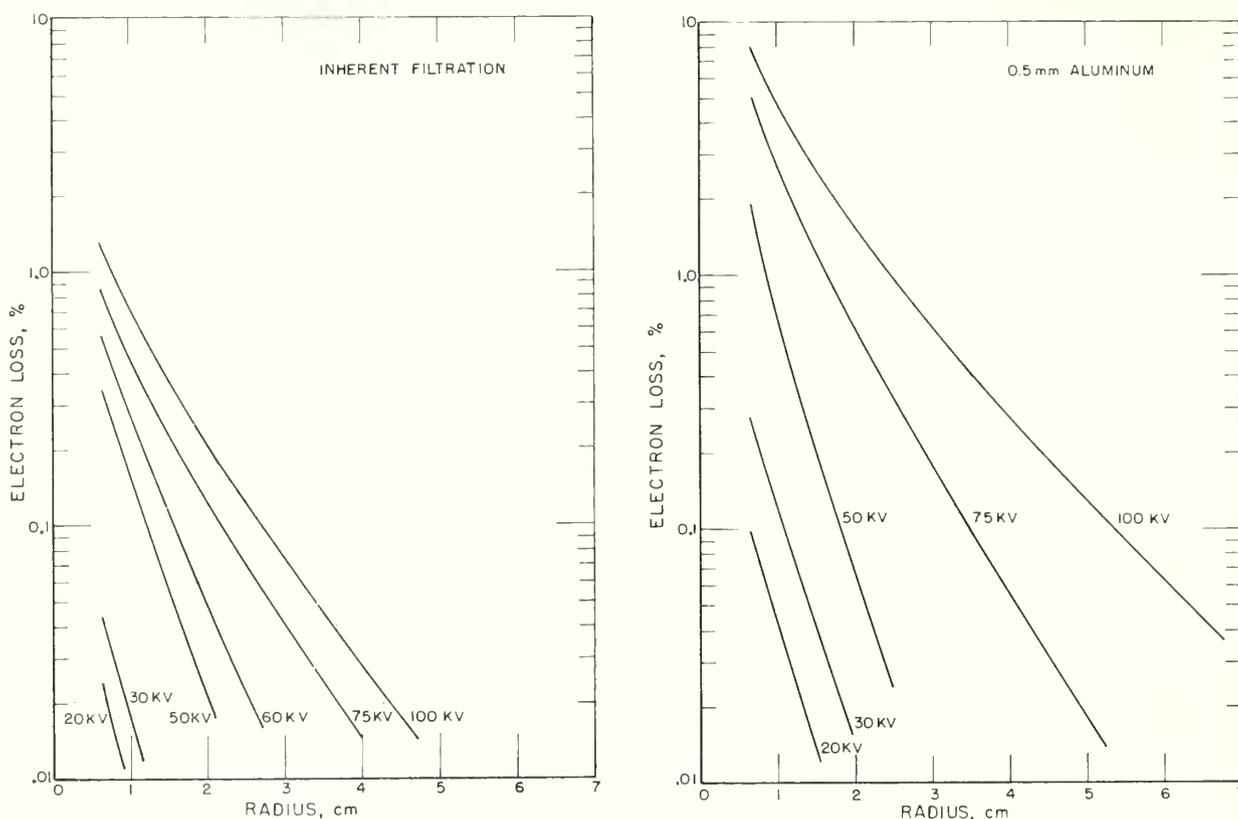


Fig. 7. Loss of electron-produced ionization (in percentage of total electron ionization) beyond different radii from a zero-diameter beam of constant potential x-rays. The filtration was 2 mm. of beryllium (inherent) + 1 meter of air.

Fig. 8. Loss of electron-produced ionization (in percentage of total electron ionization) beyond different radii from a zero-diameter beam of constant potential x-rays. The total filtration was the inherent filtration + 0.5 mm. aluminum added filter + 1 meter of air.

ment, indicating that the exit hole (1.8 cm. square) in the guard-strip system had no effect upon the distortion. This indicates that fairly large holes may be cut in the guard strips without serious distortion. In general, however, the size of the entrance and exit holes should be minimized, though they must be large enough to permit the x-ray beam to pass through the guard strips without striking them.

The primary objective of the field-distortion study was to devise a guard-strip system that minimized the distance from the diaphragm to the collector and caused negligible (<0.1 per cent) field distortion. No attempt was made to vary the height of the vertical guard strips because the chamber could be oversized in the vertical direction without a prohibitive increase in weight. A qualitative discussion of the effect of varying the height of the chamber can be given. The distortion increases

most rapidly at the top and bottom of the collector as the height of the vertical guard strips is decreased. The effect of this distortion on the total ionization measured by the chamber is decreased by the fact that the ionization density is usually small at the top and bottom of the collector. The horizontal guard strips at the top and bottom of the chamber may also interact to change the field distortion caused by the vertical guard strips, but this type of distortion should be slight (2). The heights of Roth's vertical guard strips and those in the present study were 35 and 28.5 cm., respectively. The close agreement between the two experiments indicates that the field distortion is relatively insensitive to changes in the height of the free-air chamber if the height is large. The field distortion might also depend upon the thickness of the guard strips. A check was

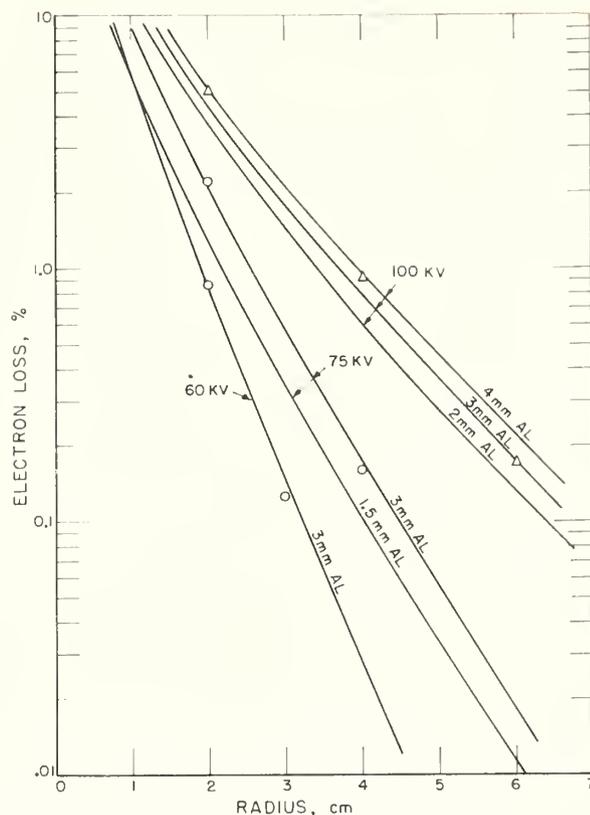
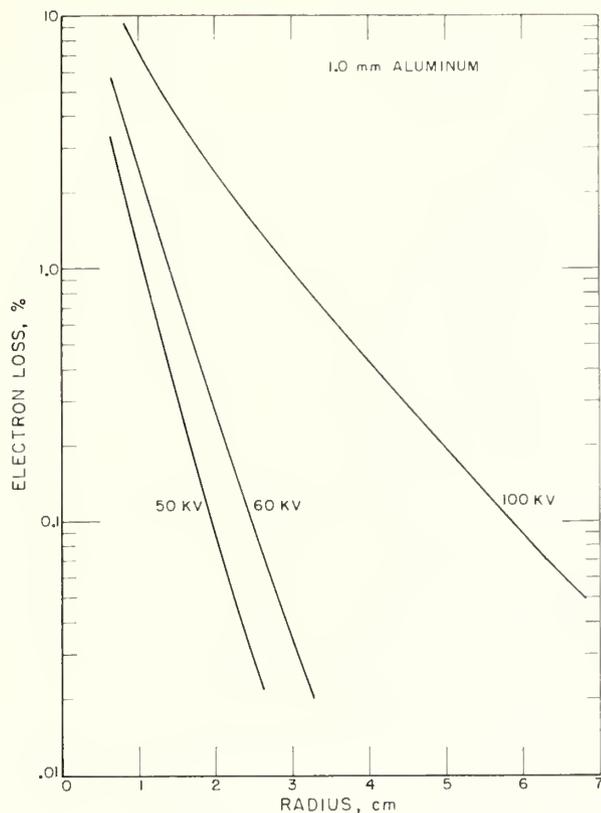


Fig. 9. Loss of electron-produced ionization (in percentage of total electron ionization) beyond different radii from a zero-diameter beam of constant potential x-rays. The total filtration was the inherent filtration + 1.0 mm. aluminum added filter + 1 meter of air.

Fig. 10. Loss of electron-produced ionization (in percentage of total electron ionization) beyond different radii from a zero-diameter beam of constant potential X-rays. \circ Data of Attix and DeLaVergne at 60 kv, 3 mm. Al filtration and 75 kv, 3 mm. Al filtration. \triangle Data of Attix and DeLaVergne at 100 kv, 4 mm. Al filtration.

made with $S = 0.9$ cm. and $D = 5$ cm. by varying the thickness of the strips from 0.16 cm. to 0.9 cm.² No change in the field distortion was observed.

Gross distortions of the electric field can occur if exposed insulators are present in the free-air chamber. In one instance the ionization changed by 25 per cent when an exposed insulator was removed. The exposed insulator surface was minimized in the present experiment and the effect of the insulators separating the guard strips was checked by placing a dummy insulator in the chamber. Small distortions of the

field can occur if the resistors in the voltage-dividing network for the guard strips are not matched properly. Measurements indicate, however, that this effect is negligible if the resistors are matched to ± 5 per cent.

The data taken in the course of the field-distortion study yielded information about the uncertainty in determining the collecting volume of the free-air chamber. The ionization per length of collector was measured under conditions of negligible field distortion from the guard strips and corrected for air attenuation. If the collecting volume was undistorted and if the length of the strip has been measured with sufficient accuracy, each strip should then measure the same ionization/length. Actually, the ionization/length determined experimentally for each strip was found to be within ± 0.2 per cent of the average. Thus the maximum uncertainty in the collecting volume due to field distortion

² A new low-energy (20–100 kv) free-air chamber with guard strips 14 cm. high has been intercompared with the NBS medium-energy (60–250 kv) standard which has guard strips of 27 cm. height. The agreement between the two systems (different capacitors, diaphragms, potentiometers, etc.) was well within the ± 0.5 per cent expected for such an intercomparison (2). This indicates that no gross distortions of the electric field occur when the guard-strip height is reduced to 14 cm. A description of the new low-energy standard and a discussion of the intercomparison will be published shortly.

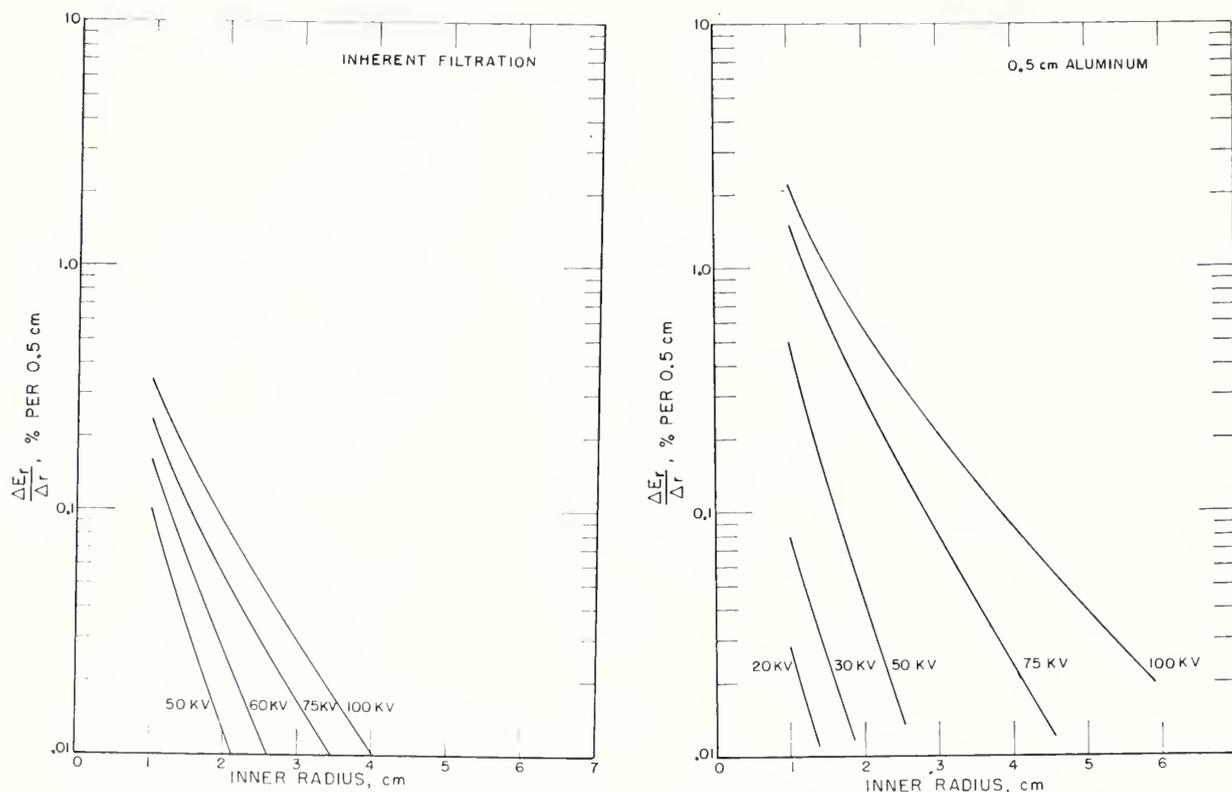


Fig. 11. Loss of electron-produced ionization per 0.5 cm. radius increment for the x-rays of Fig. 7.
 Fig. 12. Loss of electron-produced ionization per 0.5 cm. radius increment for the x-rays of Fig. 8.

caused by the collectors themselves and to difficulties in measuring the length of the collector (6 mm. in this experiment) is about ± 0.2 per cent.

To summarize, certain points are to be borne in mind in the design of a free-air chamber with minimal field distortion. Distortion caused by the lead box can be estimated from *Handbook 64*, while Figure 5 can be used to determine the distortion caused by the guard-strip system. This distortion will be independent of the thickness of the guard strips and relatively insensitive to changes in the height of the guard strips. Distortion caused by lack of planarity of the collector and its guards will be negligible if the collector and guards are machined flat, to about 0.0025 mm. per centimeter of collecting plate width (2). The size of the entrance and exit holes cut in the guard-strip system for the x-ray beam should be as small as possible. The amount of exposed insulating material in the chamber must be minimized, and the effect of any remaining exposed insulator should be determined.

ELECTRON AND SCATTERED PHOTON CORRECTIONS

Figure 2 shows a side view of the arrangement used to determine the electron distributions and the scattered photon contributions. The experimental procedure was similar to that used by previous workers (7, 8). The collecting plates were turned so that their long axes were parallel to the x-ray beam. The ionization was first sampled by the different collectors, with the plastic tube in the chamber. The tube was aligned coaxially with the beam. The ionization measured with the tube in place was due only to the scattered photons. The tube was then removed. The ionization measured under the second condition was the sum of the primary electron and scattered photon contributions. The primary electron distribution could then be determined by subtracting the photon contribution measured previously. A typical example is shown in Figure 6. The photon curve was corrected for attenuation in the walls of the tube and for the non-air

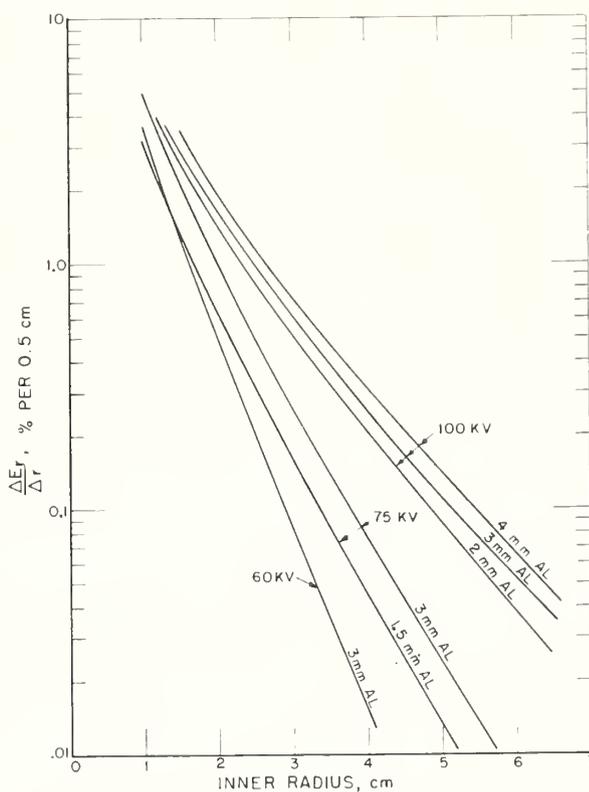
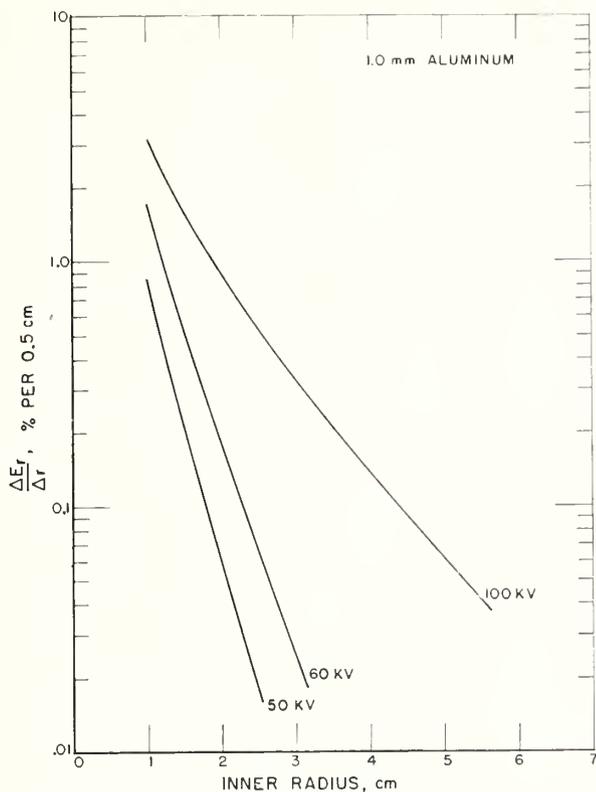


Fig. 13. Loss of electron-produced ionization per 0.5 cm. radius increment for the x-rays of Fig. 9.

Fig. 14. Loss of electron-produced ionization per 0.5 cm. radius increment for the x-rays of Fig. 10.

equivalence of the plastic substance by raising it until it coincided with the electron-plus-photon curve at large distances from the beam, where the primary electron contribution was negligible. The primary electron contribution was not negligible for the highest energy x-ray beams used in this experiment. For these cases the non-air equivalence was estimated from the data of Attix and DeLaVergne (7).

The presence of the tube tended to distort the electric field near the center of the chamber. This distortion would produce an appreciable error in the slope of the photon curve. The magnitude of this distortion was determined by irradiating the entire chamber uniformly from one side with Cs^{137} gamma rays.³ The ionization was sampled with the tube in place and found to be uniform to ± 1 per cent over all but the center three collecting strips. The photon curve was therefore extrapolated in this central region.

³ This method of checking the field distortion was suggested by H. O. Wyckoff.

Figures 7-10 show the percentage of electron loss outside of a given radius from the x-ray beam. The results are computed in cylindrical co-ordinates (7) for a zero-diameter beam (8) (air at 26°C . and 760 mm. Hg). Figures 11-14 show the electron contribution per radius increment, $\Delta E/\Delta r$, at different inner radii. Agreement with the work of Attix and DeLaVergne is seen to be excellent. Figures 7-14 can be used to determine the electron loss in any particular free-air chamber. These curves assume that the electrons striking the plate system are absorbed. The back-scatter of electrons from aluminum is at least 20 per cent in this energy region (9). Thus, in designing a chamber, the electron loss should be kept smaller than 1 per cent if the error due to back-scatter is to be kept to a few tenths of 1 per cent.

Figure 15 shows the variation of the scattered photon contribution with radius. The curve has been arbitrarily normalized to 1 at the greatest radius used in this experiment. The shape of the curve was

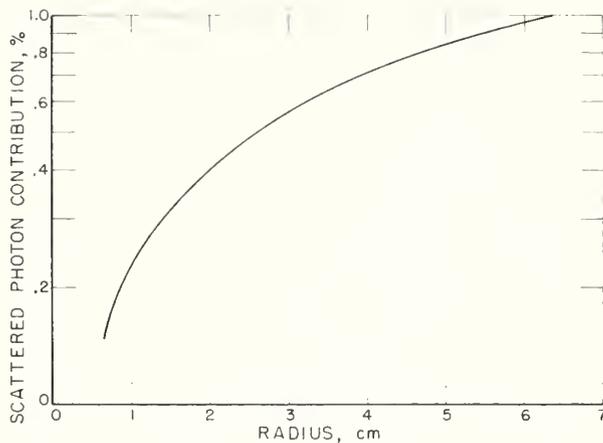


Fig. 15. Contribution of secondary photon ionization within different radii from a zero-diameter x-ray beam. Multiply the ordinate by the factors in Table I to obtain the contribution in per cent of the total primary electron ionization for the various kilovoltages and filtrations.

tube and the collecting region. Longer air paths would selectively filter the lower energy components of the x-ray beam, thereby increasing its effective energy. The effect of longer air paths on the electron and photon contributions was determined for 20 kv x-rays with inherent filtration and found to be negligible. The zero-beam diameter approximation was checked experimentally with 20 kv x-rays (inherent filtration) for 0.5 and 1.0 cm. diameter beams and found to be valid.

AIR ATTENUATION AND OTHER FACTORS

The field distortion study previously described makes possible design of a free-air chamber with a 5-cm. air path between

TABLE I: MULTIPLIERS FOR ORDINATE OF FIGURE 15

Kvcp	Inherent Filtration	0.5 mm. Al	1 mm. Al	1.5 mm. Al	2 mm. Al	3 mm. Al	4 mm. Al
20	0.9	0.9
30	0.9	0.75
50	0.9	0.7	0.6
60	0.9	...	0.6	0.55	...
75	0.9	0.6	...	0.55	...	0.5	...
100	0.9	0.5	0.5	...	0.5	0.5	0.4

found to be independent of the energy of the x-ray beam. The magnitude of the photon contribution varied with the kilovoltage and filtration used. The ordinate of Figure 15 must be multiplied by the multipliers in Table I to obtain the photon contribution within a given radius for a zero-diameter beam in per cent of the total electron contribution. Table II compares these results with the work of Attix and DeLaVergne for the higher kilovoltages and heavier filtrations. Their work involved an extrapolation of the scattered photon curve in the region where the radius was less than 5 cm. The present experiment, which measured the slope in this region, indicates that the extrapolation underestimated the scattered photon correction by about 0.07 per cent. The two experiments agree to 0.1 or 0.2 per cent when this correction is applied to their results.

TABLE II: COMPARISON WITH WORK OF ATTIX AND DELAVERGNE

Energy	Photon Contribution Within 5 cm. Radius, in Per Cent		
	Present Experiment	Attix	Attix, Corrected
100 kvcp, 4 mm. Al	0.32	0.13	0.20
75 kvcp, 3 mm. Al	0.41	0.14	0.21
60 kvcp, 3 mm. Al	0.44	0.14	0.21

The present experiment was done with about 1 meter of air between the x-ray

the chamber diaphragm and the center of the collector. If there is at least 1 meter of air plus 0.5 mm. of aluminum between the x-ray target and the free-air chamber, the air attenuation data of Day and Taylor (10) can be used with only small errors. A redetermination of the air attenuation corrections yielded corrections for a 5-cm. air path 0.15 per cent higher than the values given by those workers. For inherent filtration the difference was 0.2 per cent or less from 100 kv down to 50 kv; about 0.3 per cent at 30 kv. The differences imply a higher inherent filtration for the x-ray tube used by Day and Taylor.

TABLE III: HALF-VALUE LAYERS IN MM. OF ALUMINUM

Kvcp	Inherent Filtration	0.5 mm. Al Filtration	1 mm. Al Filtration	1.5 mm. Al Filtration	2 mm. Al Filtration	3 mm. Al Filtration	4 mm. Al Filtration
20	0.07 (0.08)*	0.24 (0.25)
30	0.08 (0.10)	0.36 (0.40)
50	0.09 (0.10)	0.50 (0.58)	0.88 (1.1)
60	0.09	...	1.1	2.1	...
75	0.09	0.66	...	1.6	...	2.5	...
100	0.09 (0.11)	0.77 ...	1.4 (2.1)	...	2.5 ...	3.5	3.9 ...

* The values obtained by Day and Taylor are given in parentheses.

Tungsten from the filament may have evaporated onto the beryllium window, increasing the inherent filtration. This, in turn, would decrease the air-attenuation corrections and increase the half-value layers that would be measured. The half-value layers measured by Day and Taylor are indeed larger than those that were measured in the present experiment (Table III). The air attenuation should be measured in each laboratory at the lower kilovoltages and filtrations if the highest accuracy is to be attained.

The roentgen is defined for a mass of *dry* air. Corrections for the humidity of the air may amount to +0.3 per cent in the soft x-ray region under ordinary laboratory conditions. Methods of calculating this correction are found in *Handbook 64*.

SUMMARY

The general requirements for the design of standard free-air ionization chambers have been summarized in National Bureau of Standards *Handbook 64*. The present experiment has determined supplementary criteria for the soft x-ray region. The maximum uncertainties in the factors investigated here may be summarized as follows: field distortion and errors in measuring the length of the collector ± 0.2 per cent, electron losses ± 0.1 per cent, scattered photon contribution ± 0.2 per cent and air attenuation ± 0.2 per cent. These values, combined with the estimates in *Handbook 64* for uncertainties in the

diaphragm area, charge measurement, etc., yield a probable limit of error of ± 0.5 per cent for the determination of exposure dose in roentgens in the soft x-ray region.

NOTE: The author expresses his gratitude to H. O. Wyckoff, F. H. Attix, S. W. Smith, and M. G. Ehrlich for many helpful discussions in the course of the experiment.

National Bureau of Standards
Washington 25, D. C.

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(Pro le summario in interlingua, vider le pagina sequente)

Absorption of X-Rays in Air

By Frank H. Day and Lauriston S. Taylor

Studies have been undertaken to determine the absorption in air of X-rays produced by voltages from 10 to 200 kv (constant potential) for various initial filtrations. A free-air ionization chamber on tracks is used to obtain absorption data over a 150-cm distance. No appreciable change in quality of X-rays is observed in this distance because of the air absorption. Hence, for short distances, effective air absorption coefficients for heterogeneous X-rays of various qualities have been determined.

I. Introduction

The use, in recent years, of beryllium-window X-ray tubes with their high-intensity outputs of soft X-rays has developed a need for air-absorption corrections where distance is a factor in X-ray exposure measurements. Such, for example, is the case where the inverse-square law is used in computing the exposure at a point when the actual measurement is of necessity made at some other point. There is also need for corrections to be applied to the free-air type of ionization chamber due to air absorption between the limiting diaphragm of the chamber and the front edge of the collector plates. Since the exposure rate is specified in the plane of the limiting diaphragm, air-absorption along the path between the diaphragm and the collector plate results in the readings of the chamber being too low. This distance in the case of the guarded-field free-air standard for calibrations up to 200 kv at the Bureau of Standards is 21.1 cm, and for unfiltered radiations produced at, say, 50 kv, the necessary corrections may be considerable.

II. Apparatus and Experimental Procedure

A diagram of the experimental arrangement is shown in figure 1, where a mobile, free-air-guard-plate ionization chamber is indicated. The opening in its lead shield is 10 cm in diameter. This opening is adequate to accommodate, at any point along the track, the total area of X-rays determined by the fixed, limiting diaphragm, the diameter of which is 1.75 cm. The effective width

of the collector plates is 10.2 cm, and the diameter of the beam with the chamber in its farthest position from the tube (position *B*, fig. 1) is 6.5 cm. The interelectrode separation is a fixed distance of 12.7 cm, and this is insufficient spacing to allow for the production and collection of all secondary electrons associated with a primary beam 6.5 cm in diameter. Hence, there is a slight loss of ionization, which earlier studies show does not exceed 1 or 2 percent.¹ No correction has been applied for this loss.

The charge collected in the ionization chamber at various points along the track is measured with a capacity compensator and string electrometer² with an accuracy of ± 1 percent. The exposure of the chamber is controlled by a timer operating a $\frac{1}{2}$ -in. lead shutter. The timer is actuated by an accurately controlled 60-cycle signal from the Radio Section of the Bureau of Standards.

Tungsten-target X-ray tubes with low inherent filtration are used in conjunction with various types and thicknesses of initial filtration. The term "initial filtration," as here used, refers to filters of aluminum, copper, or lead introduced between the X-ray tube and the column of air over which air-absorption measurements are made. The window thickness of the cerium glass in one X-ray tube used is 1.29 mm and of the beryllium in another tube, 1.5 mm.

Power supplied to the X-ray tube is furnished by a kenotron rectifier with a resistance-capacitance filter incorporated to reduce the ripple to

¹ Lauriston S. Taylor, George Singer, and A. L. Charlton, *Am. J. Roent.* **41**, 256 (1939).

² Lauriston S. Taylor, *BS J. Research* **6**, 807 (1931) RP306.

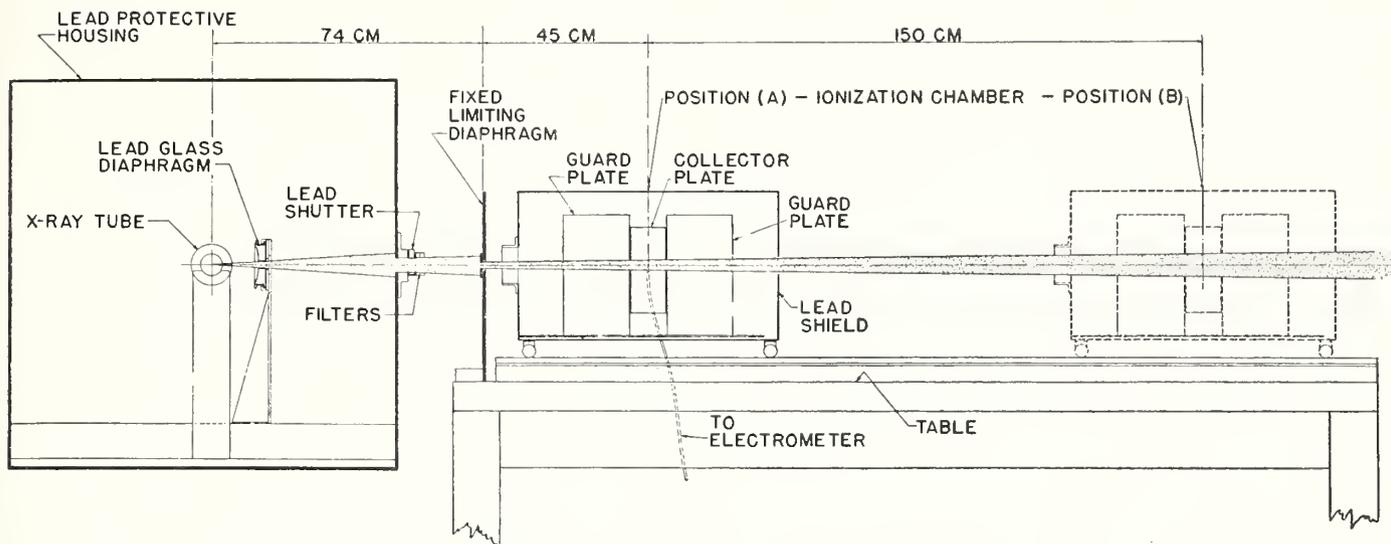


FIGURE 1. Experimental arrangement for air absorption measurements.

0.05 percent per milliamperere current drain. The tube currents used are not in excess of 10 ma.

The voltage input to the rectifier units is constant to 0.25 percent or better, the 208-volt commercial supply being fed through a voltage stabilizer. The voltage to the rectifier units is then varied by induction regulator control. It is estimated that the absolute direct-current voltage applied to the X-ray tubes is known to an accuracy of 1 or 2 percent.

The filament voltage of the X-ray tubes is stabilized by an electronic voltage stabilizer in the ground circuit feeding through an insulating transformer. The output of the stabilizer is also manually controlled by an adjustable-ratio auto-transformer to reduce long-period fluctuations.

In view of the above-described stabilization, the variation in quantity of X-ray output over a period of exposure of 10 seconds or more is not greater than 0.5 percent. This is also verified from previous experience on fluctuations in X-ray output of this equipment, as determined by the Bureau's standard chamber for measurements up to 200 kv.

III. Observations and Calculations

Under steady operating conditions of the X-ray tube, the ionization chamber is exposed for a given number of milliamperere-seconds at successive points along the track. The ratio of the ionization currents measured at two points along the track is a measure of the air absorption between these points, inasmuch as the total flux passing through the

limiting diaphragm is measured by the chamber, except for the part lost by air absorption.

Measurements are first made, using the beam in its minimum-filter condition. Filters of aluminum, copper, or lead are then interposed in the beam up to the point where the beam is rendered sufficiently homogeneous, so that further additions of filters will not lower the air-absorption coefficient by an appreciable amount. This is illustrated (fig. 2) by the semilogarithmic plots of X-ray

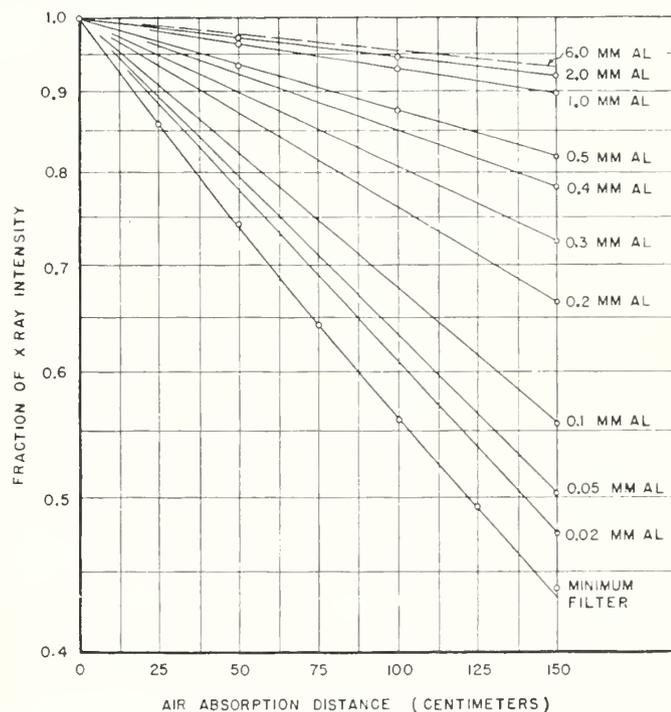


FIGURE 2. Effect of aluminum filtration on air absorption of 50-kv X-rays produced by beryllium-window X-ray tube with inherent filtration 1.5 mm Be.

intensity as a function of the length of the absorbing air path for the 50 kv X rays from the beryllium-window tube. Here it is observed that filtrations in excess of 2 mm of aluminum will not greatly affect the air absorption.³ Similarly, it is found that for any voltage up to 200 kv, filtrations of 6 mm of Al, 0.2 mm of Cu, or 0.1 mm of Pb are sufficient for the attainment of a practical lower limit to the air absorption.

These studies are made with X rays of 200 kv and less, to voltages as low as transmission through filters will allow. Minimum-filter conditions allow observations on X rays as low as 7.5 kv from a beryllium-window tube. Even these 7.5-kv X rays show no great change in quality due to absorption in the air itself over the distance of observation, as indicated by a near straight-line semilogarithmic plot in figure 3. Since the beam is effectively

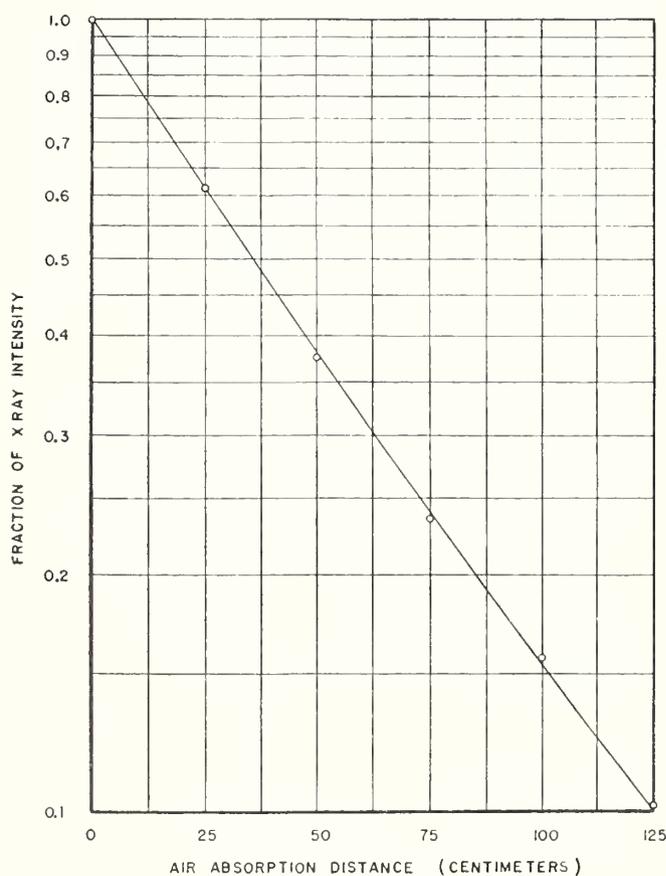


FIGURE 3. Air absorption of 7.5-kv X-rays produced by beryllium-window tube with inherent filtration 1.5 mm Be.

³ The curve for 6-mm filtration was not observed at this voltage because of the extremely long exposure which would be necessary. At 100 kv, however, the difference between the 2-mm and the 6-mm curves is approximately 1 percent. The difference would not be greater than this in the case of 50-kv X-rays, for the same amount of filter would render the beam more homogeneous in the latter case.

monochromatic, it is permissible to prepare tables of linear absorption coefficients involving a single term⁴ for air under normal atmospheric conditions, (760 mm of mercury pressure and 22° C), these to be applied over short distances.

Tables 1, 2, and 3 give effective linear absorption coefficients, μ (cm^{-1}) for air, where μ is used in the formula, $I_x = I_0 e^{-\mu x}$, I_x being the intensity at a distance X cm beyond the point where the original intensity, I_0 , is measured. The X-ray beam is here considered to be parallel.

TABLE 1. Linear absorption coefficients for X-rays of air under atmospheric conditions obtained with beryllium-window X-ray tube

Voltage		Absorption coefficient (μ)				
		Minimum filtration ¹	Added filtration			
			0.05 mm Cu	0.1 mm Cu	0.2 mm Cu	0.1 mm Pb
<i>kv</i>	<i>cm</i> ⁻¹	<i>cm</i> ⁻¹	<i>cm</i> ⁻¹	<i>cm</i> ⁻¹	<i>cm</i> ⁻¹	
50	5.8×10^{-3}	3.5×10^{-2}	1.5×10^{-3}	0.5×10^{-3}	0.3×10^{-3}	
60	5.6	3.1	1.2	.4	.3	
70	5.5	2.8	1.1	.4	.3	
80	5.3	2.5	1.0	.4	.3	
90	5.2	2.2	0.9	.3	.3	
100	5.1	2.0	.8	.3	.3	
120	4.8	1.6	.7	.3	.3	
140	4.6	1.3	.6	.3	.3	
160	4.3	1.1	.5	.3	.3	
180	4.1	1.0	.5	.3	.3	
200	3.9	0.8	.4	.3	.3	

¹ Minimum filtration is 1.5 mm of beryllium plus 119 em of air.

Data in these tables are derived by calculating μ for air between 119 cm and 269 cm from the tube target. Errors due to change in quality of the X-rays will be involved in applying these data to determine intensities at great distances⁵ from the target, and, also, at distances a few centimeters from the target. However, within the range of approximately 1 to 3 m from the target, these coefficients may be applied with little error.

For ready reference, some air absorption correction factors have been prepared and are shown

⁴ To accurately specify the absorption of a heterogeneous beam, it is necessary to employ an expression that is the sum of exponential terms involving different absorption coefficients.

⁵ "Great distances", as here used, refers to the absorption by a sufficient quantity of air to reduce the intensity of the original beam by several half-value layers. However, reductions of the order of only one half-value layer, such as observed in this experiment (fig. 2), show very little quality dependence.

TABLE 2. Linear absorption coefficients for X rays of air under atmospheric conditions obtained with beryllium-window X-ray tube

Voltage	Absorption coefficient (μ)							
	Minimum filtration ¹	Added filtration						
		0.05 mm Al	0.2 mm Al	0.5 mm Al	1.0 mm Al	2.0 mm Al	4.0 mm Al	6.0 mm Al
<i>kv</i>	<i>cm</i> ⁻¹	<i>cm</i> ⁻¹	<i>cm</i> ⁻¹	<i>cm</i> ⁻¹	<i>cm</i> ⁻¹	<i>cm</i> ⁻¹	<i>cm</i> ⁻¹	<i>cm</i> ⁻¹
7.5.....	20.5x10 ⁻³							
10.....	12.9	9.7x10 ⁻³	8.3x10 ⁻³					
15.....	7.8							
20.....	6.9	5.3	3.9	2.5x10 ⁻³				
30.....	6.1	4.9	3.3	1.8	0.9x10 ⁻³	0.8x10 ⁻³		
40.....	5.9	4.8	3.0	1.5	.8	.6		
50.....	5.8	4.6	2.8	1.4	.7	.6	0.5x10 ⁻³	0.4x10 ⁻³
60.....	5.6	4.5	2.7	1.3	.7	.6	.5	.4
70.....	5.5	4.4	2.6	1.2	.7	.5	.5	.4
80.....	5.3	4.3	2.5	1.1	.6	.5	.5	.4
90.....	5.2	4.2	2.4	1.0	.6	.5	.4	.4
100.....	5.1	4.1	2.3	0.9	.6	.5	.4	.4

¹ Minimum filtration is 1.5 mm of beryllium plus 119 cm of air.

TABLE 3. Linear absorption coefficients for X rays of air under atmospheric conditions obtained with cerium glass-window X-ray tube

Voltage	Absorption coefficient (μ)					
	Minimum filtration ¹	Added filtration				
		0.5 mm Al	1.0 mm Al	2.0 mm Al	4.0 mm Al	6.0 mm Al
<i>kv</i>	<i>cm</i> ⁻¹	<i>cm</i> ⁻¹	<i>cm</i> ⁻¹	<i>cm</i> ⁻¹	<i>cm</i> ⁻¹	<i>cm</i> ⁻¹
20.....	3.2x10 ⁻³					
30.....	2.6	1.3x10 ⁻³	0.9x10 ⁻³	0.8x10 ⁻³		
40.....	2.2	1.1	.8	.6		
50.....	2.1	1.0	.7	.6	0.5x10 ⁻³	0.4x10 ⁻³
60.....	2.0	.9	.7	.6	.5	.4
70.....	1.9	.9	.7	.5	.5	.4
80.....	1.8	.8	.6	.5	.5	.4
90.....	1.8	.8	.6	.5	.4	.4
100.....	1.7	.7	.6	.5	.4	.4
110.....	1.6	.7	.6	.5	.4	.4
120.....	1.5	.7	.6	.5	.4	.4

¹ Minimum filtration is 1.29 mm of cerium glass plus 119 cm of air.

in figures 4, 5, 6, and 7. For a parallel beam, the air absorption correction factor, C , is taken to be,

$$C = \frac{I_x}{I_0}$$

In the case of a diverging beam, it may be defined as,

$$C = \frac{I'_x}{I'_0} \left(\frac{X_0 + X}{X_0} \right)^2,$$

where I'_0 and I'_x are the intensities measured by

r-meter ⁶ at distances X_0 and $X + X_0$ cm, respectively, from the tube target.

The correction factor, C , may be used in calculating intensities at various points, where a thimble chamber ⁶ measurement is made at one point only. Its application in field use is to determine the intensity at one position by air ionization measurement, calculate by inverse-square law the intensity to be expected at some distance farther from the

⁶ John A. Victoreen, Medical physics, p. 1370-1382 (The Year Book Publishers, Inc., Chicago, Ill., 1944).

source, if it were operating alone as a reducing factor; then, multiply this intensity by the air absorption correction factor to determine the true intensity. If the intensity is to be determined

at some point closer to the source than the ionization measurement is made, the reciprocal of the air absorption correction factor will be used as the multiplying factor.

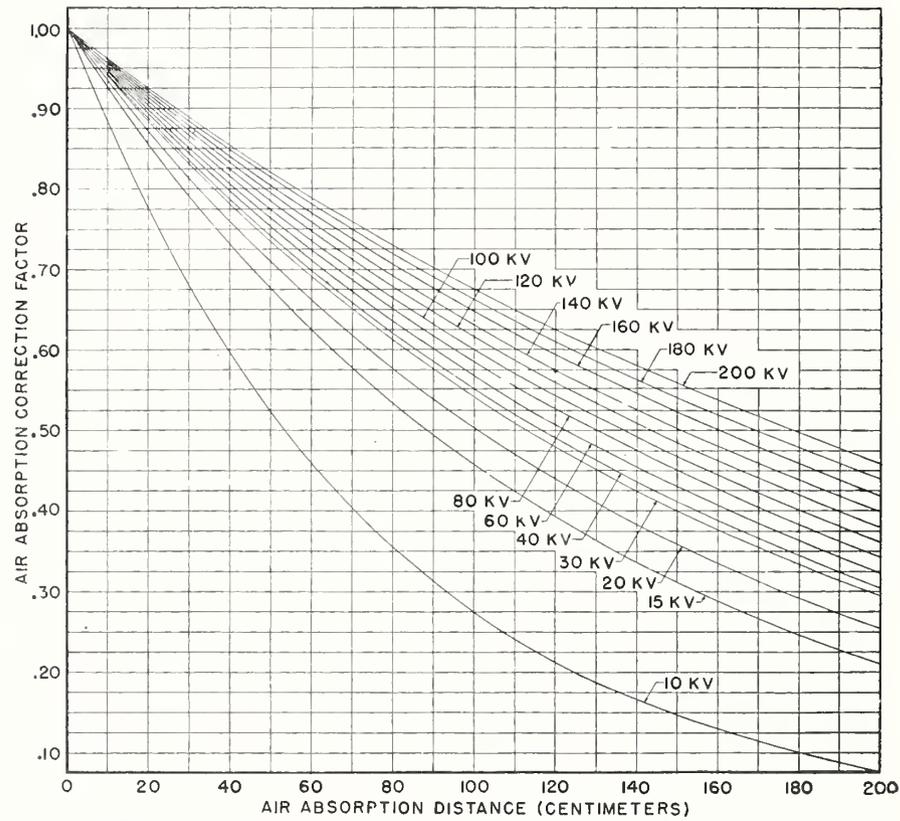


FIGURE 4. Air-absorption reduction in intensity of minimum-filter X-ray beam from beryllium-window tube with inherent filtration 1.5 mm Be.

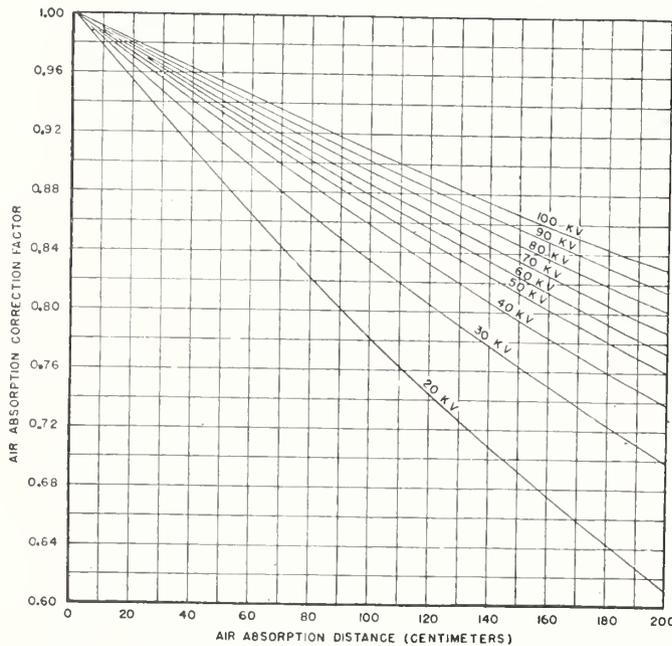


FIGURE 5. Air-absorption reduction in intensity of X-ray beam filtered by 0.5 mm of Al.

(Inherent filtration of X-ray tube used is 1.5 mm Be.)

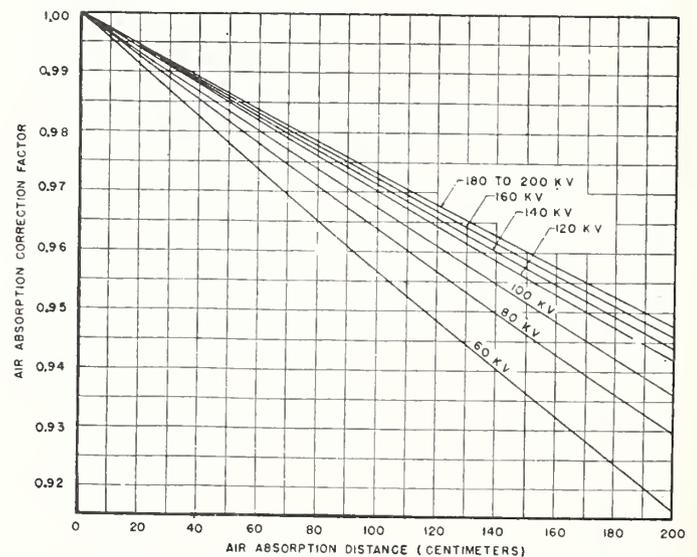


FIGURE 6. Air-absorption reduction in intensity of X-ray beam filtered by 0.2 mm of Cu.

(Inherent filtration of X-ray tube used is 1.5 mm Be.)

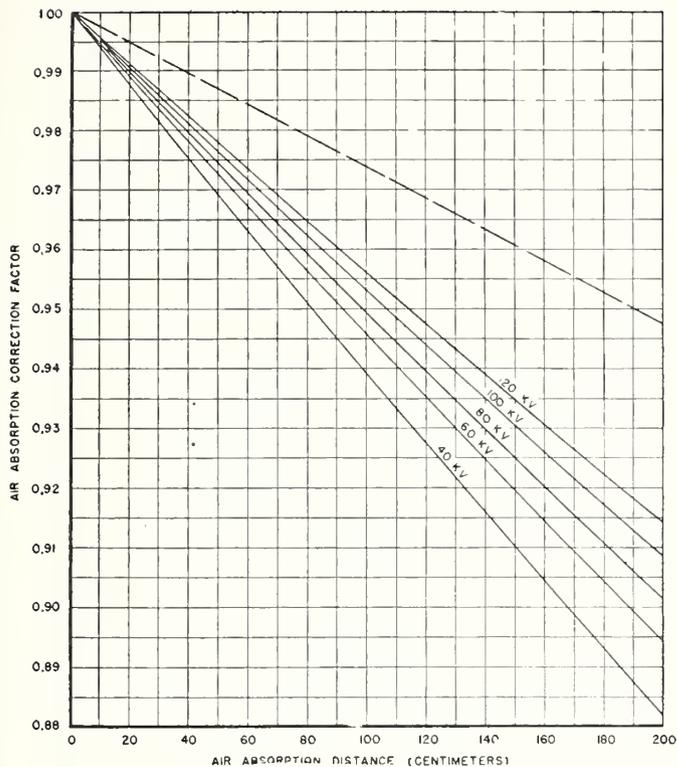


FIGURE 7. Air-absorption reduction in intensity of X-ray beam filtered by 2.0 mm of Al (solid lines).

Dashed line indicates reduction produced by 0.1 mm of Pb filter for X-rays of all qualities from 50 to 200 kv. (Inherent filtration of X-ray tube used is 1.5 mm Be.)

In cases where the graphs do not cover the conditions desired, the air absorption correction factor may be calculated, using the absorption coefficients given in tables 1, 2, and 3, and the equation, $C=e^{-\mu X}$. As an example of this calculation, table 3 indicates that μ for 40 kv with 1.0 mm Al of added filtration is $0.8 \times 10^{-3} \text{ cm}^{-1}$. C is then defined for arbitrary values of X beyond 119 cm from the tube target. It is convenient to arrange the work in tabular form, as follows:

X	μX	$e^{-\mu X}$
<i>cm</i>		
25.....	0.02	0.98
50.....	.04	.96
100.....	.08	.92
150.....	.12	.89
200.....	.16	.85

IV. Free-Air Chamber Versus Thimble-Chamber Air-Absorption Data

That the air absorption factors determined with the free-air chamber are applicable also to thimble chamber use is indicated by reference to table 4. In this case, the limiting diaphragm is removed and a 25-r thimble chamber is placed at the midpoint of the collector plates of the free-air chamber, the latter chamber being here used only as a vehicle for transport of the thimble chamber. Data obtained are at 90 kv with no filtration other than 1.29 mm of cerium glass, the tube window thickness.

TABLE 4. Comparison of air absorption data for 90-kv X-rays obtained by thimble ionization chamber with those obtained by free-air ionization chamber under minimum filter condition (1.29 mm of cerium glass)

Distance, X , of air absorption ¹	Intensity, as measured by thimble chamber ²	Thimble chamber intensity calculated to indicate air absorption only ³	Intensity as measured by free-air chamber
<i>cm</i>	<i>percent</i>	<i>percent</i>	<i>percent</i>
0	100	100	100
25	64.9	95.1	94.9
50	44.9	90.7	91.0
75	32.5	86.4	87.0
100	24.5	83.1	83.1
125	18.9	79.4	80.3
150	15.0	76.7	77.7

¹ X is the ionization chamber travel distance, the zero position of which is 119 cm from the X-ray tube target.

² This includes inverse-square reduction in intensity superimposed upon air absorption reduction.

³ The observed thimble chamber intensity times $(119+X/119)^2$ yields an intensity indicating the air absorption reduction only.

These data show reductions in intensity due both to the inverse-square law and to air absorption. The minimum target-to-thimble-chamber distance used is 119 cm. Multiplication of the thimble chamber readings by $(119+X/119)^2$, where X is the thimble-chamber travel distance, yields data indicating air absorption only. The data obtained in this manner with the thimble chamber agree within a percent with those using the free-air chamber as described in section III.

In the case of soft X-rays, thimble-chamber measurement of the ratio of intensities between two points may be made without appreciable error, because there is no significant selective absorption of the soft components of the X-rays in traversing the amounts of air considered. The calibration of thimble chambers for soft X-rays, however, will be in error, the readings being less than those for hard X-rays, because there is

appreciable absorption of the soft rays in the thimble chamber wall. The subject of such calibrations for soft X-rays will be discussed in another paper now in preparation.

The authors thank John H. Baltrukonis for obtaining much of the data reported above.

WASHINGTON, September 15, 1947.

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Thimble-Chamber Calibration on Soft X-rays

By Frank H. Day

Condenser r -meter thimble chambers of various types have been calibrated against a standard free-air ionization chamber to indicate the wavelength dependence of their readings after exposure to X-rays excited by potentials ranging from 10 to 200 kilovolts. X-ray tubes with low inherent filtration are used. Calibrations are conducted for minimum-filter conditions, and the effect of added filtration on the calibrations is also noted. It has been observed, for instance, that a chamber that reads correctly for filtered radiation of 100-kilovolt quality is in error by a factor of 2.3 for 10-kilovolt X-rays.

I. Introduction

Thimble chambers of the Victoreen type are normally designed for the measurement of relatively hard radiations and are customarily calibrated with X-ray beams that are produced by tubes with an applied voltage of 70 to 200 kv, and which are subjected to some preliminary filtration, at least 2 mm of aluminum or 0.2 mm of copper. Over this range, the chambers yield readings that are practically independent of the radiation quality when calibrated against a free-air chamber. However, in the absence of any other ionization chamber, they are frequently used for measuring very much softer radiations than those for which they are designed or calibrated. Numerous cases have come to our attention where the red Bakelite 25- r chamber has been employed to measure radiations produced by potentials as low as 20 or 30 kv, and it has been obvious in many cases that the users have had no reasonable idea as to the magnitude of the errors¹ involved. It therefore appeared to be desirable to investigate the magnitude of these errors and provide some quantitative data thereon.

Measurements cited in this paper with respect to the effect of added filtration on the calibration relate primarily to one 25- r chamber. The effect of thimble-chamber type is studied with red Bakelite 25- r , 100- r , and 250- r chambers, as well as the newly designed 250- r nylon chamber. When calibrations are made in a given X-ray beam, or in different beams of exactly the same quality, then,

as noted below, different chambers of the same type generally require the same corrections.² However, chambers of different types require different corrections.

Although the results reported here may be taken as typical, the very magnitude of the corrections required indicates that whenever a thimble chamber is to be used for precise measurement in the very soft X-ray region, it should be specifically calibrated for the exact conditions under which it will be used. This becomes more important, the lower the X-ray excitation potential. The wave form of the excitation potential becomes increasingly more important at the lower potentials also.

It is desirable for thimble-chamber readings to bear a constant relationship to those of a standard free-air chamber over a wide range of X-ray quality. To achieve this, it is necessary that the type of absorption and scattering in the thimble chamber wall be equivalent to that which is observed in air, and that the absorption in the wall thickness be negligible in comparison with the transmission through it; or that the secondary corpuscular radiation within the cavity be enhanced by means of a suitable internal coating of the wall to compensate for radiation lost by absorption in the wall thickness.

The practical attainment of this requirement is not difficult for 50 to 200-kv X-rays emitted by a tube whose inherent filtration is at least 0.2 mm of copper. For example, in the measurement of

¹ By "error" is meant the departure of r -meter readings from those of a standard free-air chamber.

² "Correction," as here used, is a factor by which r -meter readings must be multiplied to make them agree with those of a standard free-air chamber.

radiation from such a tube, the Victoreen 25-*r* thimble chamber, with its 0.6-mm red bakelite wall, reads only 5 percent low for 50-kv radiation if it reads correctly for 100 to 200-kv radiation.

The problem is much more difficult, however, in the case of long-wavelength X-rays, produced by a tube whose inherent filtration is, say, 1.5 mm of beryllium. X-rays as long as 4 Å in wavelength are available in measureable quantity from such a tube, and these are readily absorbed, even in a few centimeters of air. There are applications where it is necessary to determine the exposure at or near the portal of a beryllium-window tube. A properly designed chamber, therefore, should measure 4-Å X-rays, (approximately 3-kv quality) with a minimum of correction.

An attempt has been made in this direction,³ utilizing an ionization chamber having a beryllium wall. The use of an open-air chamber has been reported,⁴ utilizing a limiting diaphragm and a mesh-type electrode system consisting of silk threads soaked in India ink to make them electrically conducting. A similar chamber has also been constructed,⁵ substituting, in place of the mesh of silk threads, Kodapak I, which is cellulose acetate sheet 0.00088 in. thick containing some plasticizer. Its calibration at the National Bureau of Standards yields scale factors (roentgens per scale division), which vary by less than 8 percent as the X-ray excitation potential is varied from 7.5 to 90 kv and the filtration from the minimum of 1.5 mm of beryllium plus 100 cm of air to this plus 4 mm of aluminum.

II. Experimental Procedure and Results

In this study, the usual substitution method was employed in the calibration of the thimble chambers, the radiation being first measured with a standard guarded-field, free-air ionization chamber and then with the thimble chambers placed in the position of the limiting diaphragm. The filtration between the X-ray tube portal and the ionization chamber is, in all instances, 100 cm of air at approximately 760 mm Hg pressure and 22° C. It is necessarily a part of the minimum filtration, although it is not hereinafter so designated.

³ J. A. Victoreen, Z. J. Atlee, and E. D. Trout, *Am. J. Roent.* **53**, 391 (1945).

⁴ Edith H. Quimby and Elizabeth F. Foehl, *Am. J. Roent.* **50**, 653 (1943).

⁵ Personal communication, H. E. Seemann (Eastman Kodak Co., Rochester, N. Y.).

The standard ionization chamber used in this comparison was the one designed for measuring radiations produced by potentials up to 200 kv. It has an air path of 21.1 cm between the limiting diaphragm and the front edge of the collector plate.⁶ Corrections required for the air absorption over this distance vary from about 0.5 percent for 200-kv radiation to 33 percent for 10-kv radiation,⁷ and these have been applied in this calibration.

"Constant"⁸ potentials, varied in steps between 10 and 200 kv, were employed on each of two tungsten-target X-ray tubes used in the calibration. The first was a thin-walled (1.3 mm) cerium-glass tube, which offers about as little inherent filtration as any glass-window tube. The second was a 200-kv water-cooled tube whose window thickness is only 1.5 mm of beryllium, and which has almost no inherent filtration.

The *r*-meter readings were observed over approximately the same scale range to avoid any possible scale-factor error and were readily reproduced within ± 1 percent. This is an over-all error, which includes fluctuations in the calibrating equipment of not greater than ± 0.3 percent.

Figure 1 shows thimble-chamber corrections using the beryllium-window tube over the range of 10 to 100 kv, and for filtrations ranging from the minimum of 1.5 mm of beryllium to this plus 6 mm of aluminum of added filtration. For example, it is observed for the minimum filtration, that the correction is 1.52 at 100 kv and increases to 2.3 at 10 kv. It is obvious that when such a large correction is necessary, the calibration of a thimble chamber for use with very soft radiations must be made with considerable care. In fact, some earlier studies⁹ indicate the necessity of effecting a special calibration for each specific tube in conjunction with its own specific potential source. The insertion of as little as 1 mm of aluminum in the beam, however, reduces the error from 52 to 3 percent at 100 kv and from 60 to 8 percent at 50 kv.

⁶ Lauriston S. Taylor and George Singer, *BS J. Research* **5**, 507 (1930) RP211.

⁷ Frank H. Day and Lauriston S. Taylor, *J. Research NBS* **40**, 393 (1948) RP1883.

⁸ The power supply has a resistance-capacitance filter to reduce the ripple to 0.05 percent per milliampere current drain, and currents used in these calibrations did not exceed 10 ma.

⁹ L. S. Taylor, G. Singer, and C. F. Stoneburner, *BS J. Research* **11**, 293 (1933) RP592.

Similar corrections using the 1.3-mm glass-wall tube are shown in figure 2. Here, for minimum filtration, it is seen that the correction varies from 1.17 at 100 kv to 1.34 at 20 kv. Comparing these corrections with those of the 0.3-mm aluminum curve in figure 1, it is found that they are closely alike over the whole potential range, indicating

that the 1.3-mm of cerium glass is equivalent in absorption to approximately 0.3-mm of aluminum.

Calibration curves for the beryllium-window tube and copper filtrations at potentials up to 200 kv are shown in figure 3. It is observed that until approximately 0.22-mm of copper filtration is added to the beam, the errors at low potentials

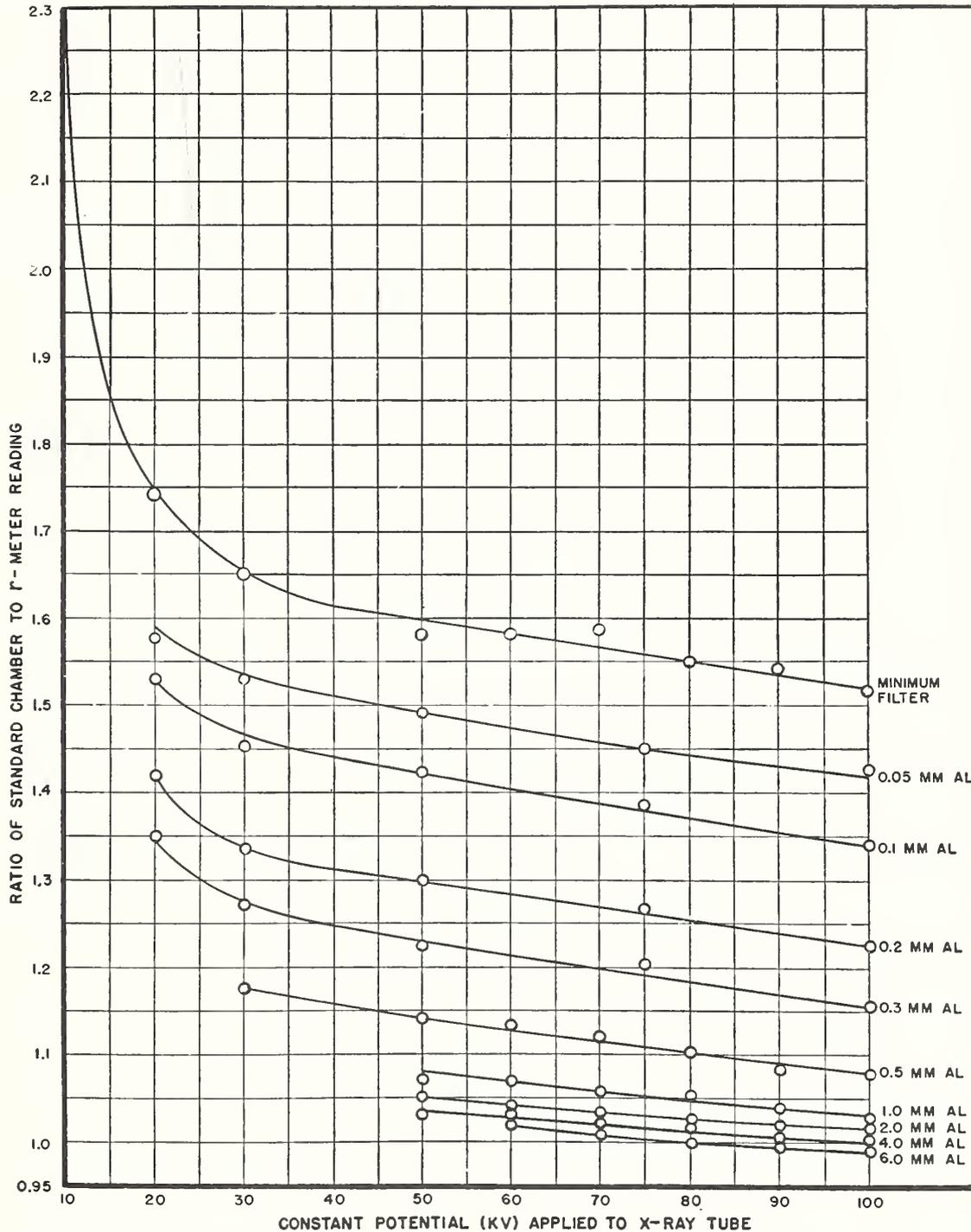
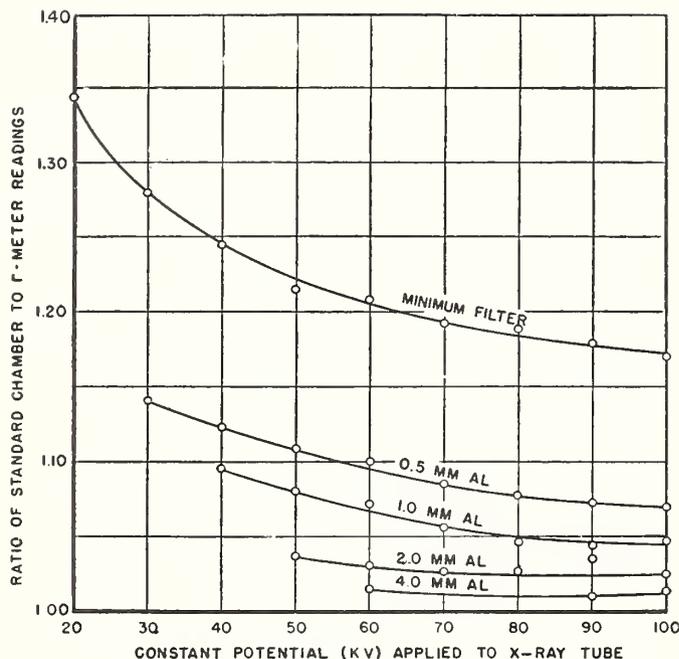


FIGURE 1. Effect of X-ray excitation potential and added aluminum filtration on the readings of r-meter No. 1193 and 25-r Bakelite thimble chamber, using X-ray tube with 1.5 mm of beryllium inherent filtration.



are relatively large, 11 percent, for example, at 50 kv and 0.11-mm of copper filtration. However, for 0.22-mm of copper filtration, the thimble chamber agrees with the standard within ± 2 percent over the range of 50 to 200 kv, and additional filtration does not change the calibration curves observably.

In figure 4, calibration curves, for X-rays from the tube with 1.5-mm of beryllium inherent filtration, and no added filtration, show the effect of thimble-chamber type. In this comparison, three types of Bakelite chambers (250-*r*, 100-*r* and 25-*r*) were used in conjunction with *r*-meter No. 1193. Another 25-*r* Bakelite chamber was

FIGURE 2. Effect of X-ray excitation potential and added aluminum filtration on the readings of *r*-meter No. 1193 and 25-*r* Bakelite thimble chamber, using X-ray tube with 1.3-mm of cerium glass inherent filtration.

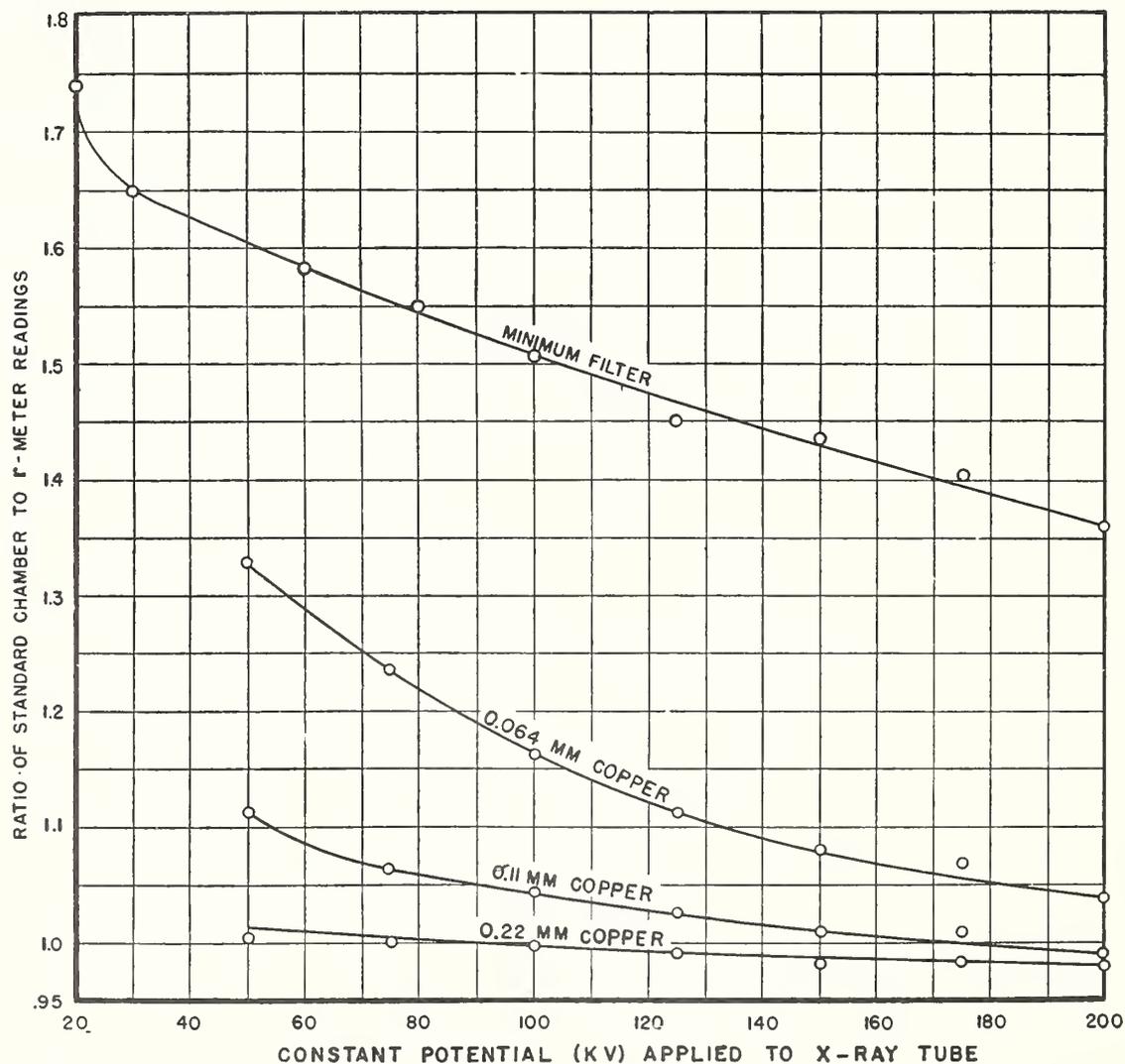


FIGURE 3. Effect of X-ray excitation potential and added copper filtration on the readings of *r*-meter No. 1193 and 25-*r* Bakelite thimble chamber, using X-ray tube with 1.5-mm of beryllium inherent filtration.

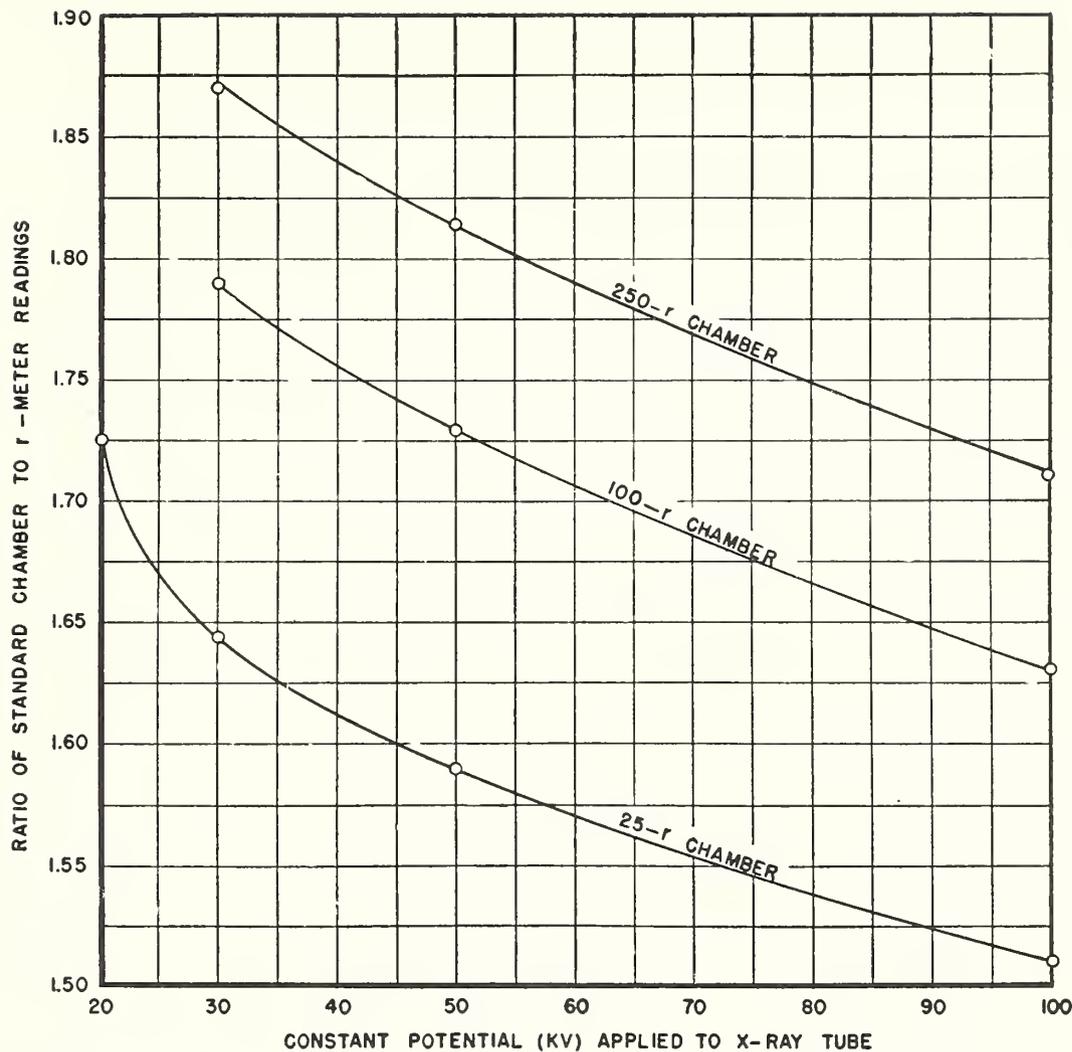


FIGURE 4. Effect of X-ray excitation potential on the readings of various types of Bakelite thimble chambers, using X-ray tube with 1.5-mm of beryllium inherent filtration and no added filtration.

The 250-r, 100-r, and 25-r chambers are each used in conjunction with *r*-meter No. 1193. Another 25-r chamber used in conjunction with *r*-meter No. 1282, yields readings that are the same within experimental error, as those observed with the 25-r chamber, which were read on *r*-meter No. 1193.

used in conjunction with *r*-meter No. 1282. The readings of the two 25-r chambers and *r*-meters are the same, within experimental error, whereas those of the 100-r and 250-r chambers show substantially greater departure from those of the standard chamber. It is interesting to note that the calibration curves for these chambers run closely parallel to each other. For example, the correction factor to the 100-r and 250-r chambers are 1.63 and 1.71, respectively, at 100-kv, and 1.79 and 1.87, respectively, at 30-kv, or differences of 0.08 at each potential.

A 250-r nylon chamber with a 0.005-in. wall thickness has recently become commercially available, and its calibration is shown in figure 5 using an X-ray tube with 1.5 mm of beryllium inherent filtration. For minimum-filtered radiation, this

chamber reads in error by 31.5 to 28 percent over the range of 30 to 100 kv. This is in contrast with an error in the 250-r red bakelite chamber readings of 87 to 71 percent in the same excitation potential range, as seen in figure 4.

Table 1 shows the aluminum half-value layers for 10 to 200-kv X-rays with filtration ranging from 1.5 mm of beryllium to this plus 6 mm of aluminum. For minimum filter the very small half-value layers, even at 200 kv, indicate that the great bulk of the absorbed radiation is extremely soft, perhaps in the region of 20 to 30 kv, in terms of the usual measuring techniques. As an example of the effect of X-ray beam quality upon thimble-chamber calibration, it is observed in figure 5 that 6 mm of aluminum are required to reduce the correction factor to a minimum at 150 kv. Table

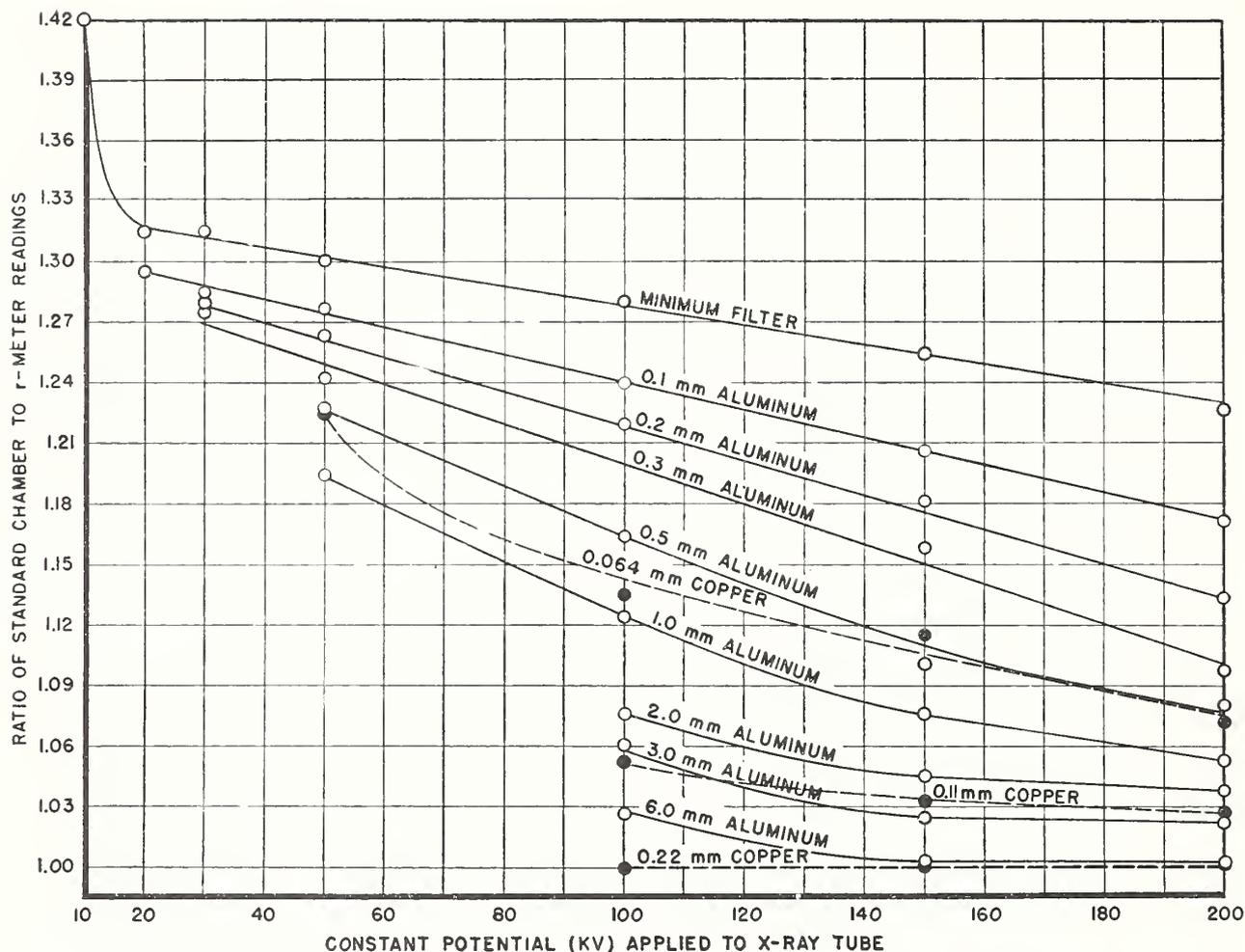


FIGURE 5. Effect of x-ray excitation potential and added filtration on the readings of r-meter No. 2449 and 250-r nylon thimble chamber, using x-ray tube with 1.5 mm of beryllium inherent filtration.

1 indicates that a 150-kv beam, prefiltered by 6 mm of aluminum, has a half-value layer of 7.6 mm of aluminum. This is in contrast with half-value layers ranging from 0.04 to 0.18 mm of aluminum

TABLE 1. Aluminum half-value layer determined with free-air ionization chamber as a function of filtration and of X-ray excitation potential

Constant potential	Half-value layer of aluminum								
	Minimum filter ¹	Added filter							
		0.1 mm Al	0.2 mm Al	0.3 mm Al	0.5 mm Al	1.0 mm Al	2.0 mm Al	3.0 mm Al	6.0 mm Al
<i>kv</i>	<i>mm</i>	<i>mm</i>	<i>mm</i>	<i>mm</i>	<i>mm</i>	<i>mm</i>	<i>mm</i>	<i>mm</i>	<i>mm</i>
10	0.04	0.06	0.10						
20	.08	.11	.15	0.18	0.22				
30	.09	.14	.19	.27	.35	0.6	0.9	1.2	
50	.10	.15	.29	.34	.57	1.0	1.4	1.9	2.8
100	.11	.19	.37	.48	1.0	1.8	2.9	3.6	5.3
150	.14	.29	.64	1.2	2.0	3.2	4.6	5.5	7.6
200	.18	.40	1.3	2.1	3.4	4.6	6.3	7.1	8.8

¹ 1.5 mm of beryllium, plus 100 cm of air.

at 10 and 200 kv, respectively, for minimum-filtered beams. In view of the large percentages of soft components in beams from beryllium-window tubes, it is to be expected that chambers used for measuring radiations from such tube, with no added filtration will be very critical as to design characteristics.

III. Conclusion

Thimble chambers can be calibrated for use with the very low-potential, lightly filtered radiations, such as produced by beryllium-window X-ray tubes. However, the errors may be as large as 130 percent for radiation produced at 10-kv constant potential.

Considerable care must be exercised in determining the amount and kind of filtration and potential employed in thimble-chamber calibrations for soft X-rays. These factors are relatively unimportant, however, where filtrations above 0.2 mm of copper or about 6 mm of aluminum are used.

WASHINGTON, January 26, 1948.

Scintillation Spectrometry of Low-Energy Bremsstrahlung*

Margarete Ehrlich

A method was devised to measure bremsstrahlung spectra from commercial X-ray tubes on an absolute scale, using a thallium-activated sodium-iodide crystal-scintillation spectrometer. The method was used to study bremsstrahlung spectra between 20- and 100-kilovolts exciting potentials from a beryllium window tube having a thick tungsten target. Fully corrected, absolute experimental spectra were obtained at exciting potentials of 50 and 100 kilovolts.

In order to compare the experimental results with theory, a calculation was made yielding the thick-target bremsstrahlung spectrum derived from Sommerfeld's theory. Experiment and theory showed order-of-magnitude agreement. However, a characteristic difference in spectral shape was observed, the experimental spectra showing a more pronounced peak. This peak is near 30 kilo electron volts and gives the impression of being superimposed on the spectral shapes expected from theory.

Finally, a point of interest to the practical user of the X-radiation from beryllium window tubes was brought out, namely, that a considerable portion of the low-energy radiation, at least in the region between 12 and 30 kilo electron volts, is strongly absorbed in the tungsten target of a conventional X-ray tube.

1. Introduction

1.1. Objective of the Present Study

Although high-energy bremsstrahlung spectra have recently been studied in great detail, a number of the more fundamental features of low-energy bremsstrahlung spectra, though under investigation for almost 40 years, have as yet not been satisfactorily explored. In fact, no absolute, fully corrected experimental data have ever been published, and no technique has been developed for a routine investigation of spectra from commercial X-ray tubes. It is the object of this paper to report on the design and performance of a scintillation spectrometer with a single thallium-activated sodium-iodide crystal that lends itself to the absolute determination of bremsstrahlung spectra from commercial X-ray tubes. So far, the instrument has been used up to 100-kv exciting potential only, but it is expected that with some modification of design it could be useful up to 150-kv and possibly to 200-kv exciting potential.

1.2. History of Bremsstrahlung Spectrometry

The history of the investigation of the nonrelativistic bremsstrahlung continuum is surveyed primarily by Kulenkampff [1, 2]¹ and by Finkelnburg [3].

a. Exploratory Experimental Work, Semiclassical Theory

The study of bremsstrahlung spectra received its first impetus from the experimental work of Laue and Bragg [4, 5], whose crystal diffraction spectrometer was until recently the only apparatus that lent itself to a satisfactory study of the bremsstrahlung

spectrum. The period between 1915 and 1930 produced the discovery and the experimental proof of the existence of a short wavelength limit of the bremsstrahlung continuum and of its dependence on electron velocity [6]. This period also produced the general exploratory studies of the spectral distribution as a function of electron energy and target material [7], the investigation of the efficiency of the process of bremsstrahlung production, and the first angular distribution studies that confirmed Sommerfeld's theoretical predictions [8, 9, 10].

In 1922, Kulenkampff measured the true spectral-intensity distribution of the bremsstrahlung from a number of different thick targets introduced into a gas-discharge tube that could be operated between 7 and 12 kv [11]. His measurements were fully corrected and absolute except for the conversion of the ionization in his detector (an air-ionization chamber) into units of absorbed photon energy. This conversion was not carried out because the wavelength dependence of the energy expended per ion pair was at the time unknown. Kulenkampff represented his corrected spectral-intensity data as a linear function of $(\nu_0 - \nu)$, where ν represents any bremsstrahlung frequency below ν_0 , the frequency at the Duane-Hunt limit. This expression is a very good approximation to his experimental spectral distributions.

Shortly thereafter Kramers published his semiclassical theory of the bremsstrahlung spectrum [12]. Kramers arrived at the frequency distribution of the total emitted radiation, starting with the classical expression for the spectral distribution of the energy radiated by an electron moving in the Coulomb field of an atomic nucleus. This yielded expressions for the bremsstrahlung intensity as a function of frequency. When these expressions are integrated over the target thickness, they take essentially the form of Kulenkampff's empirical thick-target formula.

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¹ Figures in brackets indicate the literature references at the end of this paper.

Because of the considerable difficulties associated with exact experimental crystal spectrometry of the bremsstrahlung continuum, most of the experimental work confirming Kramers' theory was carried out by indirect methods. One of these, introduced by Webster and Hennings [13], consisted in isolating narrow spectral bands by means of suitable filtration and studying their intensity as a function of electron energy (Isochromatenmethode). From the partial information gained in this way, one can reconstruct the total spectral distribution. Another method, used for thin-target work by Duane [9], Kulenkampff [14], and Nicholas [15], and recently developed to a considerable extent by others [16, 17], consists in deducing the spectral distribution from the shape of absorption curves. Such an indirect method is probably adequate for the determination of heavily filtered or of unfiltered X-ray spectra close to the Duane-Hunt limit, but breaks down for the long-wave end of the spectrum. The reason for the use of indirect methods to supplement diffraction spectrometry becomes clear if one considers the optimum X-ray target design factors and the nature of the various distorting influences of the diffraction spectrometer: On the one hand, it is desirable to work with thin targets in order to minimize electron diffusion and energy loss through multiple non-radiative collisions in the target proper and to measure the bremsstrahlung spectrum produced by a truly monodirectional and monoenergetic electron beam. It is further desirable to have low-atomic-number targets in order to prevent the absorption of a portion of the bremsstrahlung by the target itself. On the other hand, because of the low efficiency of the diffraction spectrometer, it is important to produce high radiation fluxes—a requirement that called for target-design factors exactly opposite to those outlined before as optimum for the determination of an undistorted bremsstrahlung spectrum. Further complications are introduced by the fact that the reflectivity of the crystal varies with the wavelength of the incident radiation and that the factors entering into the determination of beam intensity from the measurement of ionization currents in air-ionization chambers are quite complex, and were at the time of the early bremsstrahlung studies not at all well understood.

b. Quantum-Mechanical Theory of the Nonrelativistic Bremsstrahlung Spectrum, Experimental Verifications

The impetus for this phase came clearly from the theoretical side. In 1929 and 1931, Sommerfeld published the results of his quantum-mechanical calculations. Limiting his calculations to non-relativistic velocities, to dipole transitions, and initially to unscreened fields, he arrived at an exact expression for the production of photons of given energy and direction by incident electrons of given energy [18, 19]. Finkelburg [2] points out that when integrated over all photon directions, Sommerfeld's expression for the X-ray spectrum shows a considerable formal similarity to that derived by

Kramers. It is, therefore, not too surprising that the spectral distributions measured by Kulenkampff and others show a fairly good qualitative agreement with both Kramers' and Sommerfeld's theoretical expressions. Later, Sommerfeld's theory was refined by Nedelesky, who introduced screening corrections in order to take account of the presence of the atomic electrons [20].

The progress in theoretical understanding of the problems of the nonrelativistic bremsstrahlung continuum was not matched by experimental advances, which could have facilitated thorough checks of the theory. The available experimental methods were still essentially the same as those used in the early studies. This fact may well be considered as one of the reasons why experimental work centered solely on studies of the angular distribution and polarization of the bremsstrahlung,² while no further attempts were made to measure absolute spectral intensities or to extend the studies of the spectral intensity distribution to higher electron energies. The only recent determinations of true spectra by direct methods seem to have been those by Kulenkampff and his coworkers [21, 22]. They used a hot-filament glass-bulb X-ray tube, with a massive tungsten target, and arrived at relative bremsstrahlung spectra obtained with exciting potentials up to 50 kv. After applying suitable corrections, they showed that their experimental data followed the empirical law found by Kulenkampff in 1922. They mention, however, that the conditions for targets of low atomic number seem to be more complex. Diffraction spectrometry was never attempted by direct methods on thin targets of low atomic number, probably in part because of the lack in efficiency of the old-type diffraction spectrometer and also because of the apparent shift in general interest in basic physics from low-energy atomic to high-energy nuclear physics; the latter may also have been one of the most important reasons for the fact that the curved-crystal spectrometer, which has proved so useful in gamma-line spectroscopy up to 1 Mev and higher [23], has never been used to study bremsstrahlung continua. However, the interest in the field seems to have been kept alive, if not for fundamental then at least for practical reasons, as shown by the renewed attempts to find indirect methods to arrive at spectral distributions of bremsstrahlung continua by way of absorption measurements [16, 17].

c. Scintillation Spectrometry

Around 1945 it became apparent that by coupling Rutherford's scintillating screen (first used for the detection of individual alpha particles) with a photomultiplier tube, one could produce an extremely sensitive system for the measurement of radiation energies and intensities [24]. Application of photomultiplier and electronic techniques, in conjunction with newly developed scintillator systems, eventually transformed this simple device into one of the most

² See the literature given in W. Finkelburg [3] and Kulenkampff's article [2].

powerful tools of modern physical research [25, 26, 27, 28]. In principle, the method is based on establishing simple relations between the readily measurable intensity of the light pulses within the particular scintillator and the energy of the incident photons (or particles), as well as between the number of lightpulses in the scintillator and the number of incident photons (or particles.)

Hofstadter was the first to use thallium-activated sodium iodide, NaI(Tl), for scintillation spectrometry and to show that this material is particularly suited for spectrometry of low-intensity photon sources [29, 30]. Subsequently, he and Johansson independently observed the "photolines" caused by the strong photoelectric absorption of monoenergetic photons in the iodine of the crystal [31, 32]. Hofstadter also established that the height of the light pulses ensuing from the impinging photons was proportional to the energy lost by the secondary electrons in the crystal [33]; also, that in a NaI(Tl) crystal large enough to facilitate total absorption of the radiation, the number of light pulses is equal to the number of impinging photons, which, in turn, is proportional to the total radiation flux [31, 33]. These facts established the usefulness of the NaI(Tl) scintillation counter—when coupled with a suitable pulse-height discriminator—for X- and gamma-ray spectrometry.

Up to now the scintillation spectrometer has been mainly used for gamma-line spectroscopy. However, in spite of its rather poor resolution (as compared with a diffraction spectrometer) the advantages of the scintillation spectrometer over the diffraction spectrometer for the measurement of continuous spectra are quite apparent. The diffraction spectrometer is complicated to operate, has a low yield that depends on photon energy in a complicated way, and is limited to comparatively low energies. No such inherent limitations exist for the NaI(Tl) scintillation spectrometer. It is of near-portable character, which makes its adaptation to a number of different problems of difficult geometries possible. Provided that the crystal is large enough to absorb the incident radiation in its entirety, its efficiency is equal to unity. At present, the high-energy limit of such a total-absorption spectrometer is given only by the maximum size of the available NaI(Tl) crystals. Its efficiency is so high that the problem is usually not how to obtain sufficiently strong radiation sources, but how to make them weak enough.

The first to use the NaI(Tl) scintillation spectrometer for a study of the bremsstrahlung spectrum was Johansson, who made a qualitative study of the spectrum from a commercial, thick-tungsten-target, glass-walled X-ray tube in the energy range from about 10 to 100 keV [34]. He corrected his experimental data for the change in instrument resolution with energy, but made no attempts to assess other distorting factors, such as absorption of the radiation in the tube window, the counter wall, or the intervening air. These corrections would be needed to allow a comparison of experiment and theory.

Subsequently, the NaI(Tl) scintillation spectrometer was used on several occasions for the measurement of "internal" bremsstrahlung, associated with the beta decay of a number of radioactive elements [35, 36]. In these studies the corrections for the change of resolution with energy were carried out in a manner similar to those applied in the case of magnetic spectrometry [37, 38, 39], and additional corrections for the escape of radiation energy in the form of iodine K fluorescence from the NaI(Tl) crystal surface were calculated [36]. Recently, Ramm and Stein [40] used a scintillation spectrometer to determine rough qualitative spectral distributions of heavily filtered X-ray beams from commercial tungsten target X-ray tubes, again without any further corrections or attempts of a comparison with the true, unfiltered bremsstrahlung spectrum. No other work on scintillation spectrometry of bremsstrahlung continua has so far been reported. However, it may be noteworthy that the use of a NaI(Tl) crystal counter as detector of the low-intensity X-radiation scattered off a quartz crystal enabled Beckman [40] to measure the spectrum of a commercial X-ray tube operated at 125 kv constant potential. Beckman's main interest lay in the determination of the influence of current waveform on spectral distribution, and he therefore made no effort to obtain absolute experimental data, to apply corrections, or to compare his results with theory.

2. The Experiment

2.1. Source of Bremsstrahlung

The most basic experimental study on bremsstrahlung would be that carried out on the spectra from a number of thin targets of different compositions. Work on 1-MeV thin-target spectra was recently performed at the National Bureau of Standards by Motz and Miller [42]. A thick-target X-ray tube was chosen for the present study, mainly because it was readily accessible and a study of its radiation could therefore be carried out with the expenditure of a minimum in funds, machine time, and personnel time. However, the theoretical problem presented by the radiation spectrum from such a thick-target tube is much more complicated than that from thin targets, and it can, in fact, not as yet be solved in great detail.

In order to facilitate a calculation of the absolute-intensity distribution emerging from the tungsten target proper, an experimental X-ray tube with a beryllium window of known thickness and approximately known purity was chosen for this study. The tube was held to constant potential with a ripple of less than 0.05 percent per milliamperere. The potential across the tube was determined by means of measurements of the voltage drop across a calibrated 250-megohm resistor in series, and was maintained to an accuracy of less than 1 percent. Because of the high sensitivity of the spectrometer, it was necessary to operate the X-ray tube at extremely low currents. Such a procedure is justified under the assumption that a change in

tube current merely produces a change in X-ray intensity but not in spectral distribution. The tube currents were too low for conventional dynamical measurements, and for this reason the relation between tube current and the integral number of pulses counted in the detector system was established by the following indirect method: The tube was first operated on a relatively high current (order of magnitude of milliamperes) and a relation was established between tube current and ionization in a cavity chamber placed in the path of the radiation.³ The tube current was then lowered to fractions of a microampere (about $0.01 \mu\text{a}$) and the ionization in the cavity chamber was measured once more, this time simultaneously with the total number of pulses above a given height. The low-ionization measurements were carried out by a static method. The relation between tube current and total number of pulses above a given height was determined concurrently with the spectral measurement, which established the relation between the number of counts per minute per pulse-height interval and the total number of counts per minute above a given pulse height. The bias setting, which fixed the lower limit of pulse height for the total counts, was maintained constant to within 0.25 v throughout the entire experiment (which barely lasted more than 1 hour per spectrum).

2.2. Spectrometer Design Factors

It was essential to construct a crystal-photomultiplier assembly that would have good resolution, facilitate total absorption in the crystal, and have a window of considerable transparency to the incident radiation. A NaI(Tl) crystal lends itself especially well to this task in the low-energy region, because practically all photon absorption in the crystal takes place by photoelectric effect. In this case, the pulse-height distribution resulting from the absorption of monoenergetic photons consists of an essentially Gaussian curve, without any further disturbing distributions due to absorption by Compton effect or pair production. The broadening of monoenergetic lines into Gaussian distributions is mainly due to statistical fluctuations in pulse height originating at the emission from the cathode and the first few dynodes of the photomultiplier. Pulse-height resolution is customarily defined as a quantity proportional to the fractional mean-square deviation of pulse height for identical scintillations. Thus, as long as photon energy and pulse height are proportional, resolution should thus be inversely proportional to the square root of the photon energy.

A freshly-cleaved NaI(Tl) crystal, 0.5 in. high by 0.5 in. by 0.7 in., was chosen as the core of the detector. Calculation had shown that a crystal of these dimensions absorbs more than 99.99 percent of all incident radiation up to 80 keV in single photoelectric absorption events and about 98 percent

³ The ionization chamber had polystyrene walls, 0.001 in. thick, and although placed between the X-ray source and the spectrometer, its presence did not reduce the integral counting rate in the spectrometer, which was biased to record only pulses in the upper half of the spectrum.

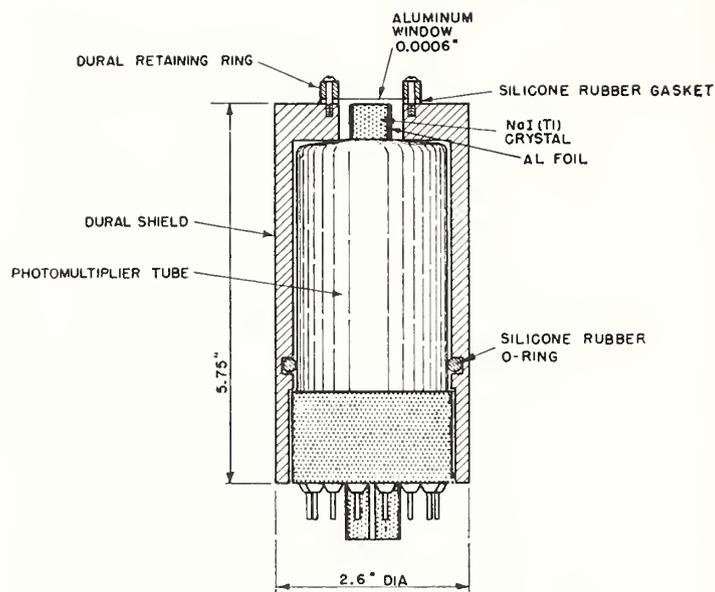


FIGURE 1. Photomultiplier—sodium iodide crystal assembly.

at 100 keV. The cube was sanded on the four side faces, which were then covered with aluminum foil for higher light-collecting efficiency. The upper and lower surfaces remained unsanded. The crystal was cemented to a Dumont K-1177 photomultiplier tube. The tube-crystal assembly was placed within an air-and-light-tight Dural can, filled with dry carbon dioxide of slightly higher than atmospheric pressure. Figure 1 shows the details of the assembly. The X-ray beam entered the assembly through an aluminum window, 0.0006 in. thick, after having been collimated by a 1-in.-thick brass diaphragm, of a diameter of approximately 6 MM. The diaphragm confined the radiation to the central portion of the crystal, without contributing any characteristic radiation within the measured energy range.

2.3. Experimental Setup

The essentials of the experimental setup are sketched in figure 2. The electrons impinged upon the target of the X-ray tube under an angle of 22 degrees, producing an X-ray beam whose central ray emerged from the X-ray tube in a direction perpendicular to the incident electrons. The X-ray beam, initially collimated by a 1-in. brass diaphragm close to the X-ray tube, reached the detector in a distance of 1 m from the target; the portion of the beam that entered the crystal subtended at the target a solid angle of 2.92×10^{-5} steradian. (The target dimensions were neglected in this estimate.) The electric pulses from the photomultiplier, corresponding to the light pulses in the NaI(Tl) crystal, were led through a cathode follower to a linear nonoverloading amplifier [43], giving pulses of a maximum height of 60 v, clipped by means of a delay-line to approximately square shape, and of about $10 \mu\text{sec}$ in duration. These pulses were monitored with an oscilloscope, and were also counted in a scaler (scaler I in figure 2). Simultaneously, the pulses were fed through a single-

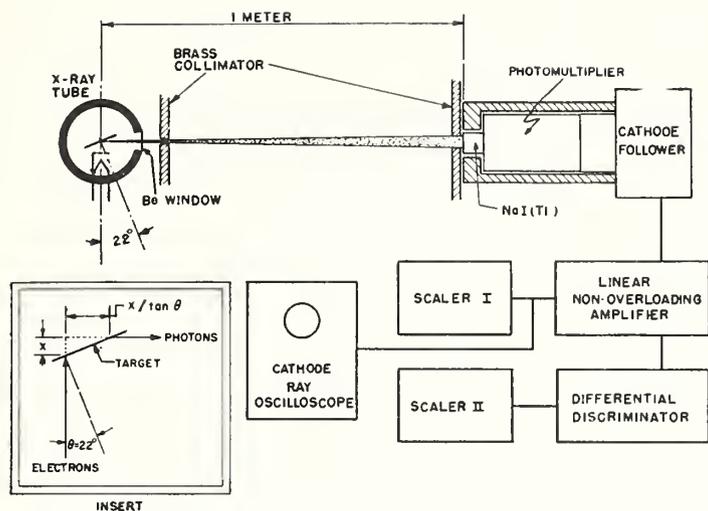


FIGURE 2. Details of the experimental setup.

Insert at lower left shows details of geometry of bremsstrahlung production.

channel differential pulse-height discriminator, and the number of pulses per channel width was determined by another scaler (scaler II in figure 2). Scaler II was biased to discriminate against low-noise pulses, and the bias on scaler I was adjusted so as to count only pulses above a certain height. This procedure allowed comparatively high differential counting rates in scaler II, at the same time keeping the integral counting rates in scaler I within reasonable limits. The differential counting rates varied between about 100 and 6,000 counts per minute; the integral counting rates did not exceed 3,500 counts per minute. In the case of the 50-kev and the 100-kev spectra, which were the only ones obtained on an absolute scale, the integral counting rates were used to tie in with the previously discussed current measurements.

2.4. Spectrometer Calibration

A calibration of resolution as a function of energy was obtained by determining the width of the pulse-height distributions at half height due to the K-fluorescence lines from nine different radiators. A radiator chamber similar to that described by Seemann [44] and a number of radiators supplied through the courtesy of Dr. Seemann were used for this purpose. The experimental setup is sketched in figure 3. Bremsstrahlung from a commercial tungsten-target X-ray tube operated at suitable voltages is made to impinge upon a sheet of the radiator material. The fluorescent radiation emerging under an angle of ninety degrees is analyzed in the NaI(Tl) crystal spectrometer. In order to isolate the fluorescence from high Z materials, suitably chosen filters have to be introduced in the path of the fluorescent radiation, in order to prevent the 90-degree Compton-scattered bremsstrahlung spectrum from being registered in the detector along with the K-fluorescence lines. No isolation of the K_{α} lines was attempted, as experiments had shown that, within the limit of the resolution of the instrument, the width of the K_{α} lines alone was the same as that

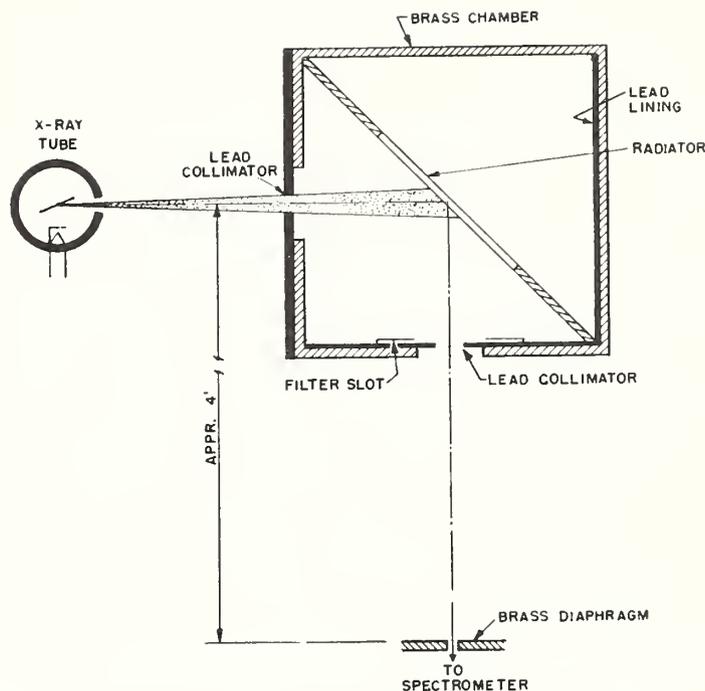


FIGURE 3. Fluorescence radiator chamber.

of the entire K series.⁴ Table 1 gives a list and description of the radiators used and of the filter materials that were found to be necessary to isolate the K-fluorescence lines, along with the exciting voltage for the bremsstrahlung used to produce the particular K fluorescence. Each pulse-height distribution was determined several times, each time with a different gain. The average resolution (in percent) was obtained for each line by computing the line width at half height in volts, expressed in percent of the pulse height at the fluorescence peak, also in volts.

TABLE 1. Fluorescence radiators and absorbers used to isolate fluorescence peaks

Element	Radiator		Exciting bremsstrahlung potential	Absorber element and thickness
	$K_{\alpha 1}$ line			
Strontium	14.21	50	None.	mm
Silver	22.23	50	Do.	Do.
Cadmium	23.24	50	Do.	Do.
Antimony	26.43	55 to 65	Do.	Do.
Lanthanum	33.53	55 to 65	Lead, 0.075.	
Samarium	40.23	80	Lead, 0.075, plus gold, 0.050.	
Tungsten	59.47	100	Silver, 0.57.	
Gold	68.94	115	Lead, 0.95.	
Lead	75.12	120 to 130	Do.	

Samples of pulse-height distributions obtained for four of the fluorescent radiators were used to check the linearity of the energy scale, that is, the proportionality of the voltage at pulse-height maximum with the energy of the X-ray photons. Figure 4 shows the results of this check. The importance of the proper choice of filtration becomes apparent

⁴ This is not at all surprising, because the other lines of the K series are much less than one-half as intense as the K_{α} lines and can therefore not influence the K_{α} line width at half height.

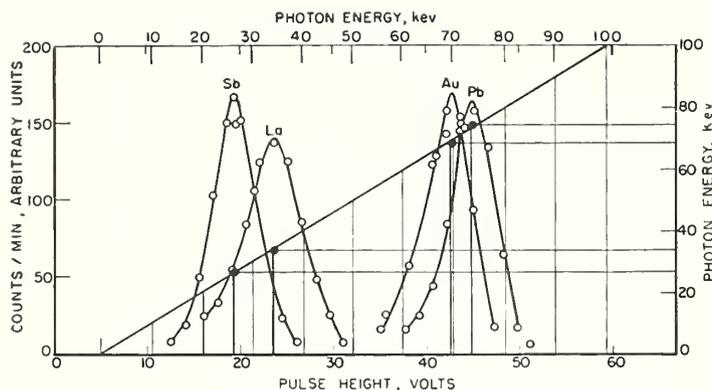


FIGURE 4. Energy-scale calibration of spectrometer.

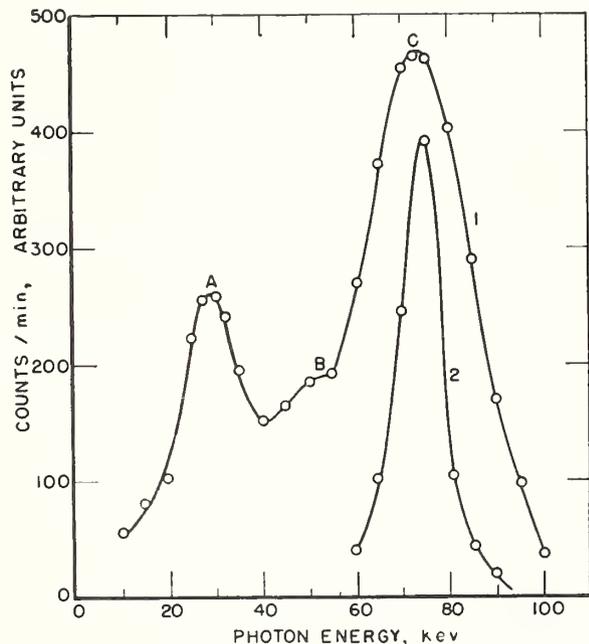


FIGURE 5. Pulse height distribution of fluorescence radiation from lead, irradiated with bremsstrahlung and observed perpendicular to the direction of bremsstrahlung incidence.

Curve 1. No filter between radiator and detector; A, peak of Compton-scattered bremsstrahlung spectrum; B, Compton-scattered K fluorescence peak from tungsten target; C, fluorescence peak from lead radiator.

Curve 2. 0.95 mm lead filter between radiator and detector.

in the pulse-height distribution shown in figure 4 for gold. This distribution was obtained with insufficient filtration and is therefore markedly skewed. The effect of filtration on the K-fluorescence pulse-height distributions is seen even better in the case of lead. Figure 5 shows the distribution obtained with a lead radiator, with and without filtration. When no filter is used, the entire bremsstrahlung spectrum, scattered through an angle of 90 degrees, is superposed on the lead K fluorescence. A 0.95-mm lead filter—although transparent to its own K fluorescence—strongly absorbs all lower energies, and thus isolates the lead K-fluorescence distribution. (The distribution below 60 keV, not shown in the graph, contained no points appreciably above background.)⁵

⁵ The resolutions shown for the lead-fluorescence pulse-height distributions, with and without added absorbers, are not quite comparable, as the two curves were obtained with different NaI(Tl) crystals.

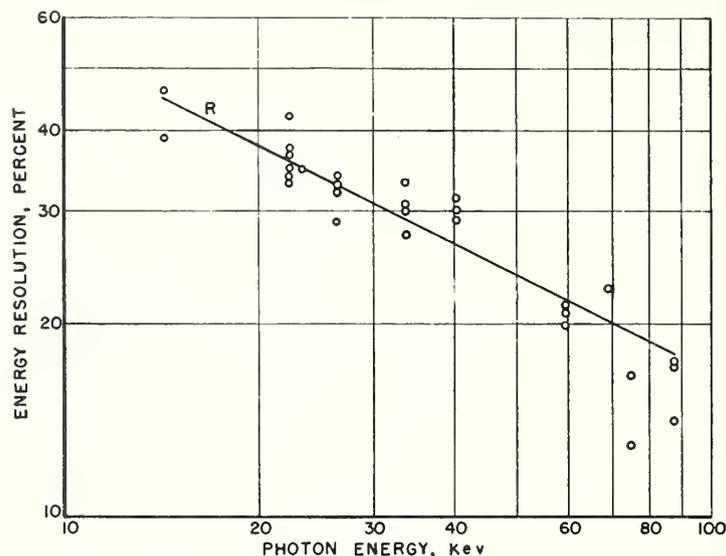


FIGURE 6. Percentage energy resolution of spectrum as a function of photon energy.

Figure 6 is a log-log plot of percentage resolution as a function of energy, obtained from the K-fluorescence data. Values obtained with the gamma rays from cadmium-109 and with the silver K fluorescence associated with the decay of cadmium-109 [45] are shown as well.

The line

$$R = \frac{168.0}{\sqrt{E}} + 0.1 \quad (1)$$

shown in the graph was fitted to the experimental data by the method of least squares. Equation (1) verifies the previously established fact that in this low-energy region the resolution depends primarily on the statistical distribution of the photoelectrons leaving the cathode of the photomultiplier. [46]

2.5. Raw Experimental Results

Figure 7 shows the differential counting rates obtained for the bremsstrahlung continua from the beryllium-window X-ray tube, operated at 50 and at 100 kv, as a function of photon energy. Due to the extremely low current levels used for this study, fluctuations in the bremsstrahlung intensity could not be entirely eliminated. For this reason, the quantity plotted on the ordinate is the following ratio:

$$\frac{C_{\text{diff}}}{C_{\text{total}}} \times \frac{C_{\text{total}}}{\text{ma}} \times \frac{1}{W} \quad (2)$$

C_{diff} is the differential counting rate per discriminator channel width; C_{total} , the integral counting rate; and W , the width of the discriminator channel, expressed in kilo electron volts. The plotted quantity is thus in units of number of differential counts per minute per milliamperere per kilo electron volt.

The two spectral distributions of figure 7 will be used in section 4 for an absolute comparison between theory and experiment. In order to give a better picture of how the spectral distributions change with exciting potential, five more spectra obtained at

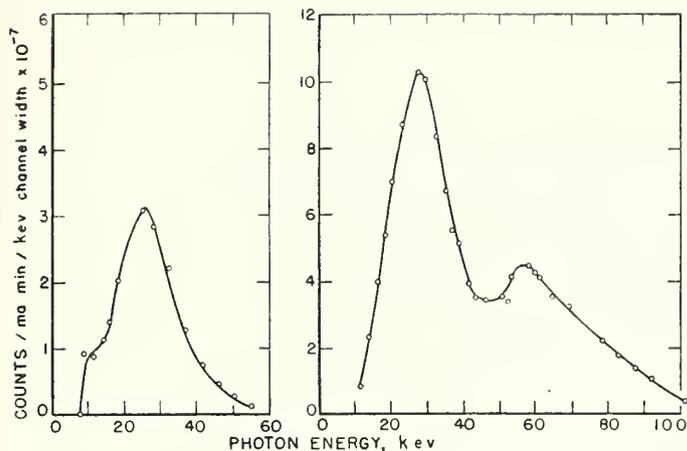


FIGURE 7. Differential counting rates, 50- and 100-keV bremsstrahlung spectra.

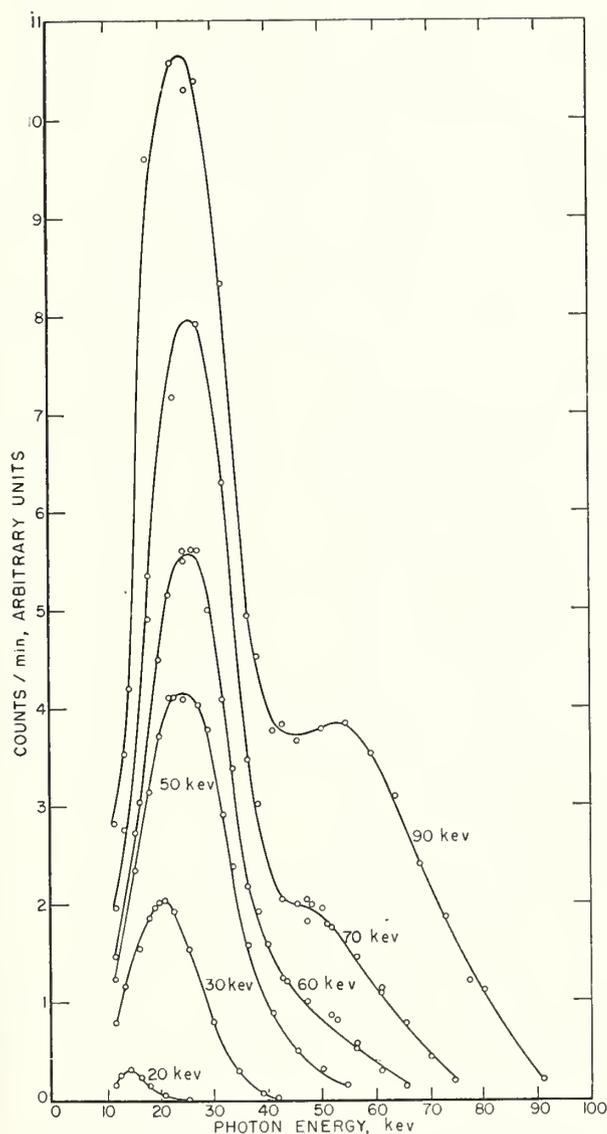


FIGURE 8. Change of spectral distribution with exciting potential.

exciting potentials between 20 and 100 kv are shown in figure 8. The ordinates of these spectra are not on a comparable scale.

The energy scales shown on the abscissas of figures 7 and 8 were obtained by a calibration of the spectrometer with the known gamma and X-ray lines of Cd^{109} . The differential counting rates are accurate to approximately ± 5 percent, the inaccuracy stemming from the limited counting statistics, as well as from the fluctuations in the discriminator channel width. There is an additional inaccuracy in the abscissa scale, amounting to about $\pm \frac{1}{2}$ volt in pulse height, due to limitations in the electronic stability of the system. The accuracy of the measurement of the current in the X-ray tube is estimated to be about ± 30 percent.

2.6. Correction of the Experimental Data

a. Variable Resolution

The problem of correcting the pulse-height distributions for variable finite resolution consists essentially in finding the relation between $C(E')$, the measured pulse-height distribution (number of counts per minute per discriminator window width)—corresponding to a particular measured photon energy interval between E' and $(E' + dE')$ —and $N(E)$, the true pulse-height distribution. It involves the solution of the following inhomogeneous Fredholm equation of the first kind:

$$C(E') = \int_0^\infty K(E, E') N(E) dE, \quad (3)$$

where $K(E, E')$, the kernel of the equation, represents a distortion function expressing the probability for a pulse due to photons of energy E to be recorded at the energy E' . Thus $K(E, E')$ is the function embodying the experimental data for the resolution of the detector as a function of energy.

In the energy region under consideration, the pulses of monoenergetic X- or gamma-ray lines were seen to be widened into Gaussian distributions of width $W(E)$ at half height due to the limited resolution. A valid representation of the distortion function K is therefore given by

$$K(E, E') = N e^{-\frac{(E-E')^2}{2\sigma^2}}, \quad (4)$$

where the mean-square deviation is given by $\sigma = [W(E)] / (2\sqrt{2\ln 2})$, and N represents the normalization factor. An equation of the type (3) lends itself readily to numerical evaluation with the aid of automatic computers. The method was first worked out by the Naval Research Laboratory group in connection with a somewhat different problem [47]. The method consists essentially in reducing the integral equation to an equation involving discrete sums. The function $K(E, E')$ becomes a matrix (K_{E_i, E_j}) whose rank r (here chosen as 19) is equal to the number of selected discrete energy points, and the functions $N(E)$ and $C(E')$ become the r -dimen-

sional vectors (N_{E_i}) and (C_{E_j}) of the matrix equation

$$(C_{E'_j}) = (N_{E_i})(K_{E_i E'_j}) \Delta E_i. \quad (5)$$

Its solution for (N_{E_i}) , the true pulse-height distribution, is readily obtained by matrix inversion of $(K_{E,E'})$ after the sum of the elements of each column of the matrix (K) has been normalized—a procedure taking the place of a normalization of the Gaussian function $K(E,E')$.

b. Crystal-Conversion Efficiency

Equation (3) actually contains another factor, namely, the efficiency of the spectrometer for the conversion of incident photons into electric pulses. As discussed earlier in this paper, at least 98 percent of all the impinging radiation are photoelectrically absorbed by the crystal. Furthermore, as no K-escape peaks [36] were ever observed on pulse-height distributions obtained with the particular spectrometer, it was concluded that the geometry of the detector was such that the escape of the iodine K fluorescence from the crystal surface was negligible. It was therefore considered legitimate to set the conversion-efficiency factor equal to 1.⁶

c. Absorption of Bremsstrahlung During Passage From Target to Detector

Table 2 lists the thicknesses and compositions of the absorbers in the path of the X-radiation and gives the values for the absorption corrections for 18 points between 10 and 100 keV. These values were computed with the aid of current absorption-coefficient tables [48]. The inaccuracies introduced in the absorption correction due to impurities in the

TABLE 2. Absorption of bremsstrahlung during passage from target to detector

Composition and thickness of absorbers			
Beryllium window, ^a 0.272 g/cm ² : 99.27% Be; 0.25% BeO; 0.10% Be ₂ C; 0.15% Al; 0.15% Fe; 0.08% Si.			
Aluminum window, 0.004267 g/cm ² : 99.5% Al; 0.1 to 1% Fe (considered as 0.5% in calculation); traces of Cu, Ni, Pb, Si, Ti, V, Zn, were neglected.			
Air, 0.1077 g/cm ² : 78% N ₂ ; 21% O ₂ ; 1% A.			
Total absorption correction as a function of quantum energy			
Quantum energy	Correction factor	Quantum energy	Correction factor
<i>kev</i>		<i>kev</i>	
13	1.52	58	1.06
18	1.29	63	1.06
23	1.19	68	1.06
28	1.12	73	1.05
33	1.09	78	1.05
38	1.08	83	1.05
43	1.07	88	1.05
48	1.07	93	1.05
53	1.06	98	1.05

^a The composition given for the beryllium window is that of the average stock of the Brush Beryllium Co., who furnished the window to the tube manufacturer.

⁶ Since this paper went to press, the spectrometer efficiency below 20 keV has decreased over the months. Some of the remarks below, about the lower portion of the spectrum, must accordingly be regarded with some caution.

aluminum and beryllium were estimated to be less than 2 percent.

2.7. Corrected Experimental Spectra

The pulse-height distributions shown in figure 7 were corrected in the manner outlined in section 2.6. Figures 9 and 10 show the uncorrected distributions,

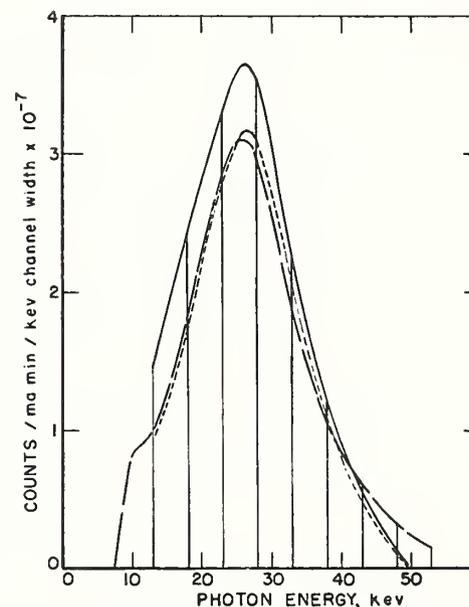


FIGURE 9. Correction of experimental data, 50-keV bremsstrahlung spectrum.

—, Uncorrected experimental data; - - -, experimental data corrected for finite spectrometer resolution; - · - ·, experimental data corrected also for absorption.

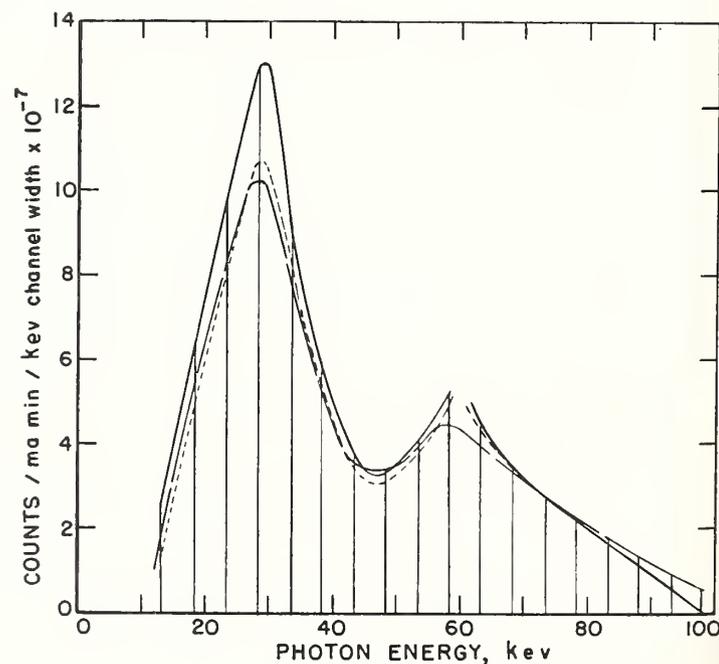


FIGURE 10. Correction of experimental data, 100-keV bremsstrahlung spectrum.

—, Uncorrected experimental data; - - -, experimental data corrected for finite spectrometer resolution; - · - ·, experimental data corrected also for absorption.

together with the distributions obtained in the energy interval between 13 and 100 keV after correcting for finite spectrometer resolution and after additional correction for absorption.

It is interesting to compare the spectra of figure 9, obtained with the beryllium-window tube, with the Compton-scattered-bremsstrahlung distribution of figure 5 from a tube with a window equivalent to $\frac{1}{2}$ -mm aluminum. The spectral peaks fall in both cases between 20 and 30 keV (note that the change in energy due to 90-degree Compton scattering of a 30-keV photon is negligible). Even after all the corrections for absorption in the materials between target and detector have been taken into consideration, the spectra from the beryllium-window tube still show a sharp intensity decline for energies below 25 to 30 keV. There is, furthermore, a quite distinct irregularity in the 50-keV spectrum near the tungsten L -absorption edge (see the uncorrected data) and only a comparatively small shift in the location of the ensuing spectral peak with an increase of the exciting energy from 50 to 100 keV. These facts make one suspect that the shape of the low-energy section of the spectrum is a property of the target rather than of the tube window.

The low-energy peak of the 100-keV spectrum is considerably more pronounced than that found by Johansson by scintillation spectrometry [34] or that found by Beckman by crystal spectrometry [41]. Experiments with two other NaI(Tl) detectors and an X-ray tube with higher inherent filtration confirmed that this difference is only in part due to the fact that an X-ray tube of lower inherent filtration was used for the present study. Also, it was found not to be a characteristic inherent in one particular NaI(Tl) detector.

The second, higher-energy peak appearing in the 100-keV spectrum and in all other spectra excited at potentials above 60 keV, is not a part of the bremsstrahlung, but represents the tungsten line spectrum ($K\alpha_1$ line at 59.5 keV). In spite of the corrections, this line spectrum - although approximately of the right over-all width - still appears entirely unresolved. This in part is probably due to the limited stability of the electronic equipment, and in part to the coarseness of the grid used in the discontinuous correcting procedure. The dotted line in figure 10 indicates the estimated course of the bremsstrahlung spectrum after the subtraction of the tungsten fluorescence peak and before correcting for absorption.

3. Theoretical Considerations

3.1. General Remarks

Kulenkampff, in his original experimental work on the thick-target bremsstrahlung spectrum [11], applied a number of corrections. These corrections stemmed in part from experimental and in part from theoretical considerations. He arrived in this way at a distribution for the bremsstrahlung spectrum from a thin target, and compared it with the then accepted theoretical expression.

For the present study, a slightly different approach was chosen. Because of the primary interest in the spectrum as it emerges from the target of a commercial X-ray tube, it was decided to apply the necessary corrections not to the experimental data but to Sommerfeld's expression for the cross section for thin-target bremsstrahlung. In this way, one arrives at an expression for the theoretical thick-target spectrum, and can compare it directly with the experimental spectrum emerging from the thick target (that is, after correcting the experimental data for absorption in air and in the other intervening materials). This procedure seems to be especially appropriate to this case, as only a rough estimate of the theoretical corrections has been made.

A precise calculation of the bremsstrahlung spectrum emerging from a massive target would require the following:

1. Integration of the thin-target spectrum over the path length of the electrons in the target, considering that, as the exciting electrons penetrate into the target, they soon lose their monoenergetic and monodirectional character due to nonradiative collisions within the target material. In consequence, the observed bremsstrahlung spectrum is not the one produced by a monodirectional, monoenergetic electron beam, as would be the case for a thin target, but is produced by a stream of electrons distributed in energy over a wide spectrum and having a complicated spatial distribution.

2. Correction of this spectrum for the losses in exciting electrons due to backscattering from the target.

3. Correction of this spectrum for the losses in emerging photons due to their absorption within the target material, taking into consideration the complicated spatial distribution of their origins, which is the result of the penetration and diffusion of the exciting electrons within the target. In spite of recent advances [49], the general theory of electron penetration and diffusion has not as yet progressed to a point where a detailed calculation of this type would be feasible. What can be done at the present time is, at best, in the nature of a rough estimate.

3.2. Present Approach

The present estimate followed the steps outlined in section 3.1. As some of the corrections are known only by order of magnitude, it was considered adequate to approximate the bremsstrahlung cross section by a simple analytical expression, and to carry out all integrations by graphical means. Following is a brief review of the procedure.

a. Bremsstrahlung Cross Section

According to theoretical considerations [50] electrons penetrating matter diffuse considerably before losing a substantial fraction of their energy. In fact, in a heavy element such as tungsten, they become practically completely isotropic before losing 10 percent of their initial energy.

It thus seemed reasonable to assume in this first

approximation that the bremsstrahlung was emitted by an isotropic distribution of electrons. Furthermore, even though the energy losses within the target are discontinuous, it is for the present purpose justified to carry out the integration as if the energy were a smooth function of path length. Then, if $i(E,k)dk$ is the nonrelativistic rate of bremsstrahlung emission per unit path length per unit solid angle, averaged over all directions, in the photon-energy interval between k and $k+dk$ (E being the exciting electron energy), the integral over the electron tracks in the target takes the form

$$I(E_0,k) = \int_{s=s_k}^{s_0} i(E,k) ds = \int_{E=k}^{E_0} i(E,k) (ds/dE) dE, \quad (6)$$

where s is the depth of penetration, E is a function of s , and E_0 is the energy of the electrons before their penetration and diffusion into the target. For the actual calculations, values for $i(E,k)dk$ as obtained from the numerical computations of Kirkpatrick and Wiedmann [51] (which are based on the Sommerfeld formula) were used. They could be approximated by a function of the form

$$i(E,k) = \frac{1}{E} [a - b \ln(k/E)].$$

If $i(E,k)$ is in units of 10^{14} kev per steradian per milliampere per minute per unit kilo electron volt range per centimeter, and k and E are in kilo electron volts, the constant a is approximately equal to 16.7 and b is approximately 4.64. The reciprocal stopping power ds/dE was calculated with the aid of the Bethe-Bloch formula [52].

b. Correction for Backscattering of Electrons

The electron backscatter from the target was estimated, using Bothe's experimental results that give the probability $\beta'(E/E_0)d(E/E_0)$ for an electron of known initial energy E_0 and of known direction, to be backscattered with a known fraction E/E_0 of its primary energy [53]. For scattering materials of high atomic number and for a scattering angle of $50 \pm 15^\circ$, Bothe obtained for this probability a function that increases first slowly with increasing values of E/E_0 , then rises sharply to a maximum for E/E_0 somewhat above 0.9, and finally falls off to zero for $E/E_0=0.1$. A graphical integration of this function yields $\beta(E/E_0)$, the fraction of the total number of electrons, averaged over all angles, which is backscattered with energies between a given energy E and the maximum energy E_0 . According to Bothe, about 75 percent of the electrons incident on a block of tungsten are scattered back. This agrees very well with the observations of Seliger [54]. It was therefore decided to use for the present estimate Bothe's backscattering results by applying a correction term $1-\beta(E/E_0)$ to the integrand of eq (6). The equation then becomes

$$I'(E_0,k) = \int_k^{E_0} i(E,k) (ds/dE) [1-\beta(E/E_0)] dE. \quad (6a)$$

If $f_k(x)dx$ represents the fraction of the radiation of energy k produced at a depth x in the target measured in the direction of electron incidence, the fraction of the photons of energy k leaving the target is given by $\int_0^\infty f_k(x) dx \exp[-\mu(k)x/\tan \theta]$, where $x/\tan \theta$ represents the layer that the photons have to traverse when leaving the target, as seen by inspection of the insert in fig. 2. The integral can be written in the form $\exp[-\mu(k)\langle x \rangle_k/\tan \theta]$, where $\langle x \rangle_k$ is a suitable average of x over the function $f_k(x)$. Although this function is not well known, the average value $\langle x \rangle_k$ has been estimated, utilizing some general knowledge recently gained about the actual depth of electron penetration and bremsstrahlung production [49]. For the present approximate calculations, the value used for this average is one-fifth of the total electron range $p = \int_0^\infty (ds/dE) dE$, for all values of k .

The fully corrected spectrum was therefore represented by:

$$I''(E_0,k) = \exp[-\mu(k)p/5 \tan \theta] \int_k^{E_0} i(E,k) (ds/dE) [1-\beta(E/E_0)] dE. \quad (6b)$$

Essentially, this amounts to the assumption of $f_k(x) = \delta(x-p/5)$. This approximation is only adequate for $\mu(k)p \ll 1$. For $\mu(k)p \gg 1$ this exponential correction factor no longer holds, and the photon correction is approximated by $[f(0) \tan \theta]/\mu$.

For the present calculations, eq (6b) was used. The absorption-correction term was evaluated by setting the average depth of penetration ($p/5$) equal to 7.53×10^{-4} cm for 100-kev electrons and to 2.29×10^{-4} cm for 50-kev electrons. The total absorption coefficient for tungsten was obtained from current tables [48].

4. Comparison of Experiment and Theory

In order to facilitate a comparison between theory and experiment, the theoretical spectra corresponding to eq (6), (6a), and (6b) are shown in figures 11 and 12, along with the experimental photon-energy spectra. The experimental spectra were obtained from the fully corrected number-of-photons spectra of figures 9 and 10 by weighting the ordinates by the respective photon energies and dividing by 2.92×10^{-5} , which is the solid angle in steradians subtended by the spectrometer.

Considering the roughness of the theoretical estimate, the over-all order-of-magnitude agreement is quite good. There is, however, a remarkable difference in shape between the calculated and the measured spectra, the peak of the measured spectra in the neighborhood of 30 kev giving the impression of being superposed upon a continuous spectrum similar in shape to that of the calculated one. Figure 8 shows clearly how the 30-kev peak becomes

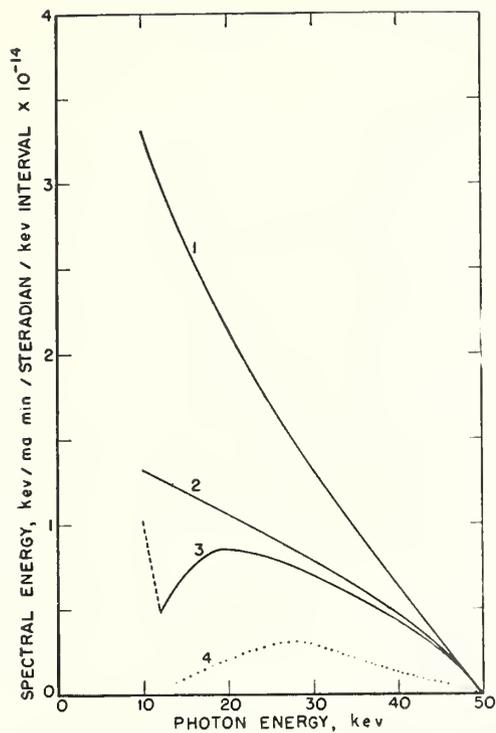


FIGURE 11. Comparison of experiment and theory, 50-keV bremsstrahlung.

1, No correction for electron backscatter or photon absorption; 2, theoretical thick-target spectrum corrected for electron backscatter; 3, theoretical thick-target spectrum, fully corrected; 4, experimental spectrum.

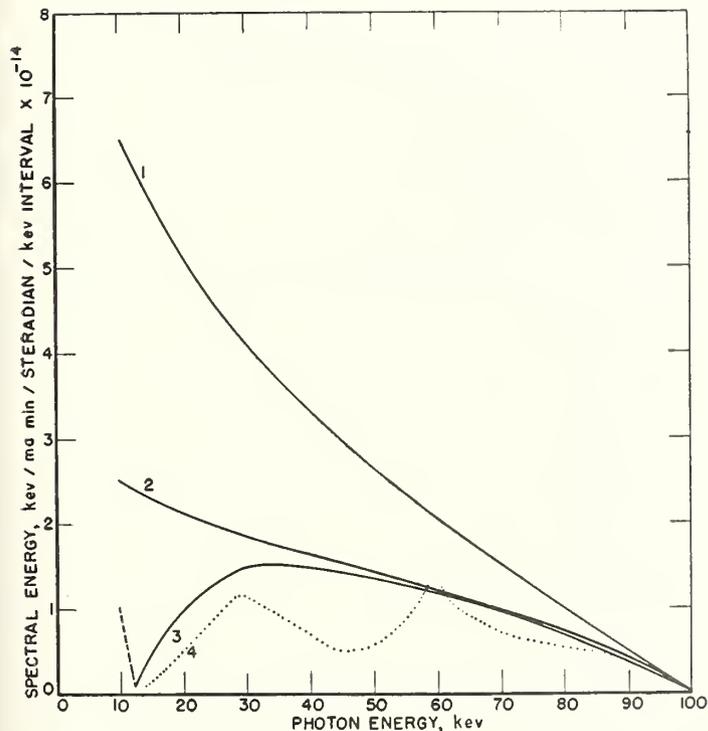


FIGURE 12. Comparison of experiment and theory, 100-keV bremsstrahlung.

1, No correction for electron backscatter or photon absorption; 2, theoretical thick-target spectrum corrected for electron backscatter; 3, theoretical thick-target spectrum, fully corrected; 4, experimental spectrum.

more and more intense as compared with the rest of the spectrum as the exciting potential is increased. As pointed out in section 2.7, the pronounced 30-keV peak was found with two different detectors and two X-ray tubes of different makes and inherent filtrations. Even if iodine K escape had not been found negligible with the detectors employed, the peak could not have been attributed to iodine K escape from the tungsten-fluorescence lines for two reasons: First of all, an approximate calculation of the iodine K escape expected from a 59.5-keV line would amount to only 4 percent of the total intensity of this line. The observed intensity increase above what is expected from theory, although at the right energy for the iodine K escape from the tungsten K-fluorescence lines, is too pronounced. Second, and quite convincingly, spectra determined experimentally behind varying thicknesses of different absorbers showed that a sufficient amount of filtration removed the spectral peak in the neighborhood of 30 keV almost completely, although it absorbed only a comparatively small fraction of the tungsten K fluorescence.

Another point of interest is the steep decrease of the experimental spectral intensity immediately below the 30-keV peak. An inspection of the theoretical curves of figures 11 and 12 shows that the absorption of the bremsstrahlung within the target is predominantly responsible for the peaks in the fully corrected theoretical distributions. This strengthens the suspicion that the shape of the low-energy portion of thick-target bremsstrahlung spectra is strongly influenced by the choice of the target material. Thus, in the case of a beryllium-window tube with a tungsten target, the effectiveness of the low-absorption window for passing low-energy X-radiation seems to be offset—at least above 12.12 keV, the energy of the tungsten L-absorption edge—by the strong absorption of these radiations within the tungsten target. The present measurements were not carried to low enough voltages to determine conclusively whether or not there is an appreciable rise in intensity below the L-absorption edge of tungsten.

The author is indebted to Professor L. F. Talbott of the Catholic University of America for his interest in the work, and to Evans Hayward of the National Bureau of Standards, whose vast experience was invaluable for the accomplishment of the experiment.

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WASHINGTON, November 22, 1954.

Errata to Accompany NBS Research Paper RP2571

Page	Column	
114	1	Footnote 6, first sentence should read: Since this paper went to press, it has been determined that the spectrometer efficiency below 20 kev had decreased over the months.
114	2	Small print of legends of figures 9 and 10 should read: Long dashes, uncorrected experimental data; short dashes, experimental data corrected for finite spectrometer resolution; solid line, experimental data corrected also for absorption.
115	1	Last sentence before part 3: omit.
116	2	Lines 9, 10, and 11 should read: First of all, the contribution of iodine K-escape to the 30-kev peak would be at most 15 percent of the intensity of a 59.5-kev line.

Standard Ionization-Chamber Requirements for 250- to 500-Kilovolt X-Rays¹

H. O. Wyckoff and F. S. Kirn²

Information is provided on the correction factor required for the determination of the dose in roentgens of 250- to 500-kilovolt X-rays from measurement with a free-air ionization chamber. Special emphasis is placed on the determination of each of the factors so that an over-all accuracy of 0.5 percent can be obtained.

1. Introduction

In 1953, the International Commission on Radiological Units recommended [1] the continued use of the roentgen up to 3 Mev for the measurement of X- and gamma-ray dose. The physical requirements of standard ionization chambers for measuring roentgens in the X-ray region up to 250 kv have already been reported [2,3,4]. The calibration of secondary instruments in at least two laboratories also seems to be in agreement [5] for cobalt-60 and radium, on the assumption of a fixed value for the emission constant (roentgens per milligram hour at 1 cm) of radium. However, there is some question about the value of the emission constant for radium [6,7] when its determination is based upon measurements with small cavity ionization chambers. In addition, many of the commercial secondary instruments show a difference in calibration of the order of 5 to 20 percent between 250-kv X-rays and cobalt, so an interpolation of the calibration for the 300- to 400-kv X-ray therapy range seems risky. Extension of the range of standard instruments, at least up to cobalt energies, therefore seems desirable. The present report contains the design criteria for the construction of a standard chamber for 250- to 500-kv X-rays.

A plan view of one type of a standard free-air chamber is shown schematically in figure 1. A parallel-plate system is contained in a radiation-shielded box, B. A known area of the horizontal X-ray beam is defined by the diaphragm, D, so that a beam of photons passes centrally between the plates. A high potential (field strength of the order of 100 v/cm) on plate H, with respect to the other plates, sweeps out the ionization produced in the air between the plates. The ionization is measured for a length, L , of beam determined by the limiting lines of force to the edges of the collector, C. These lines are made straight and perpendicular to the collector by the guard plates, G, and auxiliary wires or strips, W. The latter are connected to a resistance-dividing network to grade the potential uniformly across the end, top, and bottom gaps between C or G, and H. Generally guard plates are not con-

sidered necessary above and below the collector because there the ionization density is low.

A measurement in roentgens requires, according to the definition [1], that one measure all of the ionization produced in air by the high-speed electrons that are themselves produced within the defined mass of air. Real difficulties arise in the measurement of the ionization as required by the definition, so the principle of electronic equilibrium is always used in practice.

Within a region under uniform irradiation, according to the principle of electronic equilibrium, the ionization produced outside of a specified mass, m , by high-speed electrons generated inside m is compensated by ionization in m produced by high-speed electrons generated outside of m . Thus electrons produced out to a distance from the mass equal to the electron range contribute to the ionization in the specified mass. Therefore in a standard chamber, the guard plates not only must be long enough to eliminate field distortion but also long enough to assure electronic equilibrium in the collecting region. For the latter, a distance equal to half the plate separation has been suggested [4] for X-rays up to 250 kv. However, for the higher-energy photons used here, the range of the electrons in the photon direction may be larger than the sideways range. A determination of the distance required for electronic equilibrium is therefore desirable.

Ionization is produced in the collecting region not only by the primary electrons, but also by electrons produced by secondary (scattered and fluorescent) photons. As this secondary ionization is not included in the roentgen definition, a correction is required to the measured ionization. However, this excess ionization tends to compensate for loss of primary ionization resulting from too small a plate separation and height, so that the required electrode separations are reduced by this effect.

In the calibration of a radiation instrument by a standard chamber, a substitution method is used. The exposure dose rate is determined from measurements of the ionization in the standard chamber, this chamber is removed, and the instrument being calibrated is placed at the position formerly occupied by the aperture of the standard chamber. Therefore, it is necessary to compute the exposure dose rate at the aperture from the ionization measurements. The factors required for such a conversion

¹ Work supported by U. S. Atomic Energy Commission.

² Now with Argonne National Laboratory, Idaho Falls, Idaho.

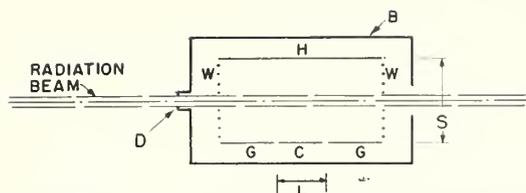


FIGURE 1. Schematic view of free-air ionization chamber showing a horizontal section through the radiation beam and chamber.

B, Shielded box; C, collector (length, L); D, diaphragm; G, guard plates; H, high-voltage plate; W, guard wires or strips; S, plate separation.

include a knowledge of the weighted position of origin of the high-speed electrons relative to the collecting volume and the air absorption between this position and the aperture.

Thus the information required for the proper design of a primary standard chamber is (1) the plate spacing, S (fig. 1), required to assure dissipation of all of the energy of the secondary electrons in the air, (2) the criteria for producing a defined electric field so that this ionization is collected from a known length, L , of the beam, (3) the correction for the ionization produced by scattered photons, (4) the weighted position of origin of the high-speed electrons relative to the collecting region, and (5) the air absorption between this origin and the diaphragm. Data for item (2) have been obtained by Miller and Kennedy for the two-dimensional case [8]. Items (1) and (3) have been considered by Attix and DeLaVergne [4], by Kustner [2], and by Block [13], and item (1) by Kemp [3], for 50- to 250-kv X-rays. The present paper gives information on items (1), (3), (4), and (5) for 250- to 500-kv X-rays and moderate filtration. Work at higher energies is now under way at the Bureau.

2. Apparatus

A vertical, continuously pumped, end-grounded, 59-section X-ray tube was used. It was connected to the ground, center, and top of a 0 to 1,400-kv generator [9]. The potential to the sections was graded uniformly by a series of resistors between the leads from the generator. The 90° beam of X-rays (fig. 2) passed through the wall, W , (about 3-mm copper) of the X-ray tube, by a remotely controlled shutter, S , through a collimator in front of the parallel-plate ionization chamber, and through a monitor ionization chamber, M . A focal-spot size of about 2-cm diameter was obtained in the X-ray tube. Although this spot size was larger than desired, and sometimes shifted during the run, the monitor provided a means of normalizing the parallel-plate chamber readings because it measured most of the beam "seen" by the parallel-plate chamber.

A lead-lined box contained the plates of the ionization chamber and limited the X-radiation measured by the chamber to that coming through the aperture. The box size was determined by pessimistic extrapolation from the requirements for lower energies. The lead protection required in the box was com-

puted according to the method used in NBS Handbook 50 [10] so that the ionization from the leakage radiation would be less than 0.1 percent of the ionization produced by the collimated beam. The front face of the chamber was shielded by 1-in.-thick lead; the sides, top, and bottom by $\frac{3}{8}$ -in.-thick lead; and the back by $\frac{1}{4}$ -in.-thick lead. The aperture was cut in a 2-in.-thick lead diaphragm. A 2-in.-thick lead plug over the aperture of the diaphragm (with the X-ray beam on) experimentally confirmed the adequacy of this protection. The snout on the box was provided to reduce the size of the box because it was anticipated that a scattering diaphragm would be necessary in order to stop radiation scattered from the primary diaphragm from entering the measuring volume. However, subsequent tests indicate, in agreement with tests of Attix and DeLaVergne [11] at lower energies, that this diaphragm is not needed.

The high-voltage plate was a single sheet of aluminum. The collector and field guards were fashioned from a single sheet (90 cm by 90 cm) of colloidal-graphite-coated polyethylene. A collector (30 cm long by 80 cm high) was defined by lines scratched through the graphite with a sharp stylus. This collector was divided into seven 5-cm strips on each side of a central 10-cm strip by lines scratched through the graphite (see fig. 2). Any or all of them could be connected to the ionization-current-measuring instrument. When not so connected, they served as part of the guard system. There was a 5-cm guard strip above and below the collector strip, and a 30-cm-long guard strip in front and in back of the collector strips. The plate separation could be varied from about 15 to 90 cm, and the dimensions of the box permitted a movement of the plate system over a distance of about 1 m along the beam.

The distorting effect of the grounded box on the electric field was reduced by a system of guard strips and wires. As pointed out previously [8], the strips are preferable but wires are required where photon absorption is important. Double wires fastened to the inner and outer edges of the strips were therefore installed over an area of 50 by 50 cm at the front and back of the chamber system (see fig. 3). Strips (3 mm by $3\frac{1}{2}$ cm) were used over the remaining area and the top and bottom. With this system the grounded box produced less than 0.2 percent distortion³ when the plate system was located in the center of the box. To reduce the field distortion when the plate system was used near the front or back of the box, the double wires at both ends of the plate system were temporarily replaced by strips except for a central area of 15 by 50 cm.

A 10-cm-i.d., 1 $\frac{1}{2}$ -mm thick, colloidal-graphite-coated, Bakelite tube was inserted between the plates to eliminate collection of the ionization produced by the primary photons. This tube was coaxial with the beam and extended in front of and behind the

³ The distortion in the electric field caused by the proximity of the lead box was taken as one-half the fractional change in the ionization obtained for the box at ground potential and at the potential of the high-voltage plate.

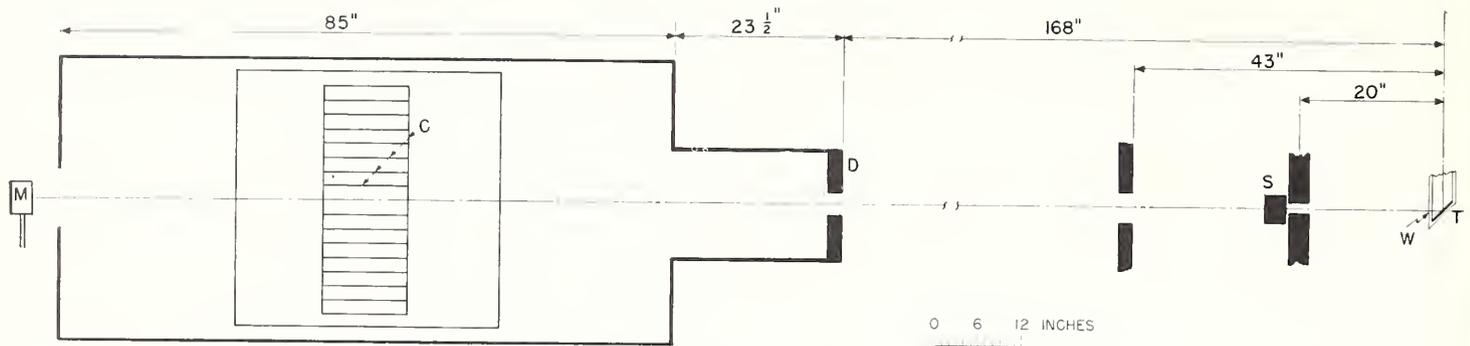


FIGURE 2. Elevation view of experimental arrangement.

T, X-ray target; S, shutter; D, diaphragm; C, collectors; M, monitor.

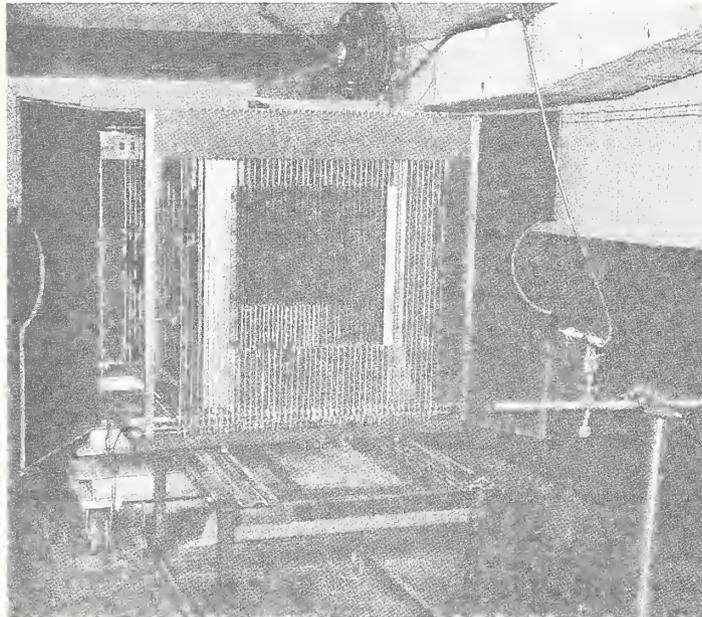


FIGURE 3. Part of free-air chamber showing plate system.

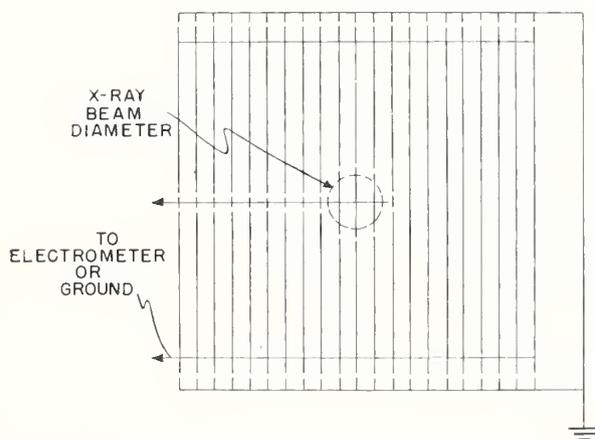


FIGURE 4. Schematic elevation of the center plane of the thread chamber.

Gaps in the straight lines indicate uncoated nylon threads.

plate system. Scribed lines through the colloidal graphite along generatrices of the tube insulated the portions of the tube from each other and permitted them to be electrically connected to the guard wires. With this arrangement, the collected ionization is due only to scattered photons. The tube undoubtedly distorted the electric field in its vicinity but the total scattered photon contribution was small (less than 1 percent of the electron contribution from primary photons), so this distortion was assumed to be negligible.

A separate arrangement, similar to that used by Failla [12] to investigate the "electron cloud," was used to measure the growth of electronic equilibrium along the beam. A grid chamber of colloidal-graphite-coated nylon thread (~ 0.2 -mm diameter) forming 5 parallel, 90 by 90-cm planes was placed in the shielded box. The thread spacing was about 1 cm in each plane, and the planes were spaced about every 1.6 cm and perpendicular to the beam. The center plane (fig. 4) was graphite-coated to form two collectors insulated from each other and from ground by uncoated nylon thread. One of these collectors was slightly larger than the photon beam and concentric with it. The other included the rest of the plane. The planes immediately on each side of this center plane were connected to the collecting potential (90 v). The outer two planes of thread were connected to ground and used to shield the collecting system from the grounded box.

One sheet of $1\frac{1}{2}$ -mm-thick 90-cm-square conducting plastic was placed in front of and parallel to the plane of the grid chamber. A second was placed behind the chamber. Each sheet had a center hole slightly larger than the photon beam and each could be moved along the beam. As each of these sheets was thicker than the electron range, the ionization measured was produced to a first approximation⁴ by electrons from the air in the space between the sheets. The growth of ionization was explored by changing the location of the sheets.

3. Procedure

In order to eliminate the effects of X-ray output variation, simultaneous ionization measurements were made with the parallel-plate chamber and the

⁴ The large size of the hole makes the exact position somewhat ambiguous.

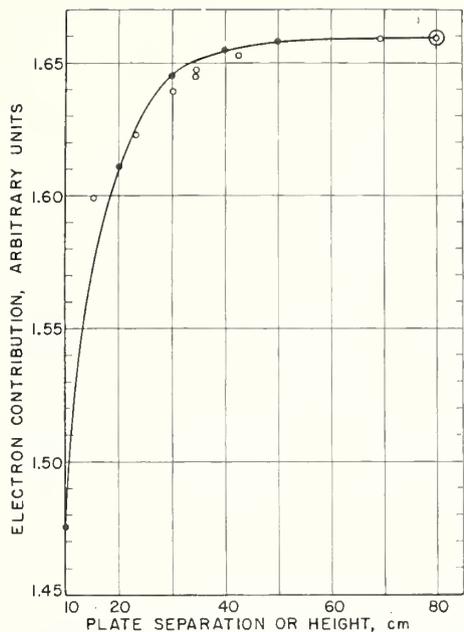


FIGURE 5. Ionization collected in air from 400-kv X-rays as a function of plate separation with a fixed height, and of height with a fixed plate separation.

○, Height; ●, plate separation.

monitor. The charge from each was collected on a capacitor and the resulting potential measured under a null condition by a vibrating-reed electrometer. The ratio of the voltage on the parallel-plate capacitor to that on the monitor capacitor was proportional to the parallel-plate current for a fixed amount of monitor ionization. This averaging of the parallel-plate current and the normalization by means of the monitor overcame the undesirable fluctuations in the X-ray output. Data were obtained at each 50 kv in the 250- to 500-kv range.

The current collected by the collector plate is made up of two components, that part due to the ionization from high-speed electrons that are produced by the primary photons (primary contribution), and that part due to ionization from high-speed electrons produced by scattered and fluorescent photons (secondary contribution). The sum of the two contributions and the secondary contribution alone are measured [2, 4, 13] so that the primary contribution can be determined by subtraction. In the present experiment, this sum was determined both by measuring the ionization collected by all of the collector strips (80 cm high) for different plate separations, and by measuring the current collected by each strip for a fixed 80-cm plate separation. The ionization collected by all of the strips at a given plate separation, a , should be equal to the ionization collected at 80-cm plate separation by all of the strips up to a height equal to a . Figure 5 shows a representative comparison of the results by the two methods. Generally, the agreement was within 0.5 percent. However, the ionization density decreases rapidly with distance from the photon beam, so the individual strip method gives a more accurate measure of this density function. Therefore, the remainder of the data presented here were obtained

by the strip method. Similar measurements were made with the Bakelite tube inserted to obtain the secondary contribution.

The current to each of the collectors of the grid chamber was measured separately. The one not used for collecting ionization was grounded. The variation of the ionization was determined as a function of the position of the conducting plastic sheets with respect to the grid chamber.

All measurements were obtained at room temperature and pressure, which averaged about 26° C and 760 mm Hg.

4. Results

Figure 6 shows the primary contribution for 5-cm-high layers of air at different vertical distances from the beam. The fractional loss in primary photon ionization (electron loss) at a particular height is obtained by summing the contributions from infinite plate height (obtained by linear extrapolation of the curves of fig. 6) to the plate height in question and dividing by the sum of all of the contributions out to infinity.

The diameter of the photon beam obviously affects the loss for a given plate separation. In figure 7 the percentage of electron loss is plotted against the plate separation minus the penumbra diameter (6.3 cm) at the center of the collecting region. On the 250-kv curve are plotted data taken from results of Attix [11],⁵ where the penumbra was about 1.8 cm in diameter and the radiation filtration was approximately 1 mm of copper plus 4 mm of aluminum. This approximate method of treating the beam diameter seems to be adequate at least for these data, as the agreement of the electron contributions for the two experiments is generally within 0.1 percent. Furthermore, the difference is in the expected direction because the higher filtration used in the present experiment results in a smaller number of high-energy photoelectrons.

In order to compare the present data with those published earlier by Block [13] and to provide more easily usable data for other chamber configurations than the parallel-plate type, the electron loss and the scattered photon contribution were computed for cylindrical coordinates. Obviously the ionization collected from a ring of a given radius will be independent of angle, so only one variable is required. The contribution to each 5-cm ring was obtained from the results shown in figure 6 by the method previously outlined [4]. Figure 8 shows the electron loss per 5-cm-thick ring obtained at different inner radii. Here again the losses may be summed from infinity to a given radius to obtain the loss beyond that radius. The results are shown in figure 9, where the beam size has been reduced to zero by subtracting the beam radius.

Figure 9 also shows some points computed from the data of Block [13] at 400 kv and a filtration of

⁵ These plotted data are slightly different from those in table 2 of reference [4] because the plotted data are for an effectively infinite plate height.

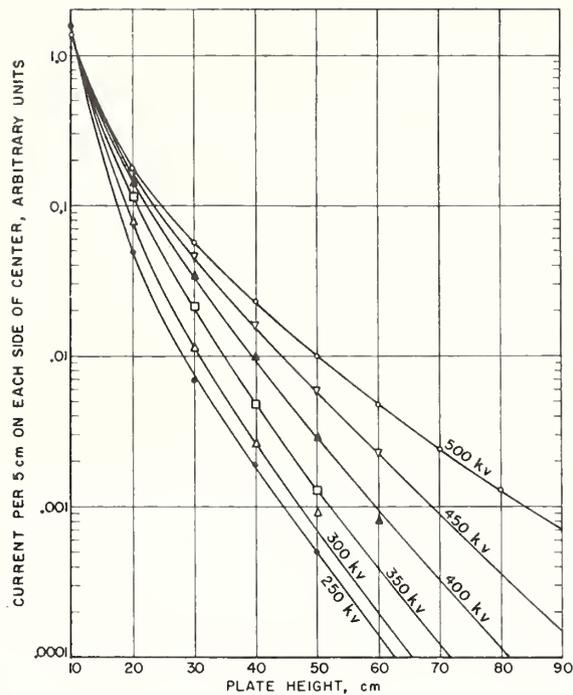


FIGURE 6. Primary electron-produced current per 5 cm of height of collector at different vertical distances from the beam and at various X-ray tube potentials.

The beam penumbra diameter was 6.3 cm in the middle of the collecting region.

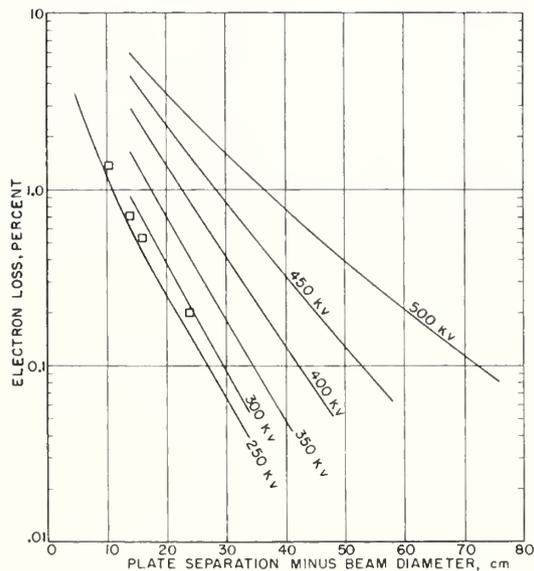


FIGURE 7. Percentage of primary electron ionization loss for different plate separations and a zero-diameter X-ray beam.

□, Results of Attix for 250 kv.

2.55 mm of tin plus 0.5 mm of copper plus 5.0 mm of aluminum. These points were obtained with a different filtration than that used for the present data; some of these had to be determined from small-scale drawings, the scattered photon contribution had to be computed from the present data, and the beam diameters were assumed to be the same for Block as for the present work. It is thus not surprising that these points differ by up to 0.5 percent from the present work.

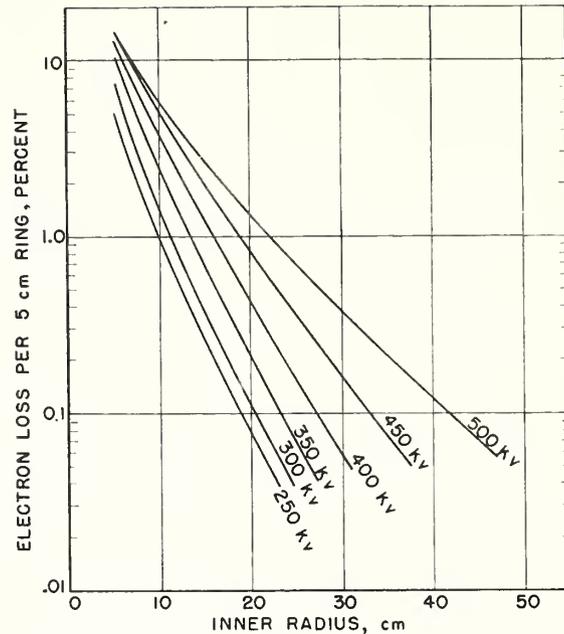


FIGURE 8. Percentage of primary electron ionization loss per 5-cm ring for different inner radii from the beam.

The beam penumbra diameter was 6.3 cm at the center of the collecting region.

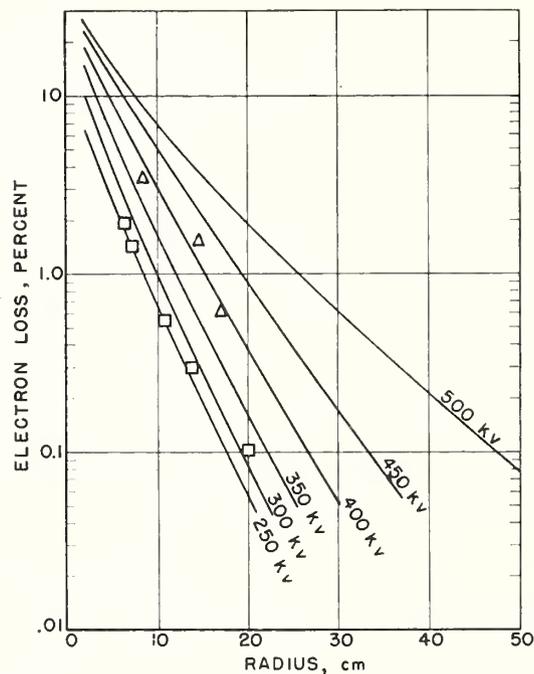


FIGURE 9. Percentage of primary electron ionization loss beyond different radii from the X-ray beam.

Results corrected to zero beam diameter. □, Data of Attix [4] at 250 kv; △, data of Block [13] at 400 kv.

Figure 10 gives the radial distribution of the ionization from the secondary photons obtained from measurements made with the Bakelite tube in a fashion similar to that for the primary contribution. The contribution is given as a percentage of the total ionization from primary electrons. As the curves for various X-ray-tube potentials have a similar shape, only the 250-kv curve is shown. The contribution at 300-kv is obtained by multiplying the

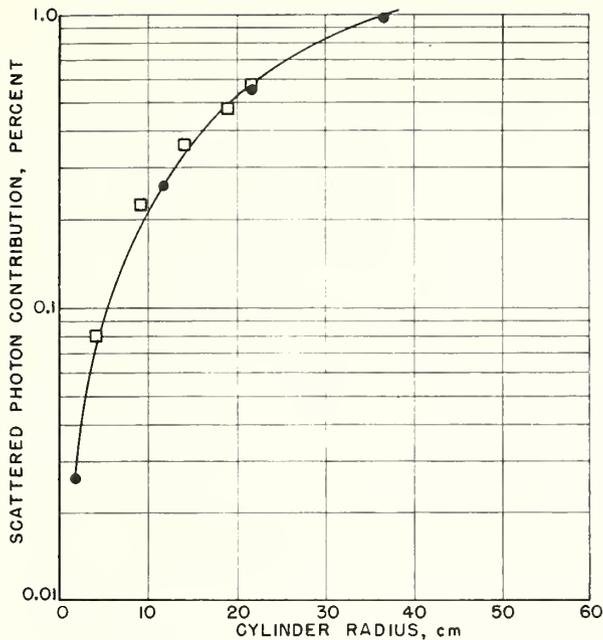


FIGURE 10. Contribution of secondary photon ionization in percentage of total primary electron ionization for different radii from a 250-kv zero-diameter X-ray beam.

●, Present data; □, data of Attix [11]. For 300 kv, multiply the ordinate by 0.9; for 400 and 500 kv, multiply by 0.8.

ordinates of the curve by 0.9; at 400 and 500 kv, by 0.8. It is interesting to note that the data obtained by Attix [11] at 250-kv on a much smaller chamber give very nearly the same results. Here again the abscissa has been corrected for beam size by subtracting the beam radius.

The following table shows the air absorption per meter at various tube potentials obtained from ionization-chamber measurements with the chamber at the front and at the back of the box. Repeat measurements agreed to within ± 0.2 percent. They also agreed to within 0.2 percent with the results of Block.

Tube potential	Air absorption
Kv	%/m
250	1.7
350	1.7
500	1.3

Figure 11 shows a typical "growth-of-electronic-equilibrium" curve for the ionization collected by the outer electrode of the thread chamber with 500-kv X-rays. The charge collected by the center electrode is 0.153 arbitrary unit, and is independent of the position of the plastic sheets. When the sheets are at a large distance from the collecting electrodes the total (equilibrium) ionization collected is $0.0444 + 0.032 - 0.153 - 0.0296$, or about 0.200 arbitrary unit. From these data for 500 kv, it is seen that $(0.153 + 0.029)/0.200$, or about 90 percent,

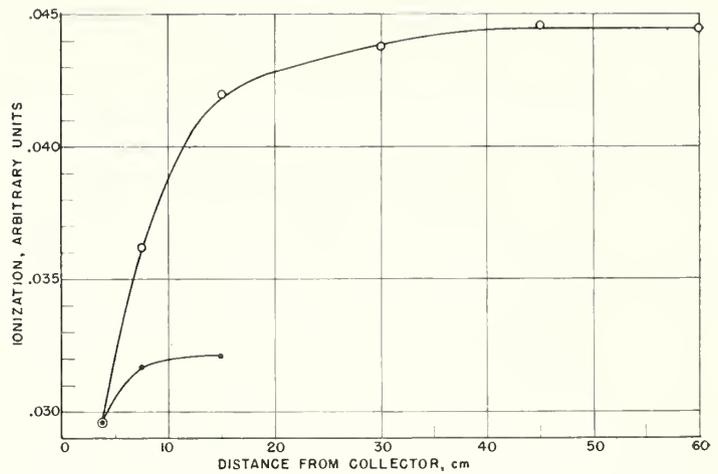


FIGURE 11. Results with the grid chamber with 500-kv X-rays.

The center electrode collected an ionization current of 0.153 arbitrary units. The abscissa gives the distance from one plastic sheet to the collector plane with the other sheet at 3.6 cm from the collector. Data for different distances of the front sheet are indicated by ○ and for the back sheet by ●.

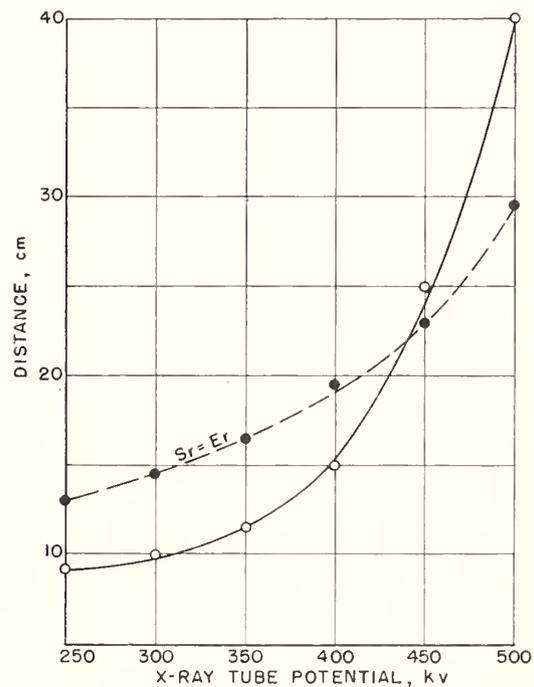


FIGURE 12. Approximate distance required for electronic equilibrium with medium filtration, and radii from photon beam at which the loss of primary electron ionization is compensated by gain of secondary radiation ionization.

○, Distance; ●, radii.

of this ionization results from electrons effectively originating within a few centimeters of the collecting region. Approximately 1 percent of the equilibrium ionization is produced by electrons originating up to about 15 cm behind, and about 90 percent by electrons originating up to 40 cm in front of the collecting region. The flux of gamma rays producing these high-speed electrons of course varies over the distance (40 + 15 cm) because of air absorption.

To a first approximation the absorption of X-rays in the air between the diaphragm and the origin of

the high-speed electrons may be computed from the air-absorption coefficient and the distance between the diaphragm and the collecting region. A correction to this distance may be obtained from curves such as those in figure 11. For 500-kv X-rays the correction is

$$\frac{1}{0.200} \left\{ - \left[+ (0.0444)(40 - 3.7) - \int_{3.7}^{40} x_f di \right] + \left[(0.032)(15 - 3.7) - \int_{2.7}^{15} x_b di \right] \right\},$$

where 0.200 is the equilibrium ionization and di is an increment of ionization at a distance x_f in front of the collector and x_b behind the collector. On substituting values from the curve, this distance correction is found to be less than 1 cm. As 8 cm of air attenuates the photon beam by only the order of 0.1 percent, the actual displacement is negligible. For all lower X-ray potentials the displacement is smaller. Therefore, the air-absorption correction in the 250- to 500-kv range should usually be computed from the center of the collector to the aperture.

Figure 12 shows the approximate distance required for electronic equilibrium. Also plotted are the distances equal to the radii from the beam, at which the loss of ionization from the primary electrons is just compensated by the gain of ionization from the secondary radiation. Thus, a collector-to-aperture distance, equal to the radius for compensation, is adequate for moderately filtered X-rays of 400 kv and below but may be insufficient for 450 and 500 kv. It is seen from figure 11, for instance, that a decrease in the collector-to-aperture distance from 40 to 29.5 cm results in an error of about $(0.0444 - 0.0438)/0.200$, or 0.3 percent.

5. Summary

The data required for the design of free-air standard chambers for measurement of 250- to 500-kv X-rays in roentgens have been obtained. The internal consistency of these data indicates that the errors of plate spacing can be estimated to about 0.2 percent. However, the much closer agreement between these results and those of Attix indicates that the uncertainty is probably less than 0.2 percent.

The assistance of George Dempsey, who helped set up the equipment and operated the X-ray unit, and of F. H. Attix, who participated in many of the technical discussions during the program of the experiment, is gratefully acknowledged.

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WASHINGTON, April 6, 1956.

Cavity Ionization as a Function of Wall Material

Frank H. Attix,¹ LeRoy DeLa Vergne, and Victor H Ritz

A study has been made of the ionization within a flat cavity chamber under irradiation by X- and gamma rays in the energy region 38 to 1,250 kilovolts effective (kev). Chamber walls were made of carbon, aluminum, copper, tin, and lead, and the wall separation was varied from 0.5 to 10 millimeters. Results are compared with cavity theory.

1. Introduction

In 1953 an experimental study of cavity ionization² was carried out at the Bureau by means of a chamber of the parallel-plate type, having walls of C, Al, Cu, Sn, or Pb, which could be varied in separation from about 0.5 to 10 mm. The gas between the plates was air at room pressure and temperature. Relative ionizations were compared when the chamber was irradiated by heavily filtered X-rays of 50 to 250 kv, and by γ -rays from Au¹⁹⁸ (411 kev), Cs¹³⁷ (670 kev), and Co⁶⁰ (1,250 kev). For the X-rays, measurements of ionization were also made with a free-air chamber.

The results were presented in informal communications which were intended for limited distribution and were not, therefore, generally available. The work has not been published heretofore because, in retrospect, it was regarded as preliminary in nature and because the chamber design was not ideally suited to the problem.

The principal objections to the design of the experimental ionization chamber are that: (a) It should have side walls to eliminate the escape of electrons from between the plates; and (b) varying the gas-pressure instead of the separation of the plates would give the experiment greater accuracy and simplicity. Several experiments have since been done here and elsewhere [1 to 4]³ employing pressure-variation in closed cavities, and there can be no doubt as to the inherent advantages of the method.

In spite of its shortcomings, however, the present experiment does yield some information about cavity ionization, particularly for small wall separations where the electron losses are negligible. Furthermore, the apparatus and results have recently been referred to in several published papers [2, 4, 5, 6, 7], indicating a general interest which makes the present publication of the work worthwhile.

Measurements with the γ -rays from Co⁶⁰ have recently been repeated to determine the degree of the electron losses at that energy, and to eliminate, by increasing the filtration of the γ -ray beam, the effect of low-energy scattered radiation originating in the source and its housing.

2. Experimental Apparatus

2.1. Ionization Chamber

Figure 1 shows a section through the chamber, indicating its design. The collecting volume was 5 cm in diameter, and the irradiated area about twice this size, providing some compensation for electron losses by irradiation of the guard-ring area. Connection with the collecting electrode was made through a fine wire embedded in the rear supporting wall. This wall was constructed of two sheets of polyethylene, bonded together (with the wire between) by heating, to give a total thickness of 1.8 mm.

Thin foils were cemented to the polyethylene and the groove cut afterward with a lathe tool. Thicker metal walls were precut into collector and guard ring, then attached to the polyethylene by means of double adhesive tape,⁴ which gave a very strong bond. It

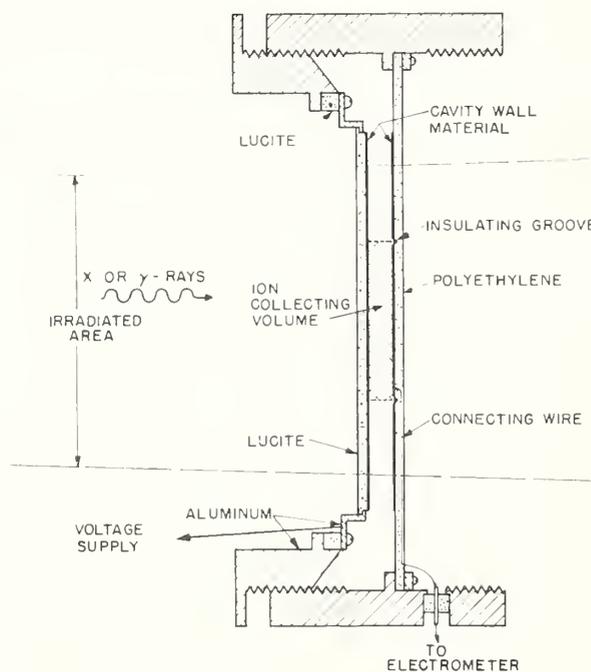


FIGURE 1. Cross section of experimental ionization chamber.

¹ A portion of this work was submitted by F. H. Attix in partial fulfillment of the requirements for the master of science degree at the University of Maryland.

² This work was supported by the U. S. Army Signal Corps, Fort Monmouth, N. J.

³ Figures in brackets indicate the literature references at the end of this paper.

⁴ "Scotch" Tape No. 400, manufactured by Minnesota Mining and Mfg. Co.

was found necessary to fill the 0.25-mm-wide gap between the guard ring and collecting electrode with a strip of polyethylene to avoid ion collection from that volume.

The front ⁵ chamber wall was supported on a screw of 1-mm pitch, to allow variation of the wall separation. The collecting voltage was applied to this wall; ± 22 v/mm was found to be an adequate gradient for saturation.

The effective position of the chamber for a given separation was taken to be midway between the front and back walls.

2.2. Wall Materials

Table 1 gives the thicknesses of the walls used for the various energies. In each case the thickness is greater than that necessary for electronic equilibrium. Thicknesses of supporting materials, if present, are also shown. Attenuation and scattering from both the front and back walls were corrected for in the usual manner by varying the thicknesses (always maintaining at least equilibrium thickness) and extrapolating the observed ionization to zero wall thickness. In most cases this correction was only a few percent, but for low X-ray energies the attenuation in the front walls of high-atomic number became larger, reaching about 60 percent for the lead wall with 38-kev X-rays.

Spectroscopic analysis of the wall materials indicated that the impurities present would alter the ionization by less than 1 percent for any of the radiations used in the experiment.

⁵ The chamber was ordinarily placed with this wall toward the source. No difference in response was observed whether the front or back of the chamber faced the source, if appropriate wall-attenuation corrections were applied.

2.3. Measurement of Ionization Current and Wall Separation

Ionization currents were measured by means of a vibrating-reed electrometer employed in a null method. Currents larger than 10^{-12} amp were passed through high megohm resistors and the IR drop balanced by a calibrated potentiometer. Smaller currents were measured by observing the rate of charge of a 10^{-11} -f calibrated capacitor.

Each current measurement was made with both polarities and averaged to eliminate the effects of: (a) Extracamerai ionization; (b) radiation-induced leakage of current through the polyethylene rear supporting wall; and (c) current of energetic electrons crossing the chamber from one wall to the other. Positive and negative currents were found to agree closely except for separations less than 1 mm, where they diverged by as much as a few percent.

The plate separation was measured electrically (in the absence of radiation), employing the circuit shown in figure 2. The two potentiometers were

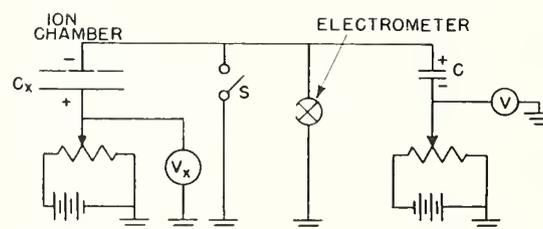


FIGURE 2. Circuit used in determining the separation of the chamber walls by measurement of the capacitance between them.

TABLE 1. Thicknesses of wall materials

Gamma or X-ray effective; ¹ photon energy	Graphite		Al		Cu		Sn		Pb	
	Front wall	Rear wall	Front wall	Rear wall	Front wall	Rear wall	Front wall	Rear wall	Front wall	Rear wall
38 <i>ker</i>	307 mg/cm ²	307 mg/cm ² +0.072 in. polyethylene.	109 mg/cm ² +0.062 in. lucite.	109 mg/cm ² +0.072 in. polyethylene.	23.5 mg/cm ² +0.062 in. lucite.	68 mg/cm ² +0.072 in. polyethylene.	10 mg/cm ² +0.062 in. lucite.	57 mg/cm ² +0.135 in. polyethylene.	60 mg/cm ² +0.062 in. lucite.	50 mg/cm ² +0.072 in. polyethylene.
70	do	do	do	do	do	do	do	do	do	Do.
118	do	do	do	do	65.5 mg/cm ² +0.062 in. lucite.	do	57 mg/cm ² +0.135 in. polyethylene.	do	do	Do.
169	do	do	do	do	do	do	do	do	do	Do.
206	do	do	do	do	do	do	do	do	124 mg/cm ² +0.062 in. lucite.	124 mg/cm ² +0.072 in. polyethylene.
411	do	do	do	do	925 mg/cm ²	925 mg/cm ² +0.072 in.	770 mg/cm ²	770 mg/cm ² +0.072 in. polyethylene.	872 mg/cm ² +0.062 in. lucite.	872 mg/cm ² +0.072 in. polyethylene.
670	do	do	do	do	do	do	do	do	do	Do.
1,250	do	do	437 mg/cm ²	437 mg/cm ² +0.072 in. polyethylene.	do	do	do	do	do	Do.

¹ Determined by attenuation in copper.

varied to balance a charge on C by an equal charge on C_x (the chamber capacitance), the balance being indicated by the vibrating-reed electrometer. This procedure was repeated at each of several settings of the chamber screw, using the same setting of potential V each time (i. e., constant charge). If V_x is plotted versus the indicated settings of the screw, a linear relationship results, intersecting the $V_x=0$ axis at the screw setting where the wall separation is zero. This can be shown as follows:

$$q = \text{constant} = VC' = V_x C_x$$

and

$$C_x = \frac{KA}{d}$$

where:

- V = voltage on known capacitor,
- C = capacitance of known capacitor,
- V_x = voltage applied to chamber wall,
- C_x = capacitance between walls of chamber,
- K = constant,
- A = effective area of collecting electrode, and
- d = wall separation.

Combining the two equations, we have

$$d = \left[\frac{KA}{q} \right] V_x$$

where the bracket term is the slope of the observed plot of V_x versus screw setting. Thus the separation is obtained for any measured value of V_x by multiplying it by the previously determined slope, which need be measured only once for each rear-wall assembly.

Note that the actual value of C is not needed in finding the separation. However, if it is known, one can solve for A and thence the diameter of the collecting electrode. This was found to agree with the directly measured diameters to within 1 percent.

3. X-ray Measurements

3.1. Source of X-rays

A Westinghouse 250-kv tube was operated with a well-stabilized constant potential generator to provide the X-ray source. The beam was collimated to give a diameter about twice that of the collector at 1-m distance, where the chamber was located. The kilovoltages and filtrations used are given in table 2, and are similar to those of Ehrlich and Fitch [8].⁶ The effective energies were obtained from attenuation data in copper, referring to the attenuation coefficients of White [9]. These X-ray spectra are, of course, not monochromatic, but consist of a spread of photon energies [8]. Further narrowing of the spectra by increased filtration would have made the beam intensities too low for accurate measurements.

⁶Note that the filtration data given for 150 kv in this reference are in error, according to private communication with the authors.

TABLE 2. X-ray potentials and filtrations

Constant potential applied to X-ray tube	Added filtration ¹	Effective X-ray energy ²
<i>kev</i>	<i>mm</i>	
50	0.125 Pb.....	38
100	.52 Pb.....	70
150	1.53 Sn+4.00 Cu.....	118
200	0.70 Pb+4.00 Sn+.59 Cu.....	169
250	2.70 Pb+1.00 Sn+.59 Cu.....	206

¹ The inherent filtration of the X-ray tube was equivalent to 3-mm Al.

² As determined from attenuation measurements in copper.

Uniformity of the beam over the area of the chamber was ascertained by photographic densitometer measurements.

3.2. Free-Air Chamber

The exposure-dose rate (in roentgens per unit time) of the X-rays in the plane of the experimental chamber was measured by replacing that chamber with a free-air chamber having a defining diaphragm 8 mm in aperture diameter. The 12-cm plate separation of this chamber [10] was inadequate for the heavily filtered X-rays used, and it was, therefore, calibrated against the NBS 250-kv standard free-air chamber [11].

3.3. Results with X-rays

Figures 3 to 7 show the curves obtained for the ratio of ionization density in the experimental chamber to that in the calibrated free-air chamber, as a function of the wall separation in the former. Corrections for wall attenuation and scattering in the experimental chamber walls have been included.

The shapes of the curves are influenced by two effects: (a) The transition from wall-dependent ionization at small separations to air-dependent ionization at large separations, and (b) the loss of electrons out the edges of the chamber, an effect that becomes more pronounced at large separations and high X-ray energies. For walls of atomic number higher than air, the flux of electrons generated within the wall material will be greater than that in air because of the greater coefficient for absorbing energy from X-rays, (due to photoelectric effect), and because of the lower electron stopping-power (in cm²/electron) of the wall material. Furthermore, the increased reflection of electrons from walls of high-atomic number results in larger electron losses from the edge of the chamber. Thus one expects the ionization to rise as the wall separation is decreased in such a chamber, as indicated in figures 4 to 7. It is interesting to note the somewhat steeper descent of the 38-kev curve for Sn (fig. 6), as compared with that for Pb (fig. 7). (At this energy the edge losses are negligible because of the short ranges of the electrons present.) The K -edge for photoelectric absorption in Sn is located at 29 kev, giving a large component of photoelectrons with energies of the order of 9 kev (see table 5). The range of

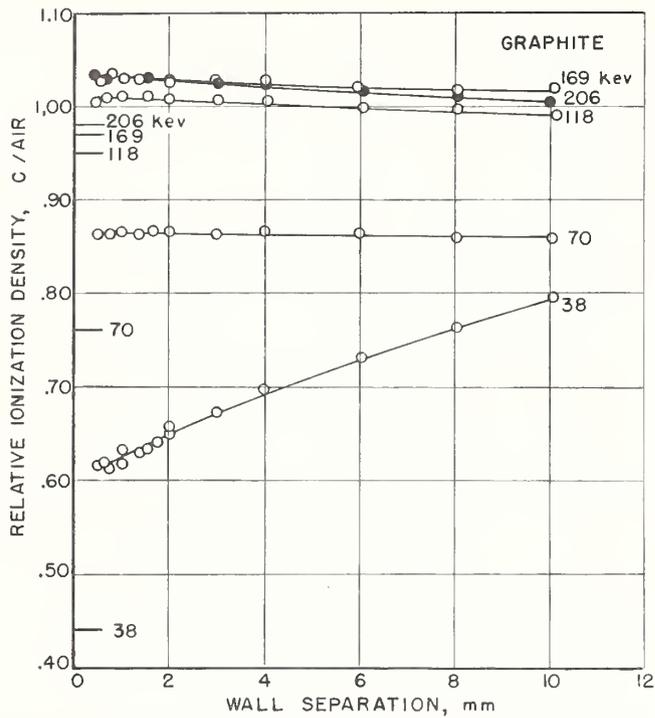


FIGURE 3. Curves showing the ratio of ionization per unit volume in the experimental chamber to that in the free-air chamber, as a function of the separation of the walls in the experimental chamber.

The marks at the vertical axis, labeled with the appropriate values of X-ray energy in kilovolts effective, indicate the corresponding theoretical ratios from the Bragg-Gray relation.

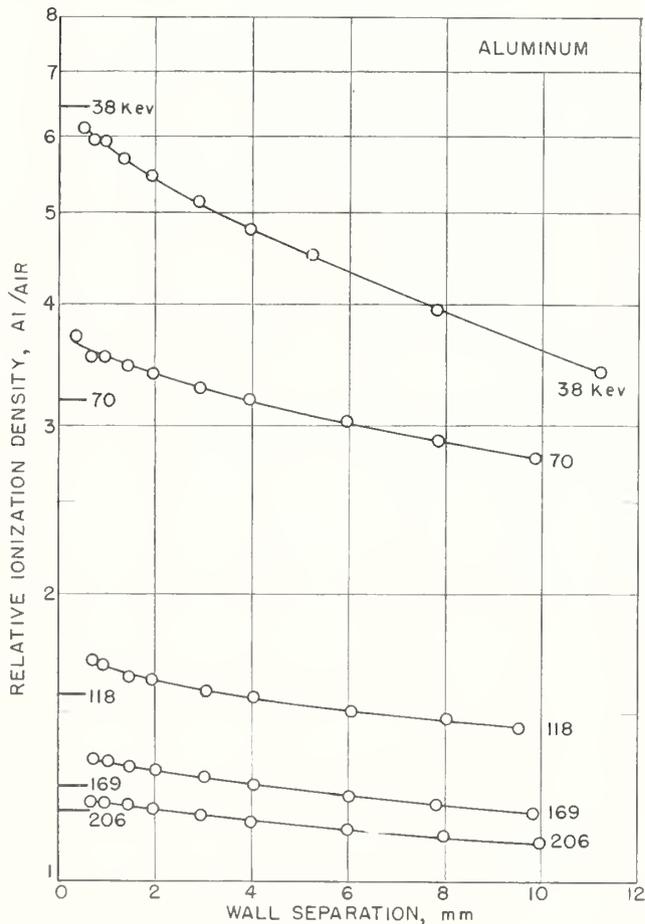


FIGURE 4.

(See caption for fig. 3)

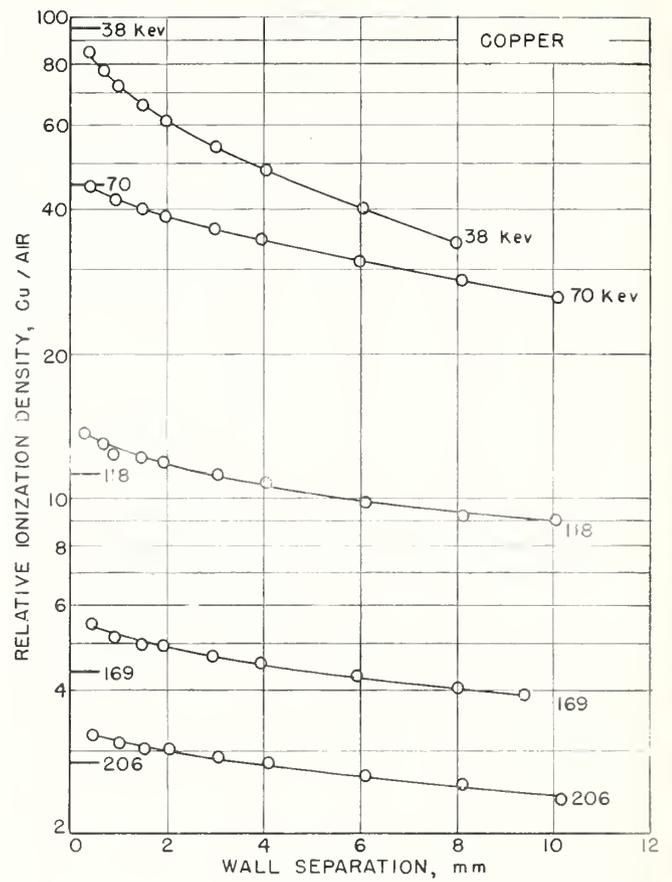


FIGURE 5.

(See caption for fig. 3)

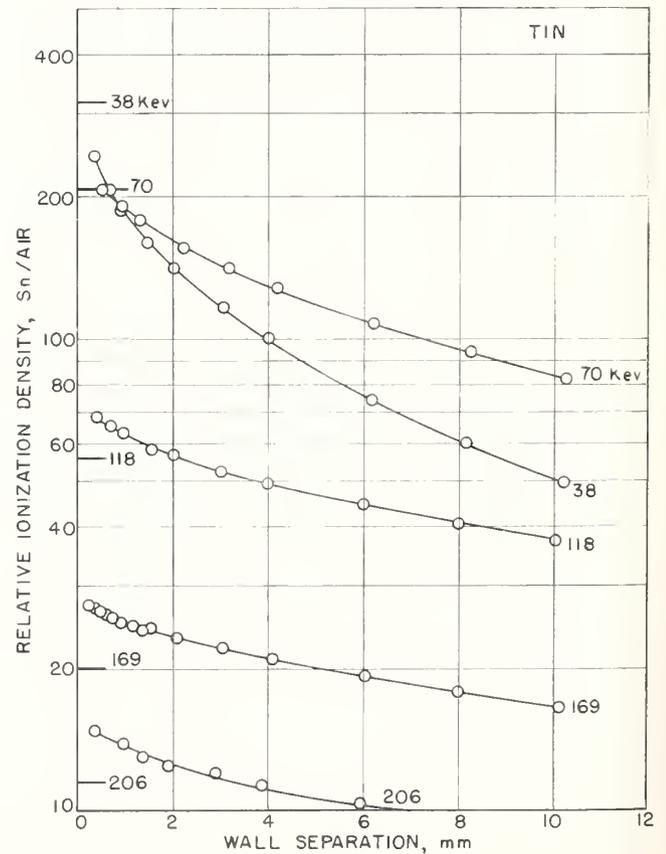


FIGURE 6.

(See caption for fig. 3)

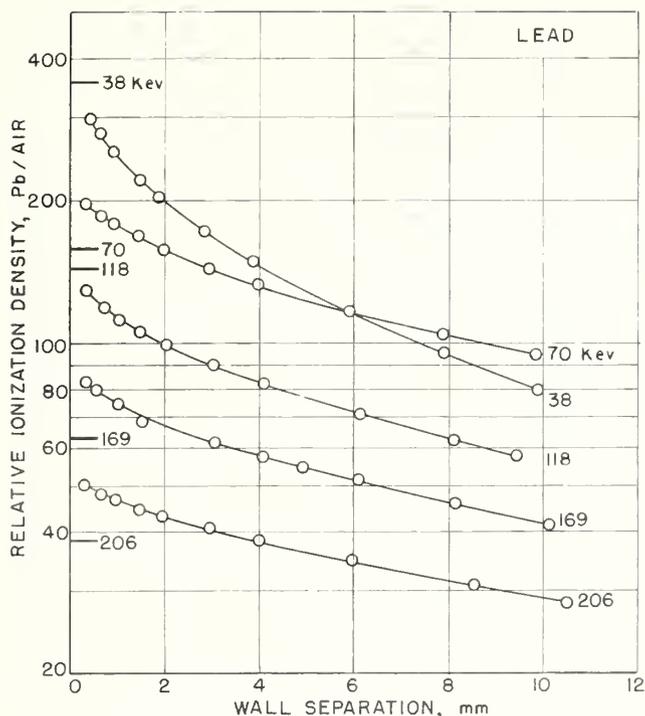


FIGURE 7.

(See caption for fig. 3)

such electrons in air is about 2 mm. On the other hand, in lead the K -orbit electrons do not interact with 38-keV X-rays, and the L -shell photoelectrons predominate. These have energies in the vicinity of 22 keV, and ranges of about 1 cm. Thus this curve is less steep than the corresponding one for Sn. Similar arguments can be applied to explain the trends of the other curves at 38 and 70 keV, referring to table 5 for dominant electron energies. At higher X-ray energies the edge losses of electrons begin to predominate. Here the slopes are not strongly dependent on X-ray energy, because the electron ranges are large compared with the lateral chamber dimensions.

3.4 Comparison with Cavity Theory

The Bragg-Gray theory of cavity ionization [12, 13, 6] gives for the ratio of ionizations per gram of air in two cavity chambers, A and B :

$$\frac{J_A}{J_B} = \frac{(\mu_{en})_A s_B}{(\mu_{en})_B s_A}$$

where s is the ratio of electronic stopping powers (per electron/cm²) of the wall material to air, evaluated for the electron spectrum present. If cavity B is a free-air chamber, $s_B=1$ and the above formula is further simplified.

μ_{en} is defined as the sum of those fractions of the photoelectric-, Compton-, and pair-production attenuation coefficients for γ -rays, representing energy converted from electromagnetic energy into electron-kinetic energy. The units of μ_{en} are cm²/electron throughout this paper [14].

For valid application of this theory, the cavity must be small in comparison with the ranges of the

electrons present, and the ionization contributed by electrons generated in the gas directly by X-rays must be negligible. These requirements are difficult to fulfill with low-energy X-rays, because of the short ranges of the electrons present. Moreover, the theory itself is only an approximation, as it neglects the production (through collision) of energetic secondary electrons by the Compton-recoil electrons and photoelectrons in traversing the wall material and the air.⁷

Table 3 lists the values of the ratio $(\mu_{en})_Z/(\mu_{en})_{air}$, calculated from the X-ray attenuation coefficients of White [9], assuming the X-ray spectra to be monochromatic at the effective energy. (μ_{en}) is the sum of the photoelectric coefficient (less fluorescence losses) and the "true" Compton coefficient σ_a .⁸

TABLE 3. Ratios of energy-absorption coefficients μ_{en} (in cm²/electron) relative to air

Effective photon energy	$(\mu_{en})_Z/(\mu_{en})_{air}$				
	C	Al	Cu	Sn	Pb
KeV					
38	0.45	5.62	70.6	176	176
70	.77	2.79	34.6	136	90.2
118	.96	1.37	8.78	38.8	78.5
169	.98	1.11	3.41	14.2	38.5
206	.99	1.05	2.24	8.29	24.4
411	1.00	1.00	1.15	2.00	5.37
670	1.00	1.00	1.04	1.29	2.55
1,250	1.00	1.00	1.00	1.10	1.56

The stopping-power calculations were done by means of Bethe's formula,⁹ using the mean-excitation potentials of Bakker and Segre [16] as modified for the binding correction by Bethe and Ashkin [15]. The values used were $I_{air}=80.5$ eV, $I_C=76.4$ eV, $I_{Al}=150$ eV, $I_{Cu}=276$ eV, $I_{Sn}=463$ eV, and $I_{Pb}=705$ eV. The correction for density effect according to Sternheimer [17] was included.

The stopping-power ratio of air to wall material ($1/s$), evaluated over the electron spectrum present, is obtained [6] from the integration

$$\frac{1}{s} = \frac{1}{T_0} \int_0^{T_0} \frac{S_{air}}{S_{wall}} dT$$

where T_0 is the initial energy of the electrons generated in the wall material by X- or γ -rays, and s is the stopping power (per electron/cm²) of the air or wall for an electron of energy T . Values of $(1/s)$ are tabulated as a function of T_0 in table 4.

⁷ A recently proposed modification to the theory [6] takes the "knock-on" secondary electrons into account and relates ionization to cavity size. This has not been applied here to the X-ray results because the necessary electron spectra have not been calculated for low-electron starting energies. This modified theory is, however, compared with the results of the γ -ray measurements, for which the calculated electron spectra are available.

⁸ A small fraction of μ_{en} for Pb and Sn at 1,250 keV is attributable to pair production, see table 5.

⁹ See eq (52), p. 254 in reference [15].

tributing to this effect is that the experimental chamber receives more scattered radiation from the X-ray beam than does the free-air chamber. The diaphragms in the latter chamber admit only the primary rays and those scattered in a nearly forward direction. This probably also contributes to the discrepancies between theory and experiment for the other wall materials in figures 4 to 7 as well.

TABLE 6. Mean stopping-power ratios $(\overline{1/s})$,¹ air relative to wall material

Effective photon energy <i>kev</i>	$(\overline{1/s})$				
	C	Al	Cu	Sn	Pb
38.....	0.99	1.14	1.35	1.80	2.03
70.....	.99	1.14	1.29	1.54	1.75
118.....	.99	1.15	1.27	1.44	1.84
169.....	.99	1.14	1.27	1.40	1.64
206.....	.99	1.13	1.27	1.39	1.58
411.....	.99	1.11	1.24	1.36	1.48
670.....	.99	1.10	1.22	1.33	1.43
1,250.....	1.00	1.09	1.19	1.29	1.38

¹ These data were obtained by graphical interpolation of the data given in table 4, at the electron energies (T_0) given in columns 1 of table 5. The stopping-power ratios $(1/s)$ so obtained were then weighted by the appropriate factors given in columns 2 of table 5 to obtain $(\overline{1/s})$.

4. γ -Ray Measurements

4.1. γ -Ray Sources

Sources consisting of several curies of Au^{198} , Cs^{137} , and Co^{60} were enclosed in lead housings to provide collimated beams as for the X-ray work already described. The Au^{198} and Cs^{137} source-housings were constructed to allow the rays going in a rearward direction to escape through a hole, thus avoiding the production of 180° backscattered γ -rays of low energy. The forward beam was filtered by 2.4-mm Sn+0.5-mm Cu+0.8-mm Al to suppress any fluorescence emitted by the lead housing.

The Co^{60} source first used was a 10-curie source in a large lead housing, closed in the rearward direction and lined with brass on the inside. The first results were taken with the same filter used for the other sources, but it was found later that the scattered radiation originating in the housing and source was not adequately removed by this filter, and as a result the ionization in the chamber with high-atomic-number walls was spuriously high because of excess photoelectric effect. The final data described here were taken with a 1-curie Co^{60} source 3 mm in diameter, enclosed in a lead housing relatively free of backscattering, and with a filter of 12-mm Pb in addition to the previously described filter of Sn, Cu, and Al. Additional thicknesses of lead were found not to change the observed ionization ratio of Pb/C.

The spectra of gamma rays from Au^{198} and Cs^{137} are nearly monochromatic, and located at 411 kev and 670 kev, respectively. Co^{60} emits two lines of equal intensity at 1.17 Mev and 1.33 Mev, but for present purposes it has been assumed to be monochromatic at 1.25 Mev.

4.2. Results with γ -Rays

For the γ -ray measurements no free-air chamber was employed. Only the experimental ionization chamber was used and the relative ionization densities observed with the various wall materials. These results are given in figures 8 to 10.

As a test of the influence of the electron losses out the edge of the chamber, side walls were constructed of the various wall materials. These consisted of rings about 9 cm in diameter, thick enough for electronic equilibrium and having various depths so that

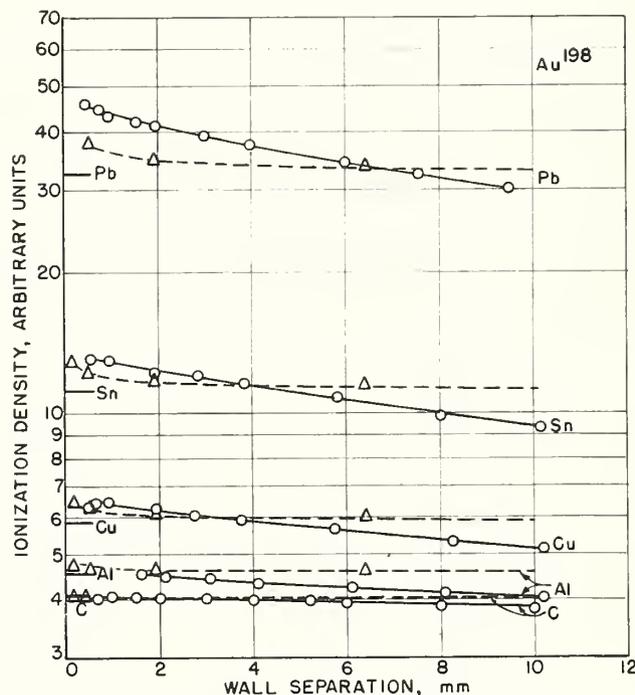


FIGURE 8. The solid curves show the relative ionization densities measured in the experimental chamber with γ -rays from Au^{198} or Cs^{137}

The marks at the vertical axis are the ionization ratios, relative to graphite, predicted by the Bragg-Gray theory. The dashed curves are the corresponding ratios predicted by the modified cavity theory. Both sets of theoretical data have been normalized to the experimental graphite curve at small separations.

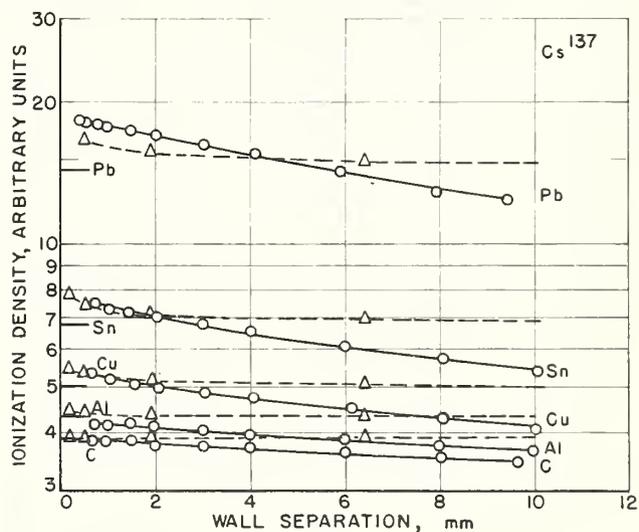
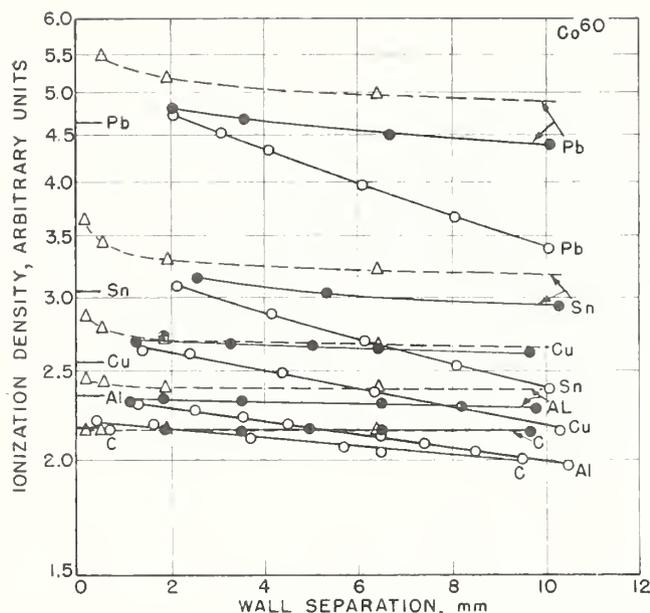


FIGURE 9.

(See caption for fig. 8)



FIGURES 10. The solid curves drawn through the open circles show the relative ionization densities measured in the experimental chamber with γ -rays from Co^{60} , without side walls.

The solid curves drawn through the filled circles show the corresponding results when side walls were added. The marks at the vertical axis, are the ionization ratios, relative to graphite, predicted by the Bragg-Gray theory. The dashed curves are the corresponding ratios predicted by the modified cavity theory. Both sets of theoretical data have been normalized to the experimental graphite curve (with side walls) at small separations.

the wall separation could be varied. The γ -ray beam was large enough to irradiate these rings, producing electrons to replace those lost from the collecting region. The rings were insulated both from the guard ring and the high-voltage wall of the chamber by polyethylene 0.025 mm in thickness. It was found that, for wall separations as great as 1 cm, there was a negligible difference in ionization collected whether the rings were operated at the potential of the high-voltage wall or at ground potential, indicating no field-distorting effect. Glancing angle attenuation (1) of the γ -rays striking the rings was checked by also constructing rings of 1-cm depth but with a cone shape to allow the rays to strike the inner-ring surface at a 45° angle rather than a glancing angle, resulting in much less attenuation.

Gamma-ray scattering from the rings was also measured by doubling their thickness and observing the resulting increase in ionization. Both of these effects were found to be negligible.

In figure 10 the open points indicate the ionization density observed without the edge rings, and the solid points show the results with the rings added. For the lead walls at 10-mm separation, the edge losses are seen to be about 23 percent, while for graphite they are about 9 percent. This difference is caused by the greater reflecting ability of the lead for electrons, increasing the effective solid angle for their escape out the edges. While the edge losses shown in figure 10 apply strictly to the Co^{60} data only, they do give some indication of the losses at other energies where the electron ranges are long compared with the chamber dimensions. They also supply an upper limit for the magnitude of the losses at lower energies.

4.3. Comparison with Bragg-Gray Cavity Theory

Bragg-Gray theory calculations have already been described in the comparison with X-ray ionization data. Tables 3 and 6 also contain data applicable to the γ -ray results in the same fashion. However, because there are, for γ -rays, no free-air chamber results available for comparison with cavity-chamber results, the theoretical ionization ratios J_z/J_{air} have been divided by J_c/J_{air} to give J_z/J_c . These ratios are then normalized to the experimental graphite-chamber ionization density at small separations, and plotted as short lines adjacent to the y -axis in figures 7 to 10. Although they generally tend to be too low, these theoretical ratios do roughly predict the experimental-ionization ratios relative to graphite, particularly for Co^{60} γ -rays.

4.4. Comparison with Modified Cavity Theory

A modified cavity theory (see footnote 7) which takes into account the production of secondary electrons and which relates the ionization to the cavity size, has also been compared with the experimental γ -ray results. The difference from the conventional Bragg-Gray treatment comes in the calculation of the stopping-power ratio ($1/s$), the details of which are given in reference [6]. In the modified theory ($1/s$) is a function not only of T_0 , but also of a parameter Δ , which is taken to be the energy needed by an electron to cross the cavity.

In table 7 are listed the values of $(1/s)$ obtained from this theory, based upon the same mean-excitation potentials (I) and density-effect data as were used before. Table 5 has again been used to weight ($1/s$) by the electron-energy flux at each (mean) energy, T_0 present in a material. The approximate electron linear ranges corresponding to the values are also given, assuming the linear range to be about 0.8 times the actual electron-track length [18].

To compare these data with the experimental results, one must assign an effective size to the experimental-chamber cavity. This has been taken to be simply the plate separation, because the accurate choice of Δ is not critical.

The product of the terms in table 7 with the corresponding terms in table 3 yields the theoretical ionization ratios relative to air, according to the modified theory. Having renormalized the data to be relative to graphite, as was done before for the Bragg-Gray results, they are plotted as dashed curves in figures 8 to 10.

It is interesting to note how closely the theory predicts the variation of ionization with chamber size for Co^{60} , where the edge losses of electrons are eliminated. The agreement between this theory and the experimental results otherwise is not very exact, although it generally seems to be an improvement upon the other theory, particularly at small separations. Recent ionization measurements by Greening [2], by Whyte [3], and by Attix and Ritz [1], also confirm that the modified cavity theory gives improved agreement and predicts closely the

TABLE 7. Mean stopping-power ratios $(1/s)$ air relative to wall material, from modified cavity theory

Wall material	γ -ray energy	Electron range (mm air)				
		0.15	0.51	1.9	6.4	22
		Δ (kev)				
		2.5	5.1	10.2	20.4	40.9
C	Mev					
	0.411	0.99	0.99	0.99	0.99	0.99
	.670	.99	.99	.99	.99	.99
Al	0.411	1.14	1.13	1.12	1.11	1.10
	.670	1.14	1.12	1.11	1.10 ₅	1.10
	1.25	1.13	1.12	1.11	1.10	1.09
Cu	0.411	1.35	1.31	1.27 ₅	1.25	1.23
	.670	1.33 ₅	1.29	1.26	1.24	1.22
	1.25	1.32	1.28	1.25	1.23	1.21
Sn	0.411	1.56 ₅	1.48	1.42	1.38	1.35
	.670	1.55	1.46	1.41	1.37	1.34
	1.25	1.52	1.44	1.38 ₅	1.35	1.32
Pb	0.411	-----	1.68	1.58	1.52	1.47
	.670	-----	1.65	1.56	1.50	1.45
	1.25	-----	1.62	1.53	1.47	1.43

variation of ionization with cavity size. In particular, reference [1] shows very close agreement between theory and experiment with Co^{60} γ -rays and chamber walls of C, Al, and Cu. Other recent work in this laboratory [19] has also confirmed the agreement between a graphite cavity chamber and a pressurized free-air chamber in measuring exposure dose (in roentgens) of γ -rays from Co^{60} and Cs^{137} , after cavity-theory corrections are applied to the graphite chamber results.

The authors express their appreciation to H. O. Wyckoff and L. V. Spencer for many helpful discussions of the work, and to J. Hopfield and P. R. Fry for assisting in the early phases of the experiment. F. H. Attix would especially like to thank F. G. Brickwedde for serving as his advisor for that part of the work constituting a master's degree thesis at the University of Maryland.

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Preparation, Maintenance, and Application of Standards of Radioactivity

W. B. Mann and H. H. Seliger



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Preface

The present Circular, which is a revision and unification of two review articles that appeared in the *International Journal of Applied Radiation and Isotopes*, attempts to survey the current position in the field of radioactivity standardization.

The most important aspects of this revision have been the inclusion of recent work to bring the text up to date, the use of a modified Harvard system of references and the incorporation of figures that illustrate specifically the methods used at the National Bureau of Standards in the field of radioactivity.

The National Bureau of Standards wishes to thank the Editors of the *International Journal of Applied Radiation and Isotopes* and the Pergamon Press Limited for their kind permission to use material in the two review articles, referred to above, in this Circular.

A. V. ASTIN, *Director*.

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Preparation, Maintenance, and Application of Standards of Radioactivity

W. B. Mann and H. H. Seliger

The methods available for the preparation and maintenance of primary and secondary standards of radioactivity are reviewed, and the applications of such standards to problems in physics are discussed.

1. Introduction

The oldest standards of radioactivity are those of radium. Just 62 years ago, in February 1896, the radioactivity of uranium was discovered by Henri Becquerel, and before the turn of the century Marie Curie, investigating the high activity arising from Bohemian pitchblende, had isolated polonium and radium.

With the great outburst of research activity that these startling new discoveries generated, it soon became essential to establish international radium standards in order that the results obtained in different countries might be related one to another. Accordingly, in 1910, the Congress of Radiology and Electricity, held in Brussels, appointed a committee to arrange for the preparation of an international primary standard of radium and of international secondary standards.

In August 1911, Mme. Curie prepared, in Paris, a standard of radium sealed in a thin-walled glass tube and comprised of 21.99 mg of the pure anhydrous radium chloride that she had used to determine the atomic weight of radium to be 226.0. At the same time, Professor Otto Hönigschmid, at the Academy of Sciences in Vienna, prepared three standards of radium containing 10.11, 31.17, and 40.43 mg of radium chloride from material that he had used to obtain a value of 225.97 for the atomic weight of radium. In March 1912 the committee that had been appointed by the Congress of Radiology and Electricity in 1910, met in Paris to compare the radium standard of Mme. Curie with those of Professor Hönigschmid.

As a result of these comparisons, the Paris standard was accepted as the international primary standard of radium, while that consisting of 31.17 mg of radium chloride was designated as the international secondary standard. These two standards, which are now generally referred to as the 1911 Paris and Vienna standards, were placed respectively in the custody of the Bureau International des Poids et Mesures at Sèvres and of the Institut für Radiumforschung in Vienna. All subsequently prepared national radium standards were, for the next two decades, compared by measuring the relative gamma-ray activities with the 1911 Paris and Vienna standards.

By 1934, however, some concern was beginning to be felt for the safety of the old international primary and secondary standards, on account of the possibility of their breakage due to the devitrification under radiation of the glass tubes in which

they were sealed and the accumulation of helium and chlorine within them. Accordingly, the International Radium Standards Commission invited Hönigschmid to prepare a new set of standards, using the radium salt with which he was making, at that time, in Munich, a precise determination of the atomic weight of radium. This salt, the chloride of radium, had been purified by Hönigschmid to such a point that spectroscopic analysis by Gerlach showed a maximum of not more than 0.002 to 0.003 atom percent, of barium. The purity of the radium chloride is, so to speak, defined by the value then obtained by Hönigschmid for the atomic weight of radium, namely 226.05, which is the value that is still current.

On the afternoon of 2 June 1934, Hönigschmid prepared twenty new standards of radium by sealing into thin-walled glass tubes quantities varying from 12.53 mg to 137.34 mg of this highly purified anhydrous chloride of radium from which the radium D had been separated on 25 May 1934 (Meyer, 1945; Hönigschmid, 1945).^{*} One of these, containing as of that date, 22.23 mg of radium chloride, was selected as the new international primary standard of radium for custody in Paris, and its value was carefully compared with that of the 1911 Paris standard by gamma-ray measurements over a period of 4 years (Chamié, 1940). The remaining Hönigschmid standards were designated for use as international secondary or national standards of radium.

With the advent of controlled nuclear fission and the consequent large-scale production and use of artificially prepared radioactive materials, the need for radioactivity standards of elements other than radium quickly arose. In the first place, approximate standards were often prepared in the establishments in different countries that were producing such radioactive materials in order to provide them with a "yardstick" for the supply of radioactive materials in bulk. However, with the increasingly important applications of radioactive materials to research and therapy, accurate standards of radioactivity became as essential as, some 40 years earlier, they had become with radium. Once more the standards were required not only for comparison of results between laboratories in one country, but also for international comparisons.

Accordingly, in 1951, representatives of the

^{*}See references on page 42.

three countries that were at that time chiefly interested in the creation of international standards of radioactivity, Canada, the United Kingdom, and the United States of America, met at the Atomic Energy Research Establishment at Harwell, England, to discuss the preparation and maintenance of radioactivity standards of the artificially prepared radioactive isotopes of elements. As a result, it was agreed to exchange for intercomparison purposes suitable samples of the following radioactive nuclides, namely, carbon-14, sodium-24, phosphorus-32, cobalt-60, bromine-82, strontium-90-yttrium-90, iodine-131, thallium-204, and gold-198. Responsibility for the establishment of such standards in the three countries was vested in the National Bureau of Standards at Washington, D. C., the National Physical Laboratory at Teddington, and the National Research Council at Chalk River. The National Research Council's responsibilities were subsequently assumed, however, by Atomic Energy of Canada Limited with regard to the artificially prepared radioactive nuclides, while the Canadian National Radium Standard continued in the custody of the National Research Council at Ottawa.

Within the United Kingdom and the United States three advisory committees or subcommittees have now been set up to provide a forum for discussion amongst those interested in the field of radioactivity standards. These are the British National Physical Laboratory's Advisory Committee on Radioactive Standards; the U. S. National Research Council's Nuclear Science Committee's Subcommittee on Beta- and Gamma-ray Measurements and Standards; and the U. S. National Committee on Radiation Protection and Measurement's Subcommittee on Standards and Measurements of Radioactivity for Radiological Use. These latter include Canadian representation, and the National Research Council's subcommittee meetings in Washington, D. C., in 1954, at Chalk River, Ontario, in 1955, at Richmond and Berkeley, California, in 1956, and at Easton, Maryland, in 1957, were also attended by British representatives. The name of the National Research Council's subcommittee was changed, subsequent to its 1956 California meeting, to the Subcommittee on Measurements and Standards of Radioactivity in order that it might not be precluded in its deliberations from considering other than beta and gamma emitters.

It had, however, always been the aim that the cooperation that had been created between the United States, Canadian, and British laboratories should be extended as quickly as possible to all interested countries. Accordingly, in 1953, at its meeting in Copenhagen, the International Commission on Radiological Units created a Subcommittee on Standards of Radioactivity, to be composed in the first instance of representatives of the Federal Republic of Germany, France, The Netherlands, Sweden, the United Kingdom, and the

United States. This subcommittee met in Frankfurt-am-Main in August 1955 to discuss the preparation, preservation, and propagation of international standards of radioactivity, and the hope was expressed at that meeting that these activities, that were so intimately related to the progress, prosperity, and health of all mankind, might soon be extended to all those countries, that were interested, throughout the world.

In April 1956 the International Commission on Radiological Units met again in Geneva and received the report of its subcommittee on radioactivity standards which met concurrently with the main commission. At this time the name of the subcommittee was changed to Committee I on Standards and Measurement of Radioactivity for Radiological Use and its membership rotated and extended to include a representative of Norway, in place of the representative of Sweden, and a representative of Uruguay. The report of the subcommittee at Geneva has now been published in the National Bureau of Standards Handbook 62, "Report of the International Commission on Radiological Units and Measurements (ICRU) 1956."

For convenience, the consideration in this Circular of methods of measurement will be broken down into sections dealing with primary and secondary methods of standardization. The dividing line between a primary method and a secondary method is, however, often extremely thin and difficult to place. It might be argued, for example, that the calorimetric method for determining disintegration rates is "secondary" because it requires a knowledge of the average energy per disintegration; it will be found, however, that in the case of those radioactive elements for which the details of the disintegration scheme are known, calorimetry has been placed in the "primary" group, whereas in the case of radium in equilibrium with its products, where the disintegration data are complicated, if not nebulous, it has been considered to be a "secondary" method. On reflection, it might be said that in general a primary method is one by which the initial standardization is made or one in which a fundamental quantity such as disintegrations per second, or rate of energy emission per gram is measured. A secondary method, on the other hand, is one in which a calibration is related to an earlier primary calibration or in which a fundamental quantity is measured in terms of a similar property that has already been measured for another radioactive nuclide, such as radium in equilibrium with its products. But lest the adherents to any particular method should feel that less than justice might have been done to it, let it be immediately placed on record that no kind of stigma whatsoever, beyond that of mere convenience, attaches to either classification.

No attempt has been made within the confines of this Circular and its bibliography to deal in detail with the wide field of radioactivity instrumentation. Many excellent and well-known text-

books are available that deal with the subject of counter- and ionization-chamber techniques. Only in cases where an instrument or method of meas-

urement may have had a somewhat specific application to radioactivity standardization, have references been given in this review.

2. Methods of Measurement

2.1. Secondary Standardization of the Naturally Occurring Radioactive Elements

The international primary standards of radium are standards by mass (figs. 1, 2, and 3). Other radium preparations are normally compared with these by comparison of the gamma-ray effects after the preparation has been sealed about thirty days to enable the radium to come into equilibrium with its early daughter products; the principal gamma-ray contribution comes from the excited levels of radium C' in the disintegration of radium C. Alternatively, radium preparations and standards may be intercompared calorimetrically or by the method of radon analysis.

The radioactivity of a given mass of radium is measured in terms of the curie. This unit was initially defined as the quantity of radon in equilibrium with 1 gram of radium. This quantity of radon was also found to be emitting alpha particles at a rate of about 3.7×10^{10} disintegrations per second, although in later years, with the improvements in measuring techniques, the measured value of this emission rate has departed significantly from 3.7×10^{10} disintegrations per second, with a trend toward lower values.¹ A recent and very careful determination of the specific activity of radium, which corresponds to the curie as first defined for radon in equilibrium with radium, has given a value of $(3.608 \pm 0.028) \times 10^{10}$ alpha particles per second per gram (Kohman, Ames, and Sedlet, 1949). Consequently, in 1950, at the VIth International Congress of Radiology in London, the definition of the curie was changed to apply to the measurement of the disintegration rate of radioactive substances other than radium and radon, being defined as the quantity of any radioactive nuclide in which the number of disintegrations per second is 3.700×10^{10} .

One of the earliest and simplest methods for measuring the relative gamma-ray effects of radium preparations was that using the gold-leaf electroscope. This instrument has indeed been retained even till the present time in national standardizing laboratories (Curtiss, 1928; Perry, 1936) and gives good reproducibility with a standard deviation of the order of 0.5 percent, or even less with care, and is ideal for long-term trouble-free routine service (fig. 4). Ionization chambers, either singly or as a balanced pair (Rutherford and Chadwick, 1912) have also been used for the precise comparison of the gamma-ray effects from radium in equilibrium with its daughter products. The use of a balanced pair of ionization chambers

has recently been carried to a high degree of precision, using the decay of a sealed source of radon in the one chamber to achieve an exact balance (Kipfer, 1953; Curie, 1954). The present position is that the relative gamma-ray effects of different preparations can be measured with a precision that is better than the uncertainties arising from the unknown self-absorption in the sources themselves, which is difficult to calculate.

The calorimetric method, in which the energy emission due to the alpha, beta, and gamma radiations is measured, is less dependent on differences in the gamma-ray absorption of the sources themselves. Some 93 percent of the energy emission of a radium sample in equilibrium with its products is corpuscular and, provided that most of the particles are absorbed, the measured relative values of different sources will be quite insensitive to any small changes of the order of 1 or 2 percent in the absorption of the gamma radiation.

The earliest experiments to measure the rate of energy emission of radium and its products by their heating effects were carried out within a few years of the discovery of radium, and published in 1903 (Curie and Laborde). Subsequently, attempts were made to measure the heating effects of radium and those of its products that were alpha emitters (Rutherford and Barnes, 1903; Schweidler and Hess, 1908; Meyer and Hess, 1912; Hess, 1912; Rutherford and Robinson, 1912; Rutherford and Robinson, 1913; Watson and Henderson, 1928; Zlotowski, 1935). Recently, the calorimetric method has been developed to compare radium preparations with a standard deviation of about 0.1 percent (Mann, 1954a and 1954b) (figs. 5 and 6).

It should perhaps be emphasized that where the electroscope can measure only relative quantities by the gamma-ray effect, the calorimeter, in the comparison of one radium preparation with another or with a standard, does measure absolutely a property of radium and its products, namely, the rate of energy emission per milligram of radium element.

The radon method of comparing radium preparations requires that the radon in equilibrium with the radium shall be removed and measured separately, nowadays usually by counting the radon alpha particles or those arising from its decay products (Rutherford and Boltwood, 1905 and 1906; Strutt, 1905; Paneth and Koeck, 1931; Evans, 1935; Urry, 1936; Urry and Piggot, 1941; Curtiss and Davis, 1943; Bryant and Michaelis, 1952; Cowper and Simpson, 1953). It is thus unsuited to the comparison of permanently sealed

¹ A complete bibliography of these is given by Kohman, Ames, and Sedlet (1949).

COMMISSION INTERNATIONALE DES ÉTALONS DE RADIUM.

CERTIFICAT.

Das als Chlorid dargestellte Radiumpräparat Nr. 6 entstammt St. Joachimstaler Uranpechblende und ist demnach praktisch frei von Mesothor.

Es enthält 21.50 Milligramm Salz.

Es wurde am 1. Juli 1913 eingeschlossen in ein Glasröhrchen (Thüringer Glas) von 0.27 mm Wandstärke, äußerem Durchmesser 3.2 mm, Länge 22 mm, an dessen Ende ein feiner Platindraht eingeschmolzen ist.

Dasselbe wurde als Secundärer Standard an den Wiener Etalons und an dem internationalen Standard in Paris nach mehreren γ -Strahlungsmethoden unabhängig voneinander geeicht.

Der γ -Strahlung nach ist es im Jahre 1913 äquivalent 20.28 mg RaCl_2 . (Die jährliche Abnahme beträgt etwa 0.4 Promille.)

Unter Zugrundelegung der Atomgewichte von

226	für Radium
35.457	für Chlor
79.916	für Brom

entspricht dies

15.44	mg Ra -Element,
20.28	mg RaCl_2 ,
26.36	mg RaBr_2 .

Die Genauigkeit dieser Angabe wird auf 0.2 % für gesichert gehalten.

La Préparation de Chlorure de Radium contenue dans l'ampoule Nr. 6 provient de la pechblende de St. Joachimsthal. Elle est donc pratiquement exempte de Mesothorium.

Elle contient 21.50 Milligrammes de sel.

Le sel a été enfermé le 1/7/1913 dans un tube de verre (Verre de Thuringe.) Epaisseur du verre 0.27 mm; Diamètre extérieur 3.2 mm; Longueur 22 mm. Un fil de platine fin a été soudé à l'extrémité du tube.

En qualité d'Étalon secondaire l'ampoule a été comparée à l'Étalon de Vienne et à l'Étalon International de Paris, au moyen de méthodes de mesures basées sur le rayonnement γ . La comparaison a été faite indépendamment à Vienne et à Paris.

D'après son rayonnement γ , la Préparation équivaut en l'année 1913 à 20.28 mg. RaCl_2 . (La diminution par année est de 0.4 pour mille.)

En adoptant les poids atomiques suivants:

Radium . . .	226
Chlore . . .	35.457
Brome . . .	79.916

on déduit la teneur correspondante en Radium-élément et en Bromure de Radium:

Ra	15.44 mg.
RaCl_2	20.28 mg.
RaBr_2	26.36 mg.

La précision de ces résultats est considérée comme assurée à une approximation de 0.2 %.

Specimen No. 6 of Radium is prepared as chloride from pitchblende of St. Joachimsthal and is consequently practically free from Mesothorium.

It contains 21.50 Milligrammes of salt.

It was enclosed the 1/7/1913 in a glass tube (Thuringian glass) of 0.27 mm thickness, exterior diameter 3.2 mm, length 22 mm, a thin platinum wire being fused into the end of the tube.

It is calibrated as Secondary Standard by comparison with the Vienna-Standard and with the International Standard at Paris, several independent γ -ray methods being used.

Measured by the γ -radiation, it is in the year 1913 equivalent to 20.28 mg. RaCl_2 . (The yearly decay is about 0.4 per mille.)

Taking the atomic weights

226	for Radium
35.457	for Chlorine
79.916	for Bromine

this corresponds to

15.44	mg Ra -element,
20.28	mg RaCl_2 ,
26.36	mg RaBr_2 .

These statements are considered correct to 0.2 %.

Für die Wiener Messung

Jefan Meyer

Pour les mesures faites à Paris

M. Curie

President of the Commission

E. Rutherford

FIGURE 1. Certificate of the International Radium Standards Commission for the first United States Primary Radium Standard, Vienna No. 6 and International Commission No. IV, which was brought to America in 1913 by Mme. Marie Curie.

It is interesting to note the somewhat large difference in the mass of the radium chloride as determined by weighing and gamma-ray measurement.

COMMISSION INTERNATIONALE DES ETALONS DE RADIUM.

CERTIFICAT.

Das als Chlorid dargestellte Radiumpräparat Nr. XIV entstammt Uranerzen von Katanga, belgisch Kongo, und ist demnach praktisch frei von Mesothorium.

Es wurde am 2. Juni 1934 von Prof. Hönigschmid in München ausgewogen und in ein Glasröhrchen von 3 mm lichter Weite und 0.27 mm Wandstärke und einer Länge von 36 mm eingebracht, an dessen Ende ein feiner Platindraht eingeschmolzen ist.

Es ist gekennzeichnet durch die Nr. 5437 des Schutzrohres, sowie durch sein Bruttogewicht von 323.93 mg und enthielt damals 50.22 mg reines, wasserfreies Salz, gewichtsmäßig entsprechend 38.23 mg Radiumelement.

Der Reinheitsgrad des verwendeten Radiumchlorids ist sichergestellt durch die von Prof. Hönigschmid ausgeführte Atomgewichtsbestimmung, die für Ra zu dem Werte 226.05 führte, sowie durch die von Prof. W. Gerlach durchgeführte spektroskopische Untersuchung, welche einen Bariumgehalt von maximal 0.002 — 0.003 Atomprozent Barium ergab.

Das Radiumpräparat wurde zum letztenmal am 25. Mai 1934 durch eine Fällung mit Schwefelwasserstoff von RaD befreit.

Das Präparat wurde mit den primären Etalons von 1911 in Wien und Paris nach mehreren γ -Strahlungsmethoden unabhängig von einander geeicht.

Der γ -Strahlung nach ist es Ende 1936 äquivalent 38.10 mg Ra-Element. Für eine mittlere Lebensdauer des Radiums von 2295 Jahren beträgt der jährliche Abgang 0.436 Promille.

Unter Zugrundelegung der Atomgewichte

226.05 für Radium
35.457 für Chlor
79.916 für Brom

entspricht dies bezogen auf die primären Etalons von 1911 für die Jahresperiode 1936/1937

38.10 mg Ra-Element
50.05 mg RaCl₂
65.04 mg RaBr₂

Die Genauigkeit dieser Angaben wird auf 0.3% für gesichert gehalten.

Für die Wiener Messungen:

Stefan Meyer

La préparation de Radium No. XIV est un chlorure qui provient de la pechblende du Katanga, Congo Belge, et est, par conséquent, pratiquement exempt de Mesothorium.

Cette préparation a été pesée le 2 Juin 1934 par le Pr. Hönigschmid à Munich et a été introduite dans un tube en verre de 3 mm de diamètre intérieur, de 0.27 mm d'épaisseur des parois et de 36 mm de longueur, sur l'extrémité duquel un fil fin de platine a été scellé.

Elle est identifiée par le No. 5437 du tube protecteur, de même que par son poids brut de 323.93 mg et elle contenait alors 50.22 mg de sel pur anhydre correspondant au poids de 38.23 mg de Radium élément.

Le degré de pureté du chlorure de Radium est mis en évidence par les mesures du poids atomique faites par le Pr. Hönigschmid, qui conduisent pour le Radium au nombre 226.05, ainsi que par les essais spectroscopiques faits par le Pr. W. Gerlach, qui donnent une teneur en Baryum au maximum de 0.002 — 0.003 pour cent d'atomes de Baryum.

La préparation de Radium a été purifiée pour la dernière fois du RaD le 25 Mai 1934 par une précipitation par l'hydrogène sulfuré.

La préparation a été comparée en rayons γ aux étalons primaires de 1911 par diverses méthodes, indépendamment à Vienne et à Paris.

Par son rayonnement γ elle est équivalente fin 1936 à 38.10 mg de Radium élément.

Pour une vie moyenne du Radium de 2295 ans, la décroissance annuelle est de 0.436 pour mille.

Prenant pour base le poids atomique

de 226.05 pour le radium
de 35.457 pour le chlore
de 79.916 pour le brome

il vient pour fin 1936, début 1937 par rapport aux étalons primaires de 1911:

38.10 mg Ra-élément,
50.05 mg RaCl₂,
65.04 mg RaBr₂.

La précision de ces données est exacte à 0.3%.

Pour les mesures faites à Paris:

J. Gauthier-Larin

The Radium-preparation No. XIV has been prepared as chloride from uranium ores from Katanga, Belgian Congo, and is therefore practically free from Mesothorium.

It was weighed on June 2nd, 1934 by Prof. Hönigschmid in Munich and transferred to a glass tube of 3 mm inner diameter, 0.27 mm thickness of wall and 36 mm length, with a thin platinum wire sealed in at one end.

It is characterized by the No. 5437 on the protection tube, as well as the gross weight of 323.93 mg and contained at the date given above 50.22 mg of pure salt free from water corresponding in weight to 38.23 mg radium-element.

The degree of purity of the radium-chloride used is warranted by the determination of the atomic weight carried out by Prof. Hönigschmid, which gave for radium the value 226.05, and the spectroscopic investigation by Prof. W. Gerlach, which showed that the barium-content was 0.002 — 0.003 per cent atoms at most.

Radium D was separated from the radium-preparation for the last time on May 25th, 1934, by precipitation with hydrogen sulphide.

The preparation has been compared with the primary standards of 1911 in Vienna and Paris independently by several γ -ray-methods.

According to its γ -rays it was End 1936 equivalent to 38.10 mg towards the Radium-element. For an average life of radium of 2295 years the loss per year is 0.436 per mille.

Taking the atomic weights:

226.05 for radium
35.457 for chlorine
79.916 for bromine

as a basis this corresponds for the end of 1936 and the beginning of 1937 compared with the primary standards of 1911 to:

38.10 mg Ra-element,
50.05 mg RaCl₂,
65.04 mg RaBr₂.

These statements are considered correct to 0.3%.

The President:

Rutherford

FIGURE 2. Certificate of the International Radium Standards Commission for United States Primary Radium Standard No. XIV.

This standard which is Hönigschmid standard No. 5437, is actually about 36.7 mm in length. According to Stefan Meyer (1945), the Paris and Vienna gamma-ray measurements of the amount of radium chloride in this standard were respectively 50.03 and 50.08 mg.

COMMISSION INTERNATIONALE DES ETALONS DE RADIUM.

CERTIFICAT.

Das als Chlorid dargestellte Radiumpräparat Nr. XV entstammt Uranerzen von Katanga, belgisch Kongo, und ist demnach praktisch frei von Mesothorium.

Es wurde am 2. Juni 1934 von Prof. Hönigschmid in München ausgewogen und in ein Glasröhrchen von 3 mm lichter Weite und 0.27 mm Wandstärke und einer Länge von 37 mm eingebracht, an dessen Ende ein feiner Platindraht eingeschmolzen ist.

Es ist gekennzeichnet durch die Nr. 5440 des Schutzrohres, sowie durch sein Bruttogewicht von 267.86 mg und enthielt damals 26.86 mg reines, wasserfreies Salz, gewichtsmäßig entsprechend 20.45 mg Radiumelement.

Der Reinheitsgrad des verwendeten Radiumchlorids ist sichergestellt durch die von Prof. Hönigschmid ausgeführte Atomgewichtsbestimmung, die für Ra zu dem Werte 226.05 führte, sowie durch die von Prof. W. Gerlach durchgeführte spektroskopische Untersuchung, welche einen Bariumgehalt von maximal 0.002 — 0.003 Atomprozent Barium ergab.

Das Radiumpräparat wurde zum letztenmal am 25. Mai 1934 durch eine Fällung mit Schwefelwasserstoff von RaD befreit.

Das Präparat wurde mit den primären Etalons von 1911 in Wien und Paris nach mehreren γ -Strahlungsmethoden unabhängig von einander geeicht.

Der γ -Strahlung nach ist es Ende 1936 äquivalent 20.36 mg Ra-Element. Für eine mittlere Lebensdauer des Radiums von 2295 Jahren beträgt der jährliche Abgang 0.436 Promille.

Unter Zugrundelegung der Atomgewichte

226.05 für Radium
35.457 für Chlor
79.916 für Brom

entspricht dies bezogen auf die primären Etalons von 1911 für die Jahreswerte 1936/1937

20.36 mg Ra-Element,
26.74 mg RaCl₂,
34.75 mg RaBr₂.

Die Genauigkeit dieser Angaben wird auf 0.3% für gesichert gehalten.

Für die Wiener Messungen:

Stefan Meyer

La préparation de Radium No. XV est un chlorure qui provient de la pechblende du Katanga, Congo Belge, et est, par conséquent, pratiquement exempt de Mésothorium.

Cette préparation a été pesée le 2 Juin 1934 par le Pr. Hönigschmid à Munich et a été introduite dans un tube en verre de 3 mm de diamètre intérieur, de 0.27 mm d'épaisseur des parois et de 37 mm de longueur, sur l'extrémité duquel un fil fin de platine a été scellé.

Elle est identifiée par le No. 5440 du tube protecteur, de même que par son poids brut de 267.86 mg et elle contenait alors 26.86 mg de sel pur anhydre correspondant au poids de 20.45 mg de Radium élément.

Le degré de pureté du chlorure de Radium est mis en évidence par les mesures du poids atomique faites par le Pr. Hönigschmid, qui conduisent pour le Radium au nombre 226.05, ainsi que par les essais spectroscopiques faits par le Pr. W. Gerlach, qui donnent une teneur en Baryum au maximum de 0.002 — 0.003 pour cent d'atomes de Baryum.

La préparation de Radium a été purifiée pour la dernière fois du RaD le 25 Mai 1934 par une précipitation par l'hydrogène sulfuré.

La préparation a été comparée en rayons γ aux étalons primaires de 1911 par diverses méthodes, indépendamment à Vienne et à Paris.

Par son rayonnement elle est équivalente fin 1936 à 20.36 mg de Radium élément.

Pour une vie moyenne du Radium de 2295 ans, la décroissance annuelle est de 0.436 pour mille.

Prenant pour base le poids atomique

de 226.05 pour le radium
de 35.457 pour le chlore
de 79.916 pour le brome

il vient pour fin 1936, vers 1937

par rapport aux étalons primaires de 1911:

20.36 mg Ra-élément,
26.74 mg RaCl₂,
34.75 mg RaBr₂.

La précision de ces données est exacte à 0.3%.

Pour les mesures faites à Paris:

J. Joliot Curie

The Radium-preparation Nr. XV has been prepared as chloride from uranium ores from Katanga, Belgian Kongo, and is therefore practically free from Mesothorium.

It was weighed on June 2nd, 1934 by Prof. Hönigschmid in Munich and transferred to a glass tube of 3 mm inner diameter, 0.27 mm thickness of wall and 37 mm length, with a thin platinum wire sealed in at one end.

It is characterised by the Nr. 5440 on the protection tube, as well as the gross weight of 267.86 mg and contained at the date given above 26.86 mg of pure salt free from water corresponding in weight to 20.45 mg radium-element.

The degree of purity of the radium-chloride used is warranted by the determination of the atomic weight carried out by Prof. Hönigschmid, which gave for radium the value 226.05, and the spectroscopic investigation by Prof. W. Gerlach, which showed that the barium-content was 0.002 — 0.003 per cent atoms at most.

Radium D was separated from the radium-preparation for the last time on May 25th, 1934, by precipitation with hydrogen sulphide.

The preparation has been compared with the primary standards of 1911 in Vienna and Paris independently by several γ -ray-methods.

According to its γ -rays it was towards the end 1936 equivalent to 20.36 mg Radium-element. For an average life of radium of 2295 years the loss per year is 0.436 per mille.

Taking the atomic weights:

226.05 for radium
35.457 for chlorine
79.916 for bromine

as a basis this corresponds for the end of 1936 and the beginning of 1937 compared with the primary standards of 1911 to:

20.36 mg Ra-element,
26.74 mg RaCl₂,
34.75 mg RaBr₂.

These statements are considered correct to 0.3%.

The President:

Rutherford

FIGURE 3. Certificate of the International Radium Standards Commission for United States Primary Radium Standard No. XV.

This standard which is Hönigschmid standard No. 5440 is about 36.3 mm in length. The Paris and Vienna gamma-ray measurements of the amount of radium chloride in this standard, if one neglects an obvious misprint in the original paper (Meyer, 1945), were respectively 26.73 and 26.76 mg. Figures 1, 2, and 3 provide an interesting historical glimpse of the personal permanence of the Commission over a period of nearly a quarter of a century.

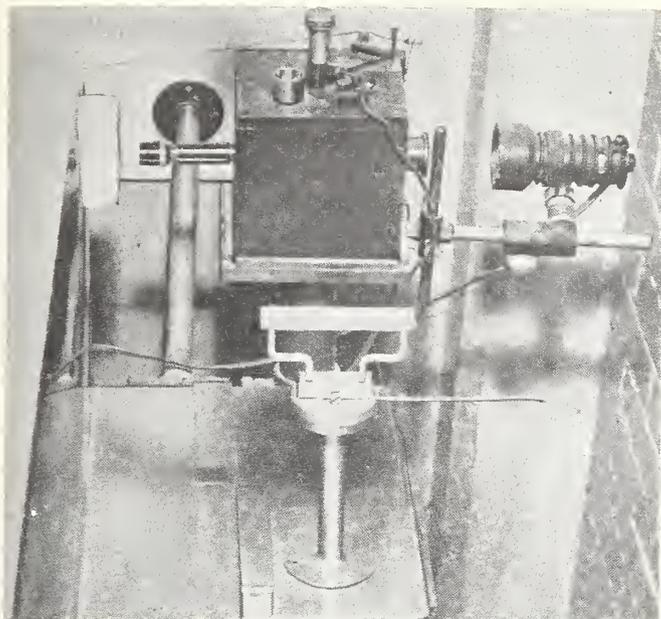


FIGURE 4. *The National Bureau of Standards standard gold-leaf electroscope (Curtiss, 1928).*

The ionization chamber consists of a 10-cm-cube free-air volume, with walls made of 1-cm thick lead sheet with $\frac{1}{2}$ -cm thick aluminum inner lining. A gold leaf is suspended near the center of the chamber and a quartz fiber, 10μ in diameter, at the free end of the leaf provides a fine line for optical projection, with a magnification of 100, onto a metric scale. Transit times of the quartz-fiber image between two fixed points on the scale 6-cm apart are normally measured.

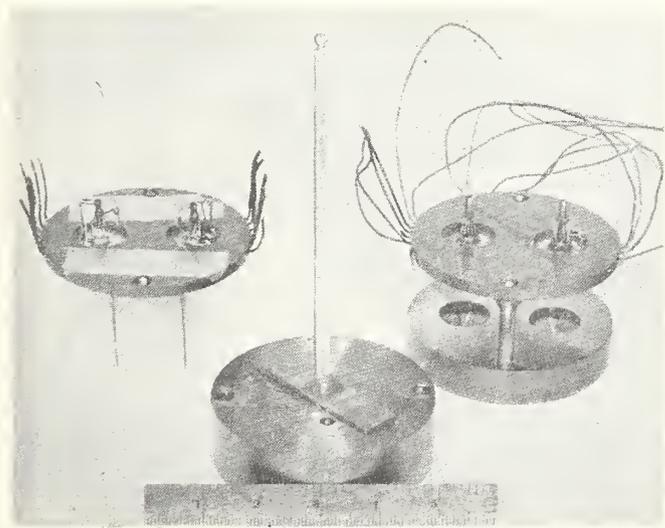


FIGURE 5. *Two early versions of the radiation balance or twin-cup Peltier-effect microcalorimeter (Mann, 1954a).*

The two copper disks on which the microcalorimeter cups are mounted both fit the same copper base and copper cover, the latter with thermometer. The microcalorimeter assembly on the left actually has four Peltier junctions attached to each cup, two above and two below the equatorially-located thermopile junctions. The assembly on the right has only one Peltier junction attached to the base of each cup. Provided that the thermal conduction in the cups is adequate, it appears that no difference in results is obtained by distributing the cooling along the cup. A precision of some ± 0.5 percent was obtained with the single Peltier-junction microcalorimeter working with no thermal isolation of the microcalorimeter from its surroundings. The lighter shaded metal on the left-hand disk is of lead and was used to assure good thermal contact between the disks and the copper base and cover when assembled.

international or national radium standards, either with each other or with other radium preparations. In order to compare radium preparations with a radium primary standard, using the radon method, it is necessary that a secondary radon standard be prepared by comparing a suitable amount of radium salt with a primary radium standard, calorimetrically or by gamma-ray measurements. From this calibrated radium salt an aqueous solution is then prepared, from which equilibrium amounts of radon can be obtained by boiling or by bubbling through the solution an inert gas such as nitrogen. Initially, electroscopic, balanced ionization-chamber or ionization-current measuring methods were used to measure the relative amounts of radon collected (Rutherford and Boltwood, 1905 and 1906; Strutt, 1905; Paneth and Koeck, 1931; Evans, 1935; Urry, 1936; Urry and Piggot, 1941), but latterly these amounts have been determined by counting the alpha particles in ionization chambers (Curtiss and Davis, 1943) (figs. 7 and 8). Recently, however, scintillation counting techniques have been applied to the comparison of amounts of radon by measuring the alpha particles arising from radium-A recoils in a gas, these ionized radium-A atoms being attracted electrostatically to an extremely thin aluminum foil and counted by means of a thin scintillator of zinc sulfide, adjacent to the aluminum foil, used in conjunction with an electron-multiplier phototube (Bryant and Michaelis, 1952; Cowper and Simpson, 1953) (fig. 9). The radon method of analysis of radium gives results that are reproducible with a standard deviation of the order of 1.0 percent, and can, in addition, be used to determine the amount of radium present, for example, in water or in uranium ores, in the latter case giving a measure of their uranium content, provided that there has been no leaching and that the radium is in equilibrium with the uranium.

Other daughters of radium, and members of the uranium family, that are of importance in standardization are radium D+E, for beta-reference sources, and radium D+E+F, or radium F (polonium-210) alone, for alpha-ray standards. Radium D sources can be electrolytically or chemically deposited onto palladium-clad silver disks, and polonium-210 can either be grown from radium D or else plated from polonium-210 solution onto the same disks. In all three cases extremely thin sources of beta or alpha activity are formed. The disintegration rate of the radium D (lead-210), in equilibrium with the energetic beta emitter radium E, is determined by electrodepositing a known amount of lead that has been quantitatively extracted from a pitchblende, the radium content of which has previously been determined by the radon method. Secondary sources of radium D+E can then be calibrated by comparison with one of these primary radium D+E standards by means of an end-window Geiger-Müller counter. The alpha emission from thin alpha standards of

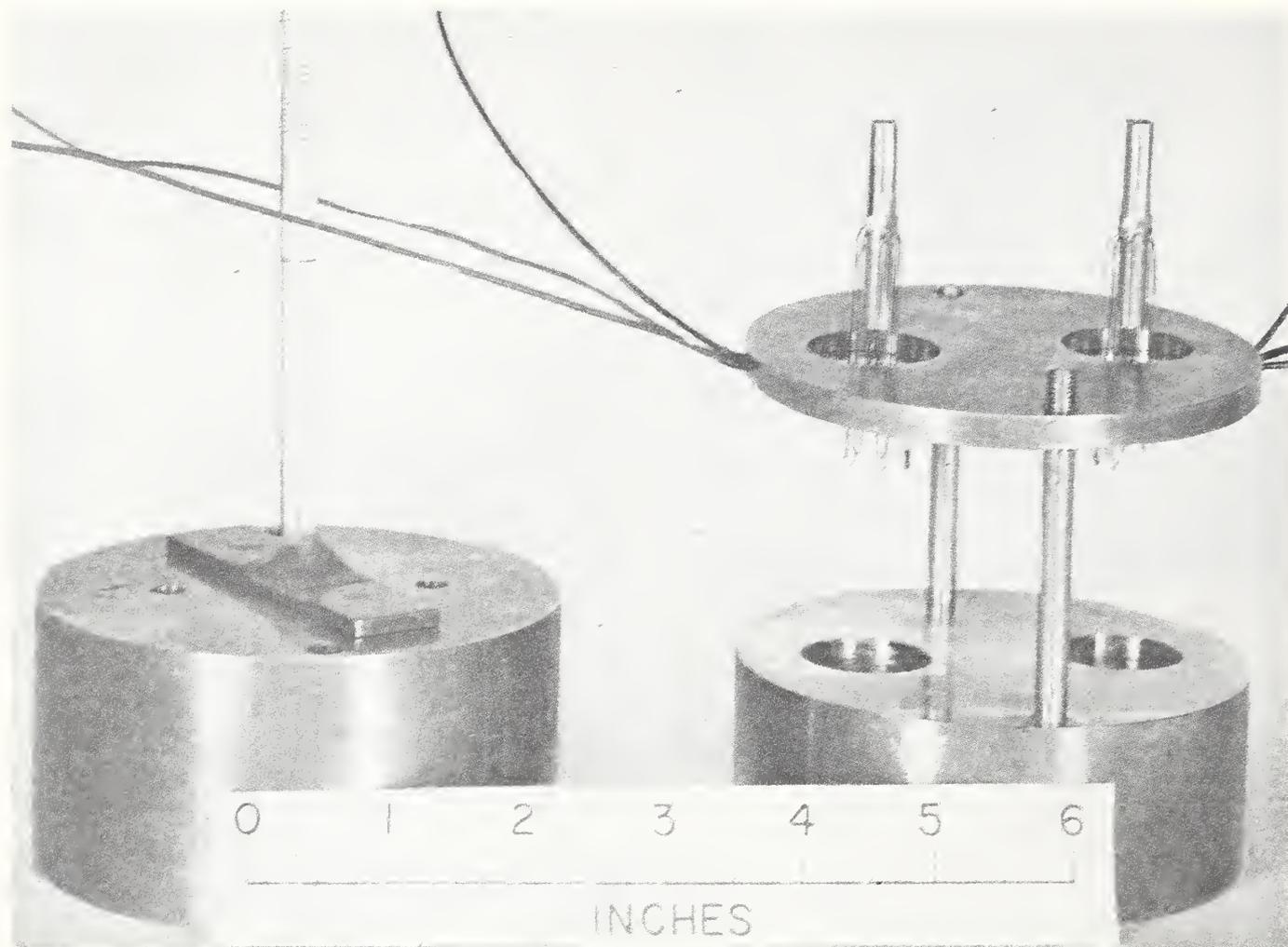


FIGURE 6. The radiation balance or twin-cup Peltier-effect microcalorimeter that was constructed for the comparison of a number of national Hönigschmid radium standards (Mann, 1954b; Davenport, McCraven, Mann, and Smith, 1954; Connor and Youden, 1954; Loftus, Mann, Paoletta, Stockmann, and Youden, 1957).

The cups of this radiation balance were of gold and it was enclosed in a temperature-attenuating enclosure to give a precision in the comparison of the national standards of about ± 0.1 percent. The scale sensitivity, which limits the precision of the lowest rate of energy dissipation measurement, is about $3 \text{ mm}/\mu\text{w}$.

radium D+E+F or of radium F (polonium-210) can be determined by means of a $2\pi\alpha$ -proportional-flow counter (fig. 10). Unless, however, the sources are thin, considerable straggling in the source will occur and give rise to plateaus that are not flat. Polonium-210 sources can also be standardized by determining their total gamma-ray emission, about 1 in every 10^5 alpha disintegrations giving rise to a less energetic alpha transition to an excited level of lead-206 with subsequent emission of a 0.804-Mev gamma ray. This alpha-branching ratio has recently been determined with a view to applying the value to the standardization of strong polonium-210 alpha sources (Grace, Allen, West, and Halban, 1951; Hayward, Hoppes, and Mann, 1955; Rojo, Hakeem, and Goodrich, 1955; Ascoli, Asdente, and Germagnoli, 1956).

Apart from radium and its products, there is little interest in the standardization of the naturally occurring radioactive elements with the possible exception of mesothorium or radiothorium as a generator of thorium X, an alpha emitter, for

incorporation into a solution or ointment for the treatment of skin lesions. The demand for this is so inconsiderable, however, that the standardizations are normally carried out, as the need arises, by measuring the number of 2.62-Mev gamma rays from thorium C'', and calculating their build-up in relation to the thorium X. This standardization can be preserved in terms of an ampoule containing a solution of about $10 \mu\text{g}$ of radium chloride solution by means of an ionization chamber in the manner that is to be discussed later in connection with the maintenance of standards of the artificially produced nuclides.

2.2. Primary Standardization of Artificially Produced Beta-, Gamma-, and X-ray Emitters

When one turns to the problem of standardizing the artificially produced radioactive nuclides, the methods of measurement become as varied as the properties of the radiations that are emitted by such nuclides. There are low-energy and high-

RADON ANALYSIS

To PROTECT THE HEALTH AND SAFETY OF PERSONNEL ENGAGED IN WORK INVOLVING RADIUM THE NATIONAL BUREAU OF STANDARDS MAINTAINS CAREFUL CHECK, THROUGHOUT THE UNITED STATES, OVER THE QUANTITY OF RADIOACTIVE SUBSTANCES INGESTED BY PERSONNEL IN RADIUM INSTALLATIONS AND OVER THE QUANTITY OF RADIATION PRESENT IN THEIR WORKING AREAS.. THE BREATH OF PERSONNEL OR THE AIR IN THE WORK ROOM IS SAMPLED PERIODICALLY AND IS ANALYZED.. ANALYSIS REVEALS THE AMOUNT OF RADIATION PRESENT AND PROVIDES THE BASIS FOR THE ESTABLISHMENT AND MAINTENANCE OF PROPER SAFETY MEASURES.

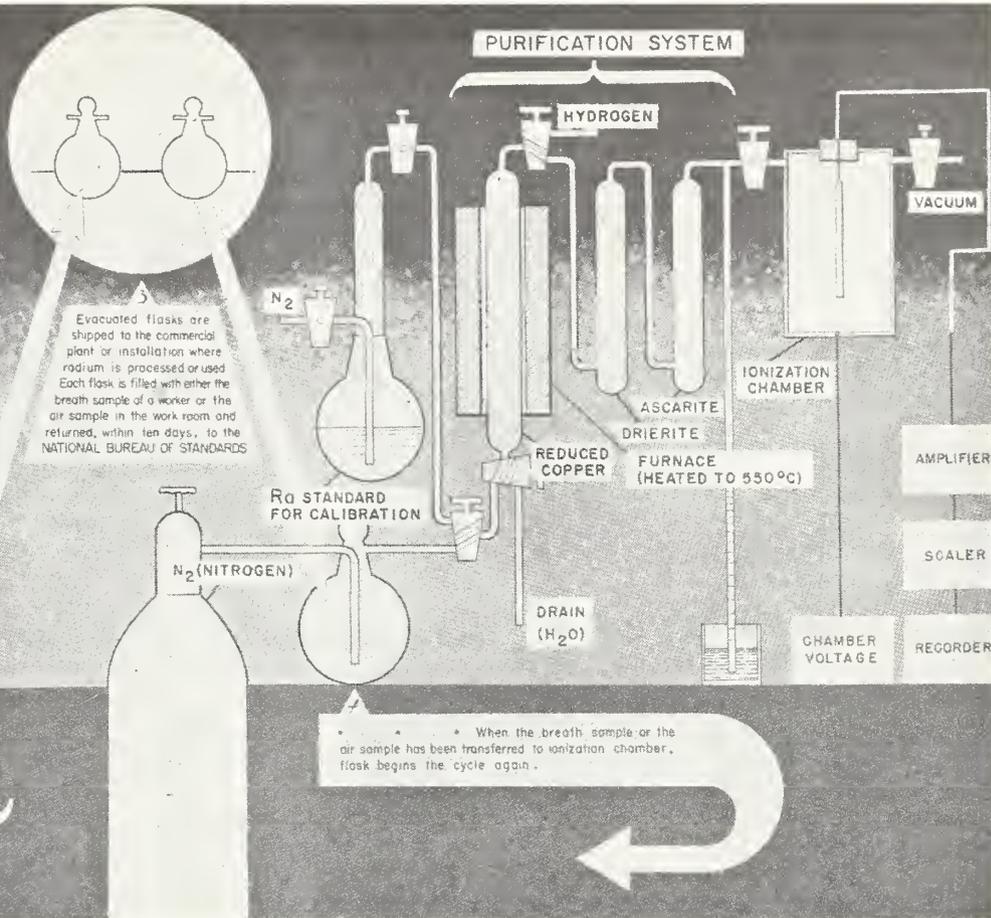


FIGURE 7. Flow chart illustrating the method of radon analysis in use at the National Bureau of Standards (Curtiss and Davis, 1943).

energy pure beta emitters, each calling for its own method of measurement, beta-gamma emitters and also electron capturers or X-ray emitters. Each particular case must be treated on its merits and may call for exhaustive prior investigations of the mode of disintegration of the nuclide. Thus a pure beta emitter may call for one kind of measurement, whereas a radioactive nuclide that decays by emitting an electron, followed immediately by the emission of a gamma ray from an excited state of the daughter nucleus, may be dealt with by a variety of methods, depending both on the degree of certainty with which the particular nuclear decay scheme is known and its complexity.

For beta emitters with maximum energy greater than about 300 kev, the best, and most widely used, method of primary standardization is that of $4\pi\beta$ counting, either in the proportional or Geiger regions. This method involves the use of a thin source mounted on as thin a conducting film as possible and looked at on both sides by two hemispherical, semicylindrical or "pill-box" counters each subtending a solid angle of essentially 2π at the source. The use of this method was suggested first in 1944, but the counter then described (Simpson) appears to have had somewhat appreciably less than a completely 4π geometry. The first such counter to attain to what is now re-

garded as essentially a 4π geometry was designed by Schutzmeister-Meyer and described in 1948 (Haxel and Houtermans). The use of such counters has since been carried to a high standard of working efficiency by a number of investigators (Putman, 1949; Cohen, 1949 and 1952; Sawyer and Wiedenbeck, 1950; Borkowski, 1950; Charpak and Suzor, 1950; Seliger and Cavallo, 1951; Suzor and Charpak, 1951, 1952a, and 1952b; Hawkings, Merritt, and Craven, 1952; Houtermans, Meyer-Schutzmeister, and Vincent, 1952; Meyer-Schutzmeister and Vincent, 1952; Gray, 1952; Mann and Seliger, 1953; Smith, 1953 and 1954; Smith and Wildblood, 1953; Pate and Yaffe, 1955a, 1955b, 1955c, 1955d, and 1956) (figs. 11, 12, and 13).

To avoid source self-absorption of electrons at the low-energy end of the beta spectrum, it is essential to reduce solids in the source to a minimum. For this reason, it is often important to precipitate the source material chemically from the drop of solution that has been deposited on the thin-film source holder, or to "freeze-dry" it in position. In the process of simple evaporation, the slow reduction in physical size of the drop of solution results in an ever-increasing concentration of solute in the residual drop, and a final deposition of solids from the vanishing drop. The structure of precipitated low-solids sources has



FIGURE 8. A rack of six ionization chambers with associated linear amplifiers and scalars.

The outputs of the scalars, giving the numbers of alpha particles counted, are recorded on traffic recorders hourly throughout the night. The transfer of the samples from the test flasks (shown on the bottom shelf) to the ionization chambers is effected during the day. Oxygen is removed, to reduce recombination effects, by passing the gas samples over heated copper in electric furnaces, which can be seen in the top left-hand corner.

been investigated by the method of electron-shadow-micrography in the case of silver-iodide (iodine-131) sources (Seliger and Schwebel, 1954), and it has been found that the deposition of such sources occurs in almost spherically shaped "islands" surrounded by large vacant areas (fig. 14). The addition of solids therefore merely serves to increase the number of "islands" without essentially changing their size and, thus, to fill in the empty spaces without increasing the source self-absorption in any manner that can be subjected to analysis by the method of extrapolation. The counting rate on addition of inactive solids can, in fact, up to a certain point remain unchanged. It has been shown that in the case of cobalt-60, which has a maximum beta energy of 310 kev, the source self-absorption can be as high as 10 percent in $4\pi\beta$ -proportional counting (Seliger and Schwebel, 1954). The method has, on the other hand, been used for sulfur-35 (Pate and Yaffe, 1955c; Seliger, Mann, and Cavallo, 1958), sources of which can be prepared with practically negligible solids, even though the maximum beta energy is considerably lower than that of cobalt-60.

Measurements of the charge carried away from a source of radium have been made by several investigators (Wien, 1903; Rutherford, 1905; Makower, 1909; Moseley, 1912). Recently, how-

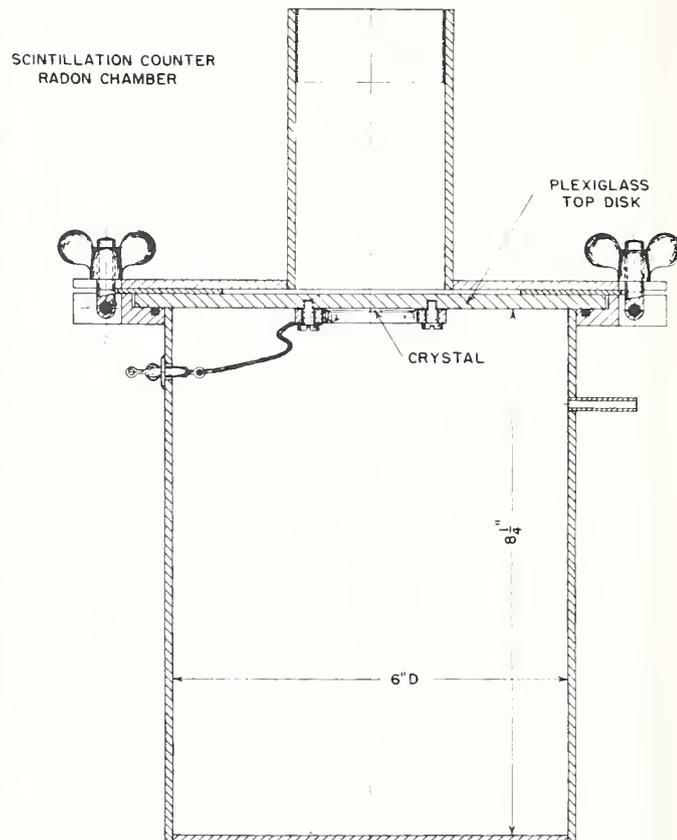


FIGURE 9. Cross-sectional drawing of scintillation counter for radon analysis constructed at the National Bureau of Standards and based on the method of Bryant and Michaelis (1952).

The multiplier phototube is located in the upper tube and looks at the scintillator consisting of a thin layer of zinc-sulfide crystals. An aluminum foil close to and parallel to the zinc-sulfide layer is maintained at a negative potential of some 2,000 v, with respect to the surroundings. On admitting a radon sample into the chamber, radium-A recoils are attracted electrostatically to the aluminum foil and the subsequent alpha particles striking the zinc-sulfide screen are counted. The equipment is calibrated by admitting a sample of radon from a radium solution standard.

ever, this method has been applied to the standardization of beta emitters (Clarke, 1950; Gross and Failla, 1950; Keene, 1950). In 1950, experiments were described using both spherical (Clarke) and parallel-plate (Gross and Failla) geometry. The former achieved agreement with National Bureau of Standards standard samples of phosphorus-32 to within 5 percent, but has not since been in general use. In the parallel-plate geometry the source is a very thin deposit of the emitter in question on one plate of a parallel-plate collecting system, the second plate being sufficiently close to balance the secondary electron emission and backscattering effects. Low-energy secondary electrons are bent back by means of a weak magnetic field, and the measurement is made in a vacuum to eliminate air ionization. General agreement with the National Bureau of Standards standard samples of phosphorus-32 to within ± 2 percent has been obtained by this method over a number of years (Seliger and Schwebel, 1954). In another development of the charge-measuring

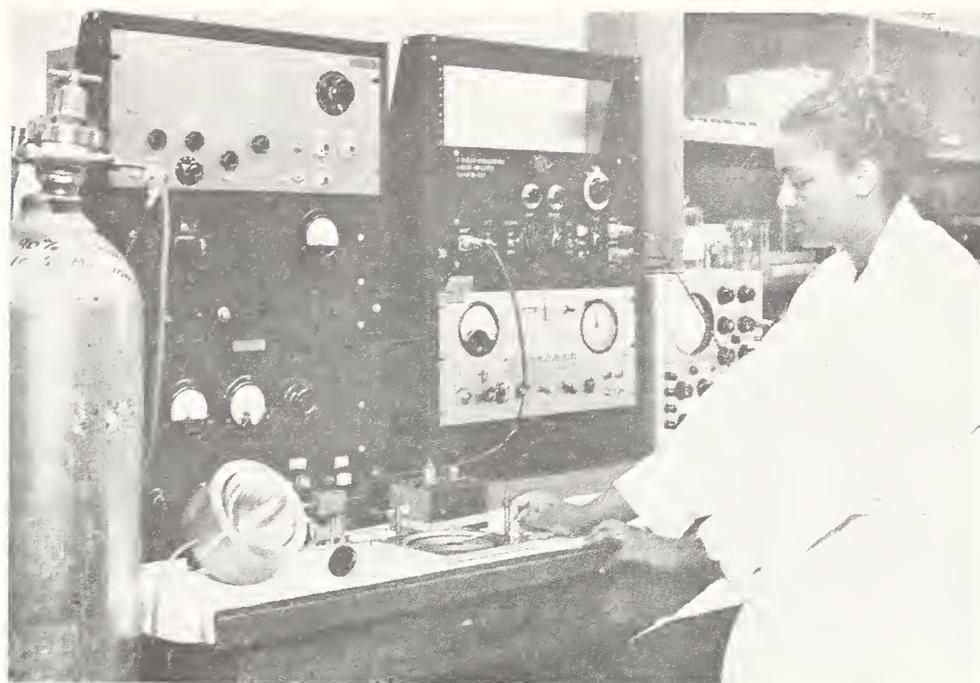


FIGURE 10. *The $2\pi\alpha$ -proportional counter used for the measurement of alpha-particle standards.*

The diameter of the steel hemisphere of the counter is 11.9 cm and the counter has been found, by surveying with an approximately point alpha-particle source, to give a flat response over an area in the center, which is considerably greater than that of the alpha-particle sources to be measured. Hemispheres of smaller diameter had previously been found to give too rapid a fall-off in response with distance from the center.

method (Keene, 1950), the source is deposited on a thin aluminum foil that is completely surrounded by an open-mesh wire-grid enclosure. This experiment, like the first, is carried out in a vacuum, and the current from the foil to the grid is measured for different positive and negative voltages between them. Agreement to within about 10 percent for the calibration of a phosphorus-32 source from the Atomic Energy Research Establishment at Harwell is quoted.

For low-energy beta emitters there are two general methods of primary standardization available. There are the methods of gas counting and of calorimetry. In the former the radioactive nuclide such as hydrogen-3, carbon-14, or sulfur-35 is admitted in the form of a gas into a cylindrical-cathode gas counter operating in either the proportional or Geiger regions. With the investigators who have used this method there are two general philosophies, namely, that which advocates the use of long counters having negligible or calculable end corrections due to the fall-off in electric field or change in active volume (Miller, 1947; Brown and Miller, 1947; Anderson, Libby, Weinhouse, Reid, Kirshenbaum, and Grosse, 1947; Engelkemeir, Hamill, Inghram, and Libby, 1949; Jones, 1949; Bernstein and Ballentine, 1950; Engelkemeir and Libby, 1950), and that which promotes the simultaneous use of two or more counters identical in every respect except for the cathode and anode wire lengths (Mann and Parkinson, 1949; Hawkings, Hunter, Mann, and Stevens, 1949; Hawkings, Hunter, and Mann, 1949; Manov and Curtiss, 1951; Mann, 1958). By taking the difference in counting rates for such

a compensated set of counters, the counting rates are obtained for ideal cylinders, equal in length to the differences in the lengths of the individual counters (figs. 15, 16, 17, and 18). Investigators of the former predilection have investigated the effect of varying length, and most of the workers in both disciplines have experimented with counters of varying radii, with a view to determining the magnitude of the wall correction arising from the fact that beta particles proceeding towards the wall from a finite layer of gas contiguous to the wall may not be counted. The thickness of this layer will depend on the number of ion pairs formed per unit length of beta path, and upon the minimum number of ion pairs required to give a count. As the number of ion pairs formed per unit path-length will be proportional to gas pressure, this may also be varied in an investigation of the wall effect.

It is of importance in gas counting to be assured that any chemical reactions used in the preparation of the counting gas from the radioactive sample shall have been carried to completion, so that the measured radioactivity of the gas may be quantitatively related to the radioactivity of the original sample independent of any change in isotopic abundance that might occur in an incomplete reaction. Alternatively, it may be more satisfactory to convert the counting gas itself quantitatively back to compounds that can be totally absorbed or trapped after the counting experiment has been completed. Thus, in the standardization of hydrogen-3 or carbon-14, the quantitative preparation of, say, radioactive acetylene, in which the hydrogen-3 or carbon-14

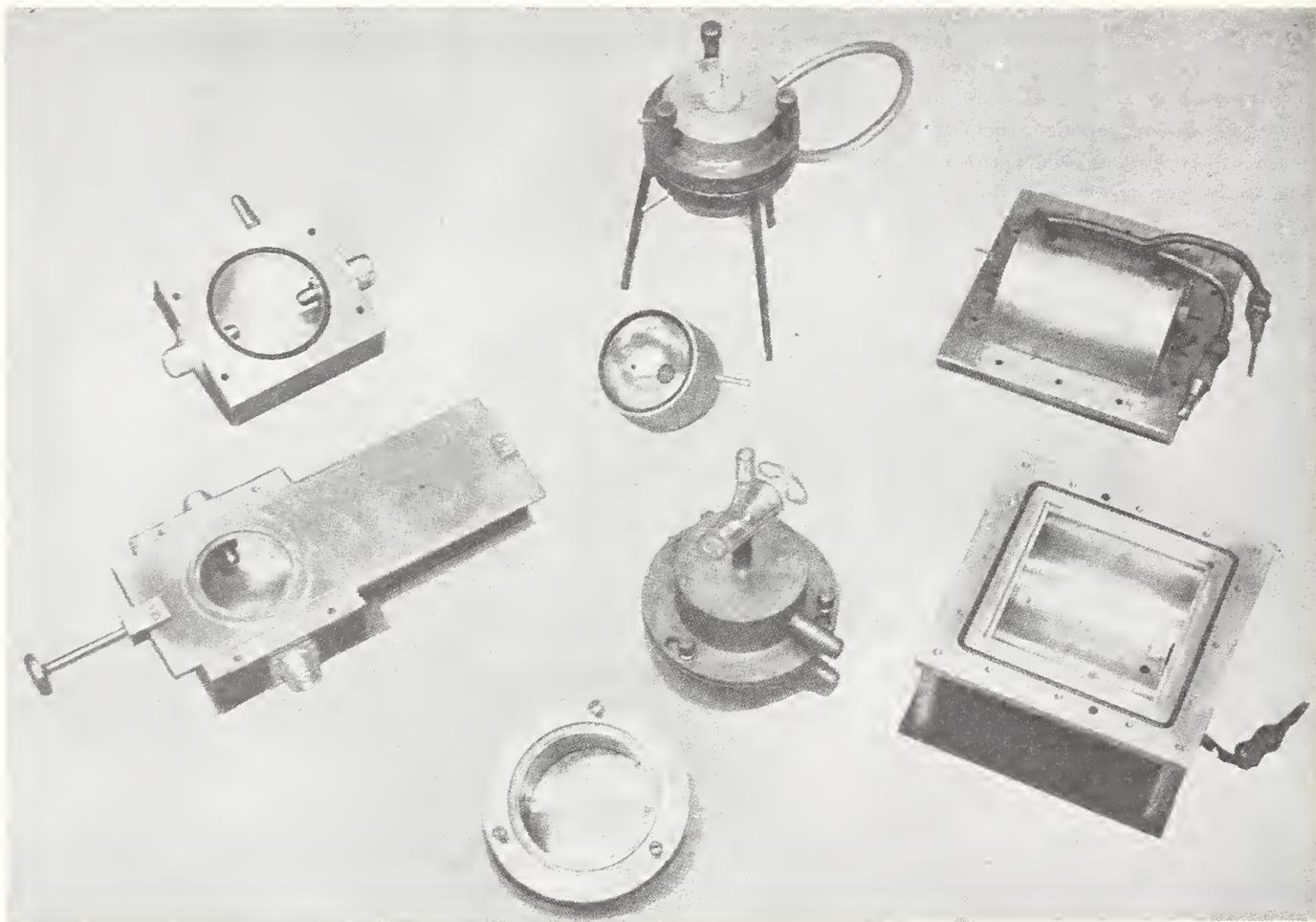


FIGURE 11. A selection of the $4\pi\beta$ -proportional counters that have been used at the National Bureau of Standards for the calibration of beta-ray standards (Seliger and Cavallo, 1951; Mann and Seliger, 1953; Seliger and Schwebel, 1954).

The parts of the type currently in use are shown in the center of the photograph, and in figures 12 and 13.

is incorporated in the acetylene molecule, may be difficult. Any sample of radioactive acetylene that has not been quantitatively prepared can, however, be quantitatively converted by complete combustion, after counting, to water and carbon dioxide. The measured radioactivity of this water or carbon dioxide can then be compared with the original sample by relative counting techniques, such as the liquid scintillation counter or the gas ionization chamber. The only slight drawback to this method would lie in the fact that the amount of the primary standard available would be somewhat limited.

Electron-capturing nuclides that exist in suitable gaseous compounds should be susceptible of primary standardization by gas-counting methods, provided that adequate information regarding the fluorescence yield is available.

In some cases where macroscopic amounts of the disintegrating nuclei can be obtained, the rate of disintegration can be determined by collecting and measuring the amounts of the decay products. Such techniques, that have in the past been used to determine the ages of rocks containing the naturally occurring radioactive elements, have recently been most successfully applied (Jones,

1955) to obtaining the half-life of tritium by determining the initial isotopic abundance and the number of disintegrations in a given time, the latter being measured by collecting the helium-3 formed and assaying it mass-spectrometrically. This method provides an absolute measurement of the disintegration rate, but it has application in only a limited number of specific cases.

Microcalorimetric methods have been used at the Chalk River laboratories of Atomic Energy of Canada Limited to measure the disintegration rate of hydrogen-3 (Hawkings and Merritt, 1954), and at the National Bureau of Standards to determine the disintegration rate of both hydrogen-3 and sulfur-35 (Gross, Wingate, and Failla, 1956 and 1957; Bay, Mann, Seliger, and Wyckoff, 1957). As there is no published account of the hydrogen-3 standardization carried out at the National Bureau of Standards, it may be appropriate to mention that the rate of energy emission from about 4.8 curies of tritiated water contained in a small quartz tube was measured over a period of some 4 months in the radiation balance developed to compare the 1934 Hönigschmid radium standards (Mann, 1954b). The tritiated water had been sealed into the quartz tube under some 2-cm-Hg pressure of



FIGURE 12. A closeup view of the $4\pi\beta$ -proportional counter in use at the National Bureau of Standards showing details of the source mounted on plastic supported by aluminum foil.

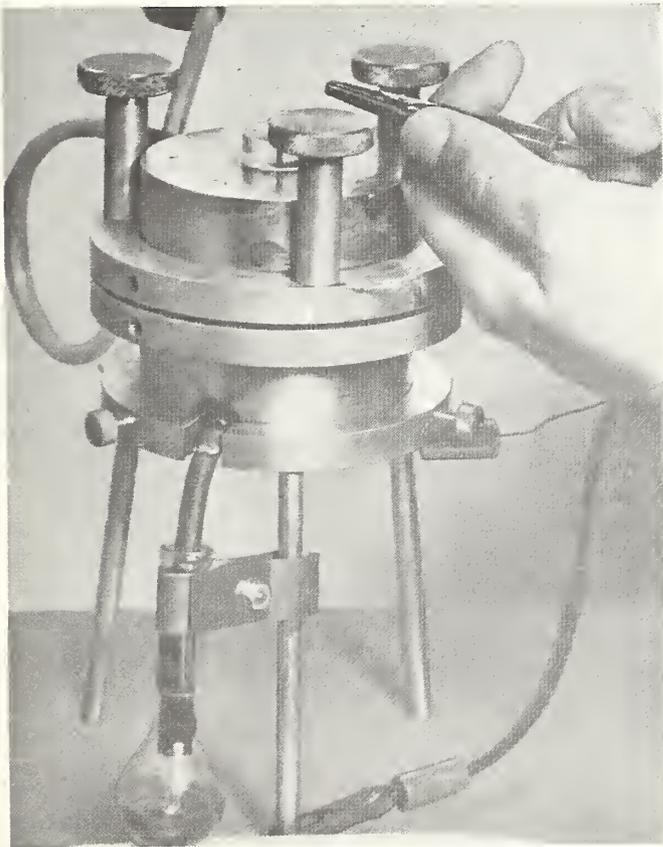


FIGURE 13. The National Bureau of Standards $4\pi\beta$ -proportional counter assembled for use.



FIGURE 14. Chromium-shadowed electron micrograph of a silver-iodide source deposited on a plastic film.

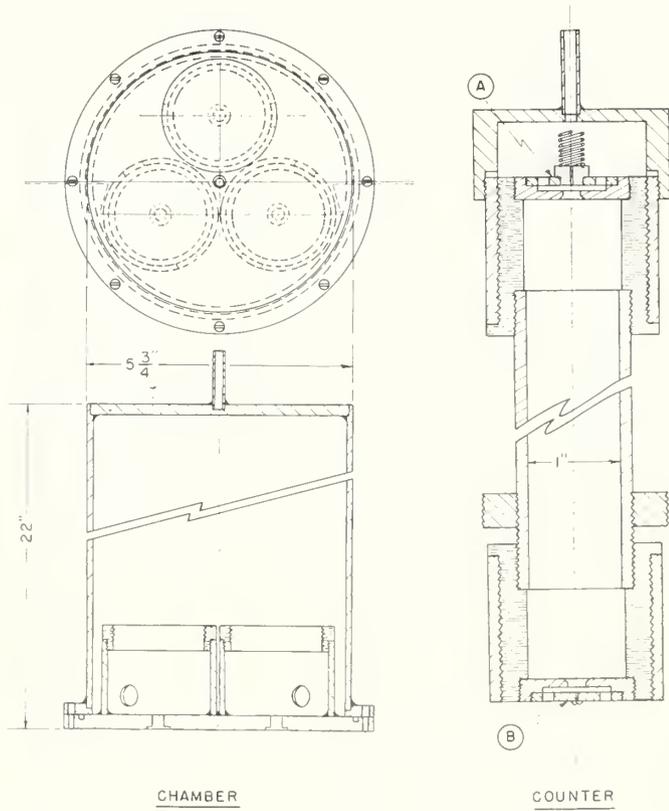


FIGURE 15. Workshop drawing of a set of compensated gas counters that have been constructed with copper anodes with Teflon-insulated stainless-steel anode-wire supports at each end (Mann, 1958).

The tolerances of the end construction have been kept to within a few thousandths of an inch so as to achieve accurate compensation for end effects. The internal diameter of the copper anodes is 1 in. The anode lengths are 6.0012, 8.0017, and 16.0003 in.

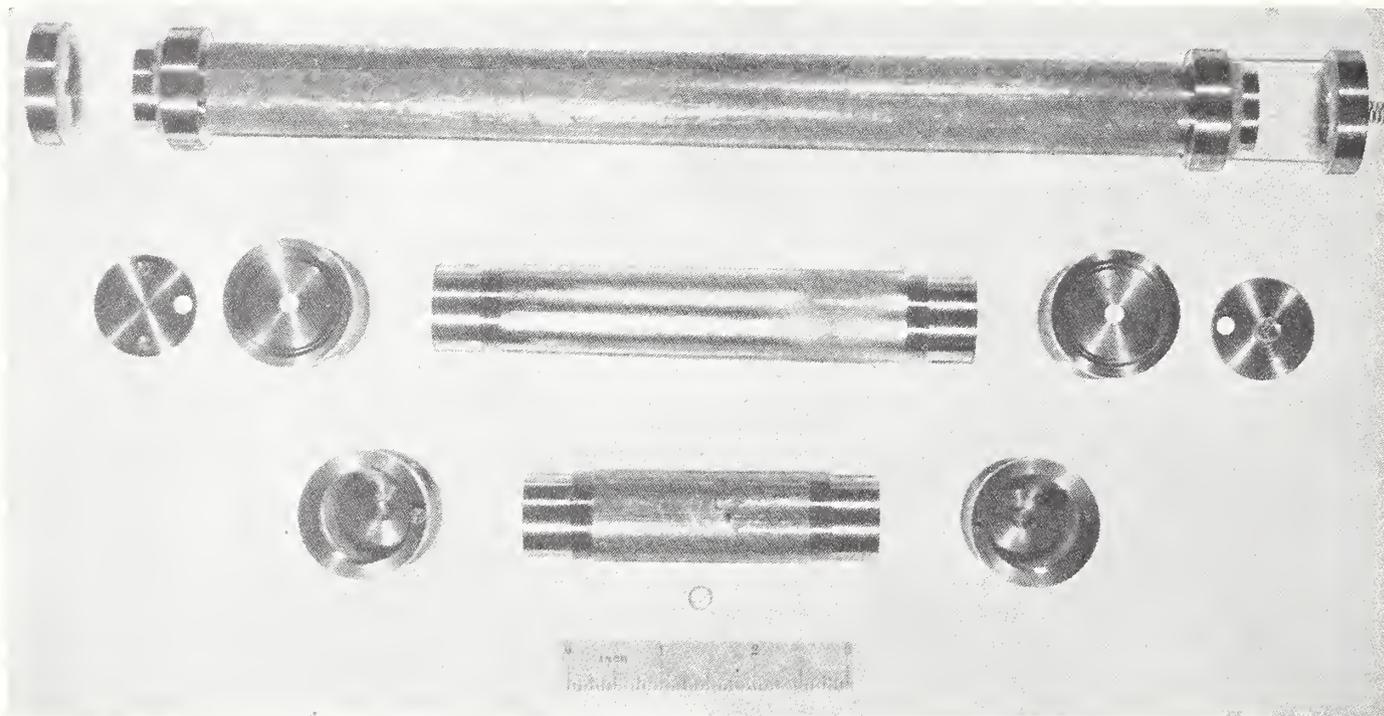


FIGURE 16. A set of three stainless-steel compensated counters with glass and stainless-steel ends (Mann, 1958)

The glass insulators are cemented into stainless-steel rings which are in turn screwed on to the stainless-steel cathodes by means of carefully cut micrometer threads. The ends were mounted onto the cathodes using a traveling microscope to insure that the distance from the end of each cathode to the stainless-steel disk supporting the tungsten anode wire was the same in all of the counters. The anode lengths of the stainless-steel counters are 3.7586, 5.7410, and 11.7623 in.

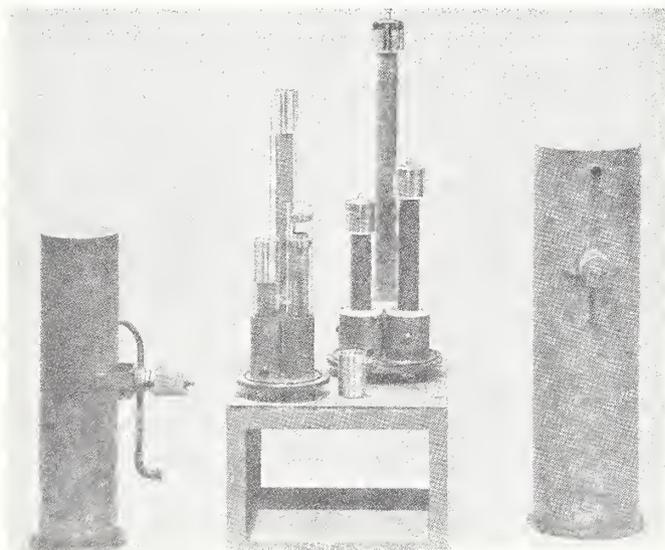


FIGURE 17. The compensated gas counters described in figures 15 and 16 assembled ready for enclosure in their respective vacuum containers.

The stainless-steel counters are on the left and the copper on the right.

hydrogen to inhibit the formation of H and OH radicals as a result of irradiation of the water (Ghormley and Allen, 1948). After measurement in the microcalorimeter, this tube of tritiated water was crushed under 1 kg of water in a specially constructed stainless steel container, and the master solution so obtained diluted to give standards of activity around 10^4 and 10^6 dps/ml. The exact disintegration rate was obtained from the energy emission rate per milliliter and the assumed mean energy of 5.67 kev per disintegration for

hydrogen-3² (Jenks, Ghormley, and Sweeton, 1949).

For beta emitters that are associated with one or more gamma rays in coincidence, the method of respectively beta-gamma or gamma-gamma coincidence counting is one of the most powerful standardizing tools available. The earliest known application of this method was by H. Geiger and A. Werner (1924), to determine the efficiencies of two observers looking at the same zinc-sulfide screen through two microscopes and counting the number of scintillations due to alpha particles. The application of the method of coincidence counting to the determination of the efficiencies of beta or gamma counters (Dunworth, 1940) has the added advantage that these efficiencies normally remain constant and do not suffer from such human frailties as fatigue and susceptibility to diversion. The method of coincidence counting has been used in conjunction with $4\pi\beta$ counters (Smith, 1953 and 1954) in the standardization of cobalt-60. In the standardization of cobalt-60 by the method of gamma-gamma coincidence counting, using sodium-iodide (thallium-activated) crystals as the detectors in each channel, the effects of back-scattered gamma rays from one counter giving rise to a coincidence count in the other is readily eliminated by setting the discrimination levels to exclude all such backscattered gamma rays. By introducing varying resolving times by the use of delay lines of varying length, it is also possible to obtain a linear plot of coincidence counting rate against time (or delay-line length) and to

² The mean energy for disintegration is the mean beta-ray energy less the difference in the total electron binding energies of hydrogen-3 and helium-3.

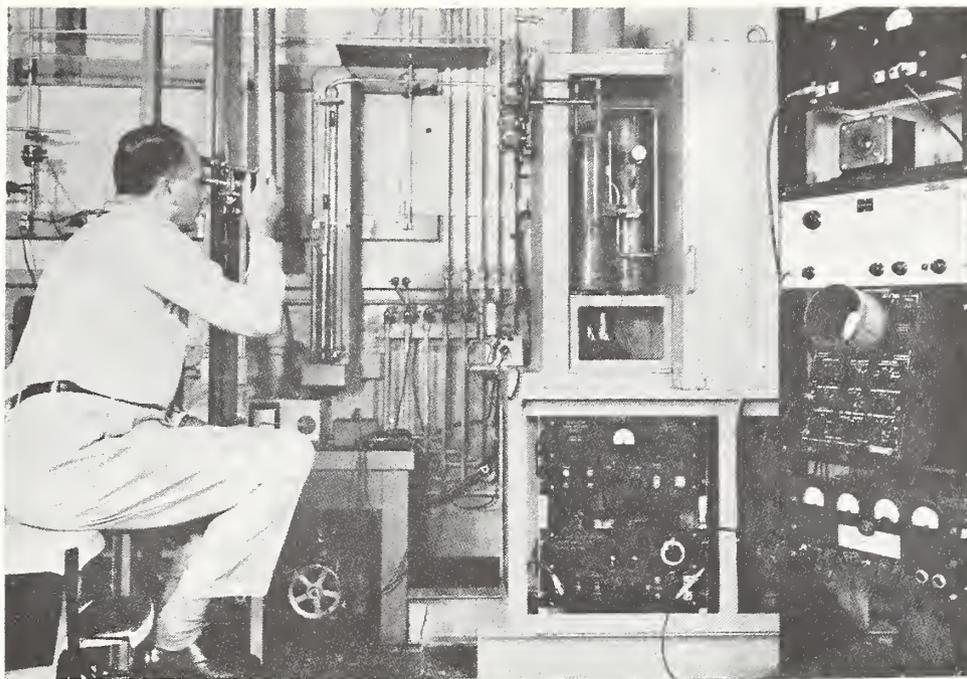


FIGURE 18. The National Bureau of Standards compensated gas counters for the counting of low-energy beta emitters and electron-capturing nuclides assembled within a steel shield and with their auxiliary gas-handling equipment.

extrapolate to zero resolving time (or zero length) to obtain the true coincidence rate without accidentals (Hayward, 1953). In conjunction with fast circuitry, this method is of great use in the standardization of such nuclides as cobalt-60 and sodium-24 (fig. 19).

In the application of beta-gamma coincidence scintillation counting it is important to minimize the efficiency of the beta detector to gamma rays. This can readily be done by grinding the crystal detector used in the beta channel down to a thickness of about 0.5 mm (Seliger and Schwebel, 1954) (fig. 20), although there is always a finite effect due to Compton electrons produced in the glass-faced photocathode of the electron-multiplier phototube. While it had been tacitly assumed that the source, in the coincidence technique, was a point source it was later shown that the method is equally applicable to extended sources provided that the efficiency of either detector is the same for all parts of the source (Putman, 1950). Such would be the case using a $4\pi\beta$ counter as the detector in the beta channel (Campion, 1958).

Electron-capturers that emit a nuclear gamma ray in coincidence with the K and L X-rays can also be standardized by the method of X-ray—gamma-ray coincidence counting.

The principle of $4\pi\beta$ counting has recently been extended to crystal scintillation counting by forming a sandwich of two anthracene crystals with the source deposited between the sandwich on one of the crystals (Smith, Seliger, and Steyn, 1956; Mann and Seliger, 1956) (fig. 21). Two phototubes look at the sandwich from opposite sides, and true counts in the crystal are recorded as coincidences between the two phototube channels. Efficiencies of better than 99 percent have

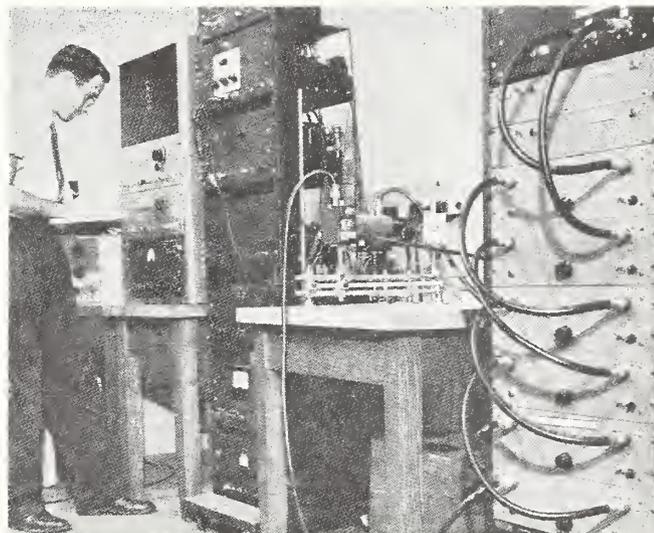


FIGURE 19. Equipment for the determination of the disintegration rate of a source of cobalt-60 by the gamma-gamma coincidence method (Hayward, 1953).

The angular correlation between the directions of emission of the two gamma rays is used to obtain the disintegration rate under different experimental conditions such that the presence of many spurious effects may be detected easily.

been obtained by this method for phosphorous-32, strontium-90-yttrium-90, and thallium-204. The low threshold of about 4 or 5 keV of this method enables one to hope that it will be possible to use it to standardize electron-capturing nuclides.

The method of $4\pi\beta$ -liquid-scintillation counting has been applied to the standardization of alpha and beta emitters (Belcher 1953; Basson and Steyn, 1954; Steyn 1956, Seliger 1958a, 1958b, and 1958c). Belcher (1953), using beta emitters, supported the source in the liquid scintillator between a sandwich of two thin foils. The absorption

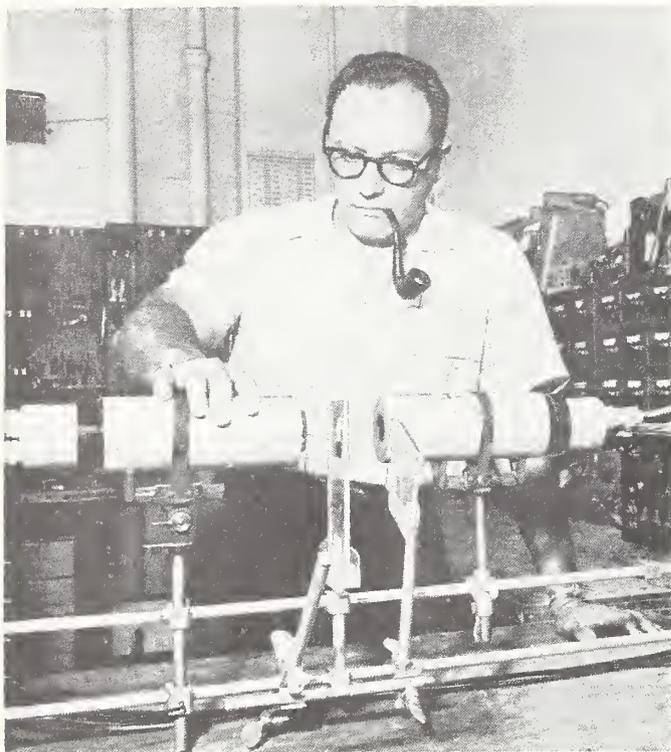


FIGURE 20. Photograph of the low-geometry β - γ scintillation coincidence-counting arrangement at the National Bureau of Standards.

The source is mounted on a thin plastic film between the beta detector on the right and the gamma detector on the left. The aluminum absorbers that are used to estimate the gamma-counting rate in the beta detector can be rotated to be directly in front of the beta detector which is an anthracene crystal ground down to a thickness of approximately 0.5 mm. The gamma detector is a sodium-iodide (thallium-activated) crystal. Coincidence resolving times of the order of 0.5 μ sec are usually used. In actual practice, the β , γ , and β - γ channels are always counted over the same interval. In this way the disintegration rate obtained is independent of any changes in efficiency of the β or γ detector due to slight drifts in the electronic circuits.

correction for these foils and for source self-absorption can, however, be quite high. Basson and Steyn (1954) and Seliger (1958c) have standardized alpha particle solutions of polonium-210 by incorporating the solution directly into the liquid scintillator, and Steyn (1956) and Seliger (1958a and 1958b) have pushed the technique to the standardization of beta-emitting nuclides incorporated as a solution in the liquid scintillator.

2.3. Secondary Standardization of Artificially Produced Beta-, Gamma-, and X-ray Emitters

The simplest way of effecting a comparison between a primary and secondary standard of radioactivity is by means of a substitution method under reproducible conditions of geometry, the relative intensities of radioactivity being measured by any instrument that records either individual beta or gamma rays or else measures their ionizing effects. Such a method, whatever the nature of the recording device, can, in the hands of a competent observer, be relied upon to give results having a standard deviation of from 0.1 to 1.0 percent. It is also to be understood that within the meaning of "reproducible conditions of

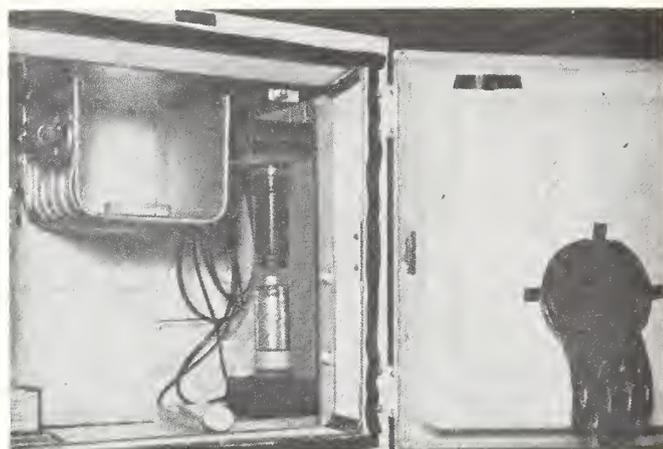


FIGURE 21. Photograph of the 4π -crystal-sandwich-coincidence-counting arrangement (Smith, Seliger, and Steyn, 1956; Mann and Seliger, 1956).

The source is delivered directly to the polished face of one anthracene crystal. The second crystal is placed directly on top and the sandwich surrounded by an aluminum-foil cylinder. The flat phototube faces make contact with the crystal faces during the measurement. The refrigerator also serves as a light-tight enclosure.

geometry" is to be included the method of source preparation. The method of comparing radioactive sources of unknown strength with calibrated radium D+E sources is no longer very acceptable, on account of the fact that the geometrical conditions are, on the whole, somewhat irreproducible and involve considerations of absorption and backscattering. The method has, however, been shown to give consistent results with the use of "weightless" sources mounted on thin films (Novoy, 1950). Radium D+E sources are now, in general, only recommended for use as reference sources for the maintenance of the calibration of measuring equipment by the method of substitution in which the initial calibration has been made using a standard sample of the nuclide in question.

The recording device used in such a substitution method can be a gold-leaf or quartz-fibre electro-scope; a beta ionization chamber or gamma ionization chamber used in conjunction with a Lindemann-Ryerson, or vibrating-reed electrometer, or any other low current-measuring device; any kind of beta-, gamma-, or X-ray counter; or a formamide solution in a $2\pi\beta$ -proportional counter.

At the National Bureau of Standards the primary standardizations of the short-lived nuclides are maintained by means of a $2\pi\beta$ -ionization chamber in conjunction with radium D+E reference sources, and a $4\pi\gamma$ -ionization chamber in conjunction with reference standards of radium consisting of radium bromide solution in flame-sealed glass ampoules (Seliger and Schwebel, 1954). More recently both types have been replaced by radium reference sources, with the radium salt sealed in stainless steel. Primary standards of the short-lived nuclides sodium-24, phosphorus-32, potassium-42, iodine-131, and gold-198 are calibrated by $4\pi\beta$ or coincidence counting. These primary standards are then used to effect a calibration of the appropriate ionization chamber in

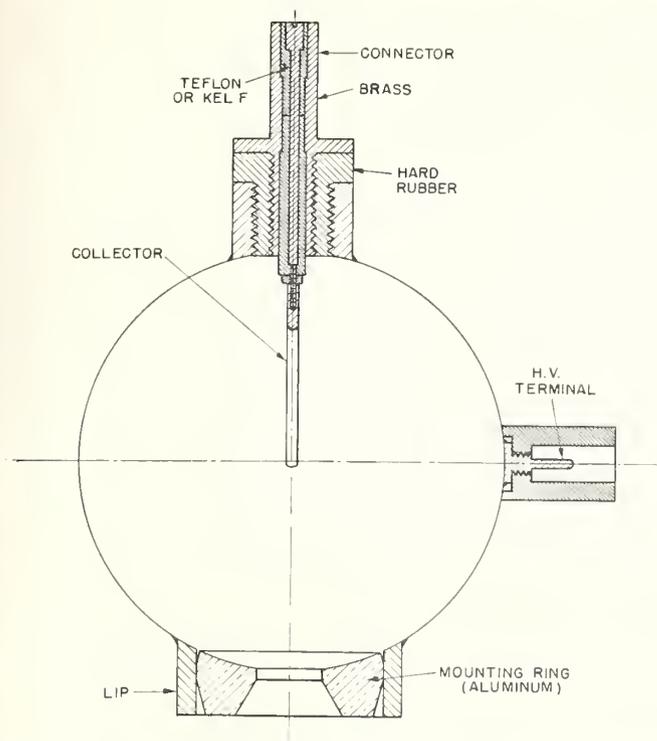


FIGURE 22. Schematic drawing showing the construction details of the NBS $2\pi\beta$ -ionization chamber (Seliger and Schwebel, 1954).

The chamber proper is made from a flagpole ball graciously donated by Dr. S. Feitelberg of Mt. Sinai Hospital, New York, N. Y. The body is a gold-plated sphere approximately 6 in. in diameter. At the base is a cylindrical shoulder which slides smoothly over the source-mounting ring. The center of the source-mounting ring is recessed to hold reproducibly a 1-in. diameter disk on which an aliquot of the active solution is evaporated. The length of the $\frac{1}{8}$ -in. diameter brass-collecting electrode has been adjusted empirically so that a point source of beta rays can be displaced slightly from the center of the source mount without affecting the observed ionization current. This ensures that no error will be introduced into the secondary standardization due to off-center drying of solutions delivered to the disks. Current measurements are made with a Lindemann-Ryerson electrometer in a Townsend rate-of-charge null method. The chamber is mounted together with the Lindemann-Ryerson electrometer and a compensating condenser in such a manner that the entire assembly can be raised several inches above the aluminum mounting ring, into which the sources are placed for measurement. Collecting voltages are applied to the chamber so that the collector is always very close to ground potential during a reading.

conjunction with a radium reference source. Further standards of these artificially produced nuclides are then issued as secondary standards by calibration against the reference sources in these chambers (figs. 22, 23, and 24).

A cavity ionization chamber has also been used to determine, to within ± 5 percent, the disintegration rate of a beta or gamma emitter (Gray, 1949), the former being incorporated in a gelatin lining to the chamber and the latter being in the form of an external source. The gamma rays from radium in equilibrium with its products were used to calibrate the chamber. The relative stopping powers of the wall and air, and the energy loss per ion pair formed in air for the gamma rays of the radium in equilibrium with its daughters and for the beta or gamma rays of the nuclide in question, can then be used to calculate its rate of disintegration (Gray, 1949; Robinson, 1954; Perry, 1954a). Any gamma emitter other than radium may be used to calibrate the ionization chamber, provided that the dose rate in air to which it

gives rise is known in terms of its disintegration rate.

In this connection it is interesting to note that in addition to using counting methods to standardize such gamma emitters as sodium-24, potassium-42, iron-59, cobalt-60, and bromine-82, some laboratories in the United Kingdom have also maintained standards of such nuclides by determining the roentgens per hour at 1 cm from samples standardized by counting methods. This was partially to accommodate medical and therapeutic needs. As a result, the British standard of radioactive iodine-131 is a standard that has been defined by measurement of its gamma-ray effect, in terms of roentgens per hour at 1 cm, using a graphite-wall ionization chamber that had been calibrated with radium (Bullard, 1953), assuming a conversion factor of 8.3 roentgens per hour per milligram at 1 cm. However, in order to derive the millicurie content of the iodine-131, it is necessary to adopt an arbitrary conversion factor to correlate the response of the chamber with absolute counting measurements. This factor is 2.25 roentgens per hour in air at 1 cm from 1 mc of iodine-131, and compares favorably with the factor 2.29 deduced from recent determinations of the disintegration scheme (Cavanagh, 1952; Bell and Graham, 1952a and 1952b) if the average energy lost in producing one ion pair in air is assumed to be 32.5 ev and the conversion factor for radium is assumed as above. However, recent measurements (Jesse and Sadaukis, 1957; Gross, Wingate, and Failla, 1957; Bay, Mann, Seliger, and Wyckoff, 1957) have shown the energy per ion pair in air to be around 33.7 ev. The use of this value would reduce the factor of 2.29 to 2.21 but leave the agreement with experiment unchanged. None of these assumptions affects the value of the standard in millicuries, which is ultimately based on counting methods, once the arbitrary conversion factor has been adopted. It would therefore seem that this method, while preserving the primary standard, is capable only of producing secondary standards for future use. The dose-rate as measured in air will be unchanged by changes in other atomic and nuclear data, and is therefore the same for the clinician, but is relative to the corresponding figure for radium, so long as this is used to calibrate the graphite-walled chamber.

For low-energy beta emitters, such as hydrogen-3 or carbon-14, comparisons with the primary standards can be carried out by liquid scintillation counting (Raben and Bloembergen, 1951; Audric and Long, 1952 and 1953; Ziegler, Seliger, and Jaffe, 1956; Steyn, 1956; Seliger and Ziegler, 1957) (fig. 25). The method of formamide counting (Schwebel, Isbell, and Karabinos, 1951; Schwebel, Isbell, and Moyer, 1954) is particularly useful for the rapid comparison of the activities of aqueous solutions of salts of the radioactive nuclides. In this method up to 10 percent of an aqueous solu-

tion of radioactive material can be dissolved in the organic solvent, formamide, and this resulting solution can be introduced into a $2\pi\beta$ -proportional flow counter without in any way impairing the

electrical or counting properties of the counter. A great advantage of this method is that it completely eliminates problems arising from differences in source self-absorption.

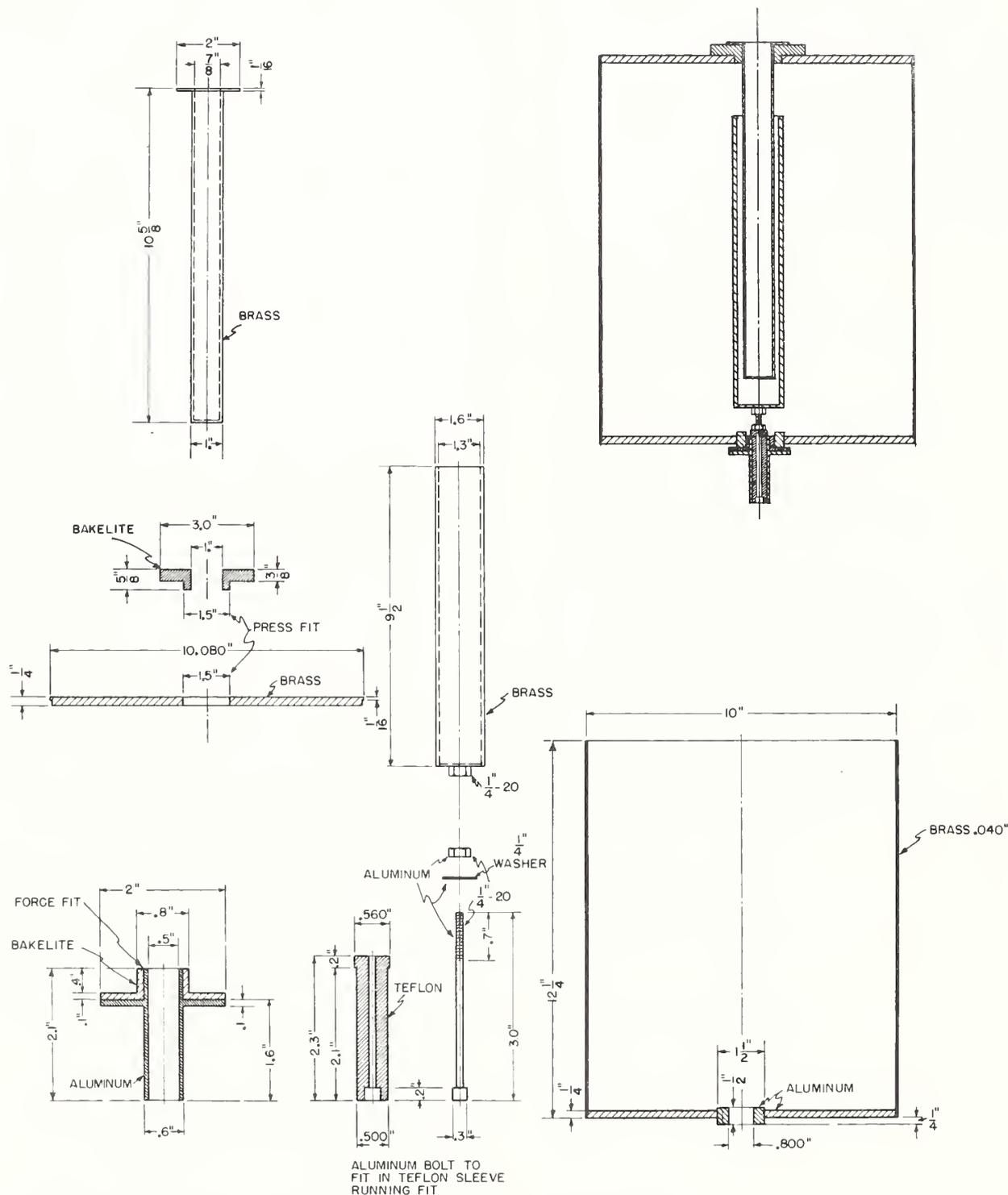


FIGURE 23. Schematic drawing showing the construction details of the NBS $4\pi\gamma$ -ionization chamber (Smith and Seliger, 1953).

The chamber is constructed of brass and aluminum and has a volume of approximately 15 liters. The reentrant cylinder is separated from the main body of the chamber and is grounded. Sources are inserted into the cylinder and supported at the geometrical center on a Lucite spacer. If a point source of gamma radiation is moved vertically through ± 4 cm about the geometrical center of the chamber, the response remains uniform to ± 1 percent. Thus it is possible to standardize solutions of different volumes provided self-absorption is negligible. High voltage is applied to the chamber so that the collector cylinder is always very close to ground potential during a reading. Current measurements are made with a Lindemann-Ryerson electrometer in a Townsend rate-of-charge null method.

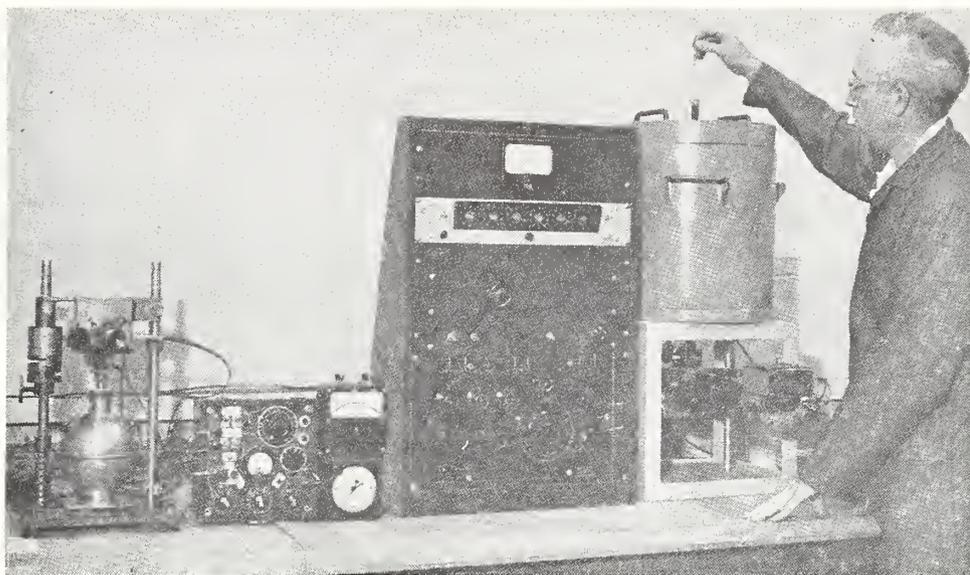


FIGURE 24. Photograph of the NBS $2\pi\beta$ - and $4\pi\gamma$ -ionization chambers (Garfinkel, 1958).

The $4\pi\gamma$ chamber is mounted inside a 1-in. thick lead shield to reduce the background which is less than 4×10^{-14} ampere. A source is being inserted into the $4\pi\gamma$ -ionization chamber. On the left are the manual controls for applying compensating charge and for measuring the timing interval. On the right is a semiautomatic timing and charge-compensating circuit. Diode phototubes, actuated by the projected shadow of the electroscope fiber, switch on and off a decade scaler and a motor-driven 10-turn precision potentiometer, respectively. The decade scaler is driven by the NBS standard 1,000-cycle frequency and the compensating voltage is read by means of the precision potentiometer.

3. Problems Arising in the Preparation and Distribution of Radioactivity Standards

In some countries where the use of radioactive materials is still relatively on a small scale, facilities exist for the standardizing of each consignment *en route* to the laboratory to which it is being consigned. In the United States and the United Kingdom this practice is no longer feasible, so that both the National Bureau of Standards and the National Physical Laboratory produce radioactivity standards for distribution to laboratories using radioactive materials for experimental purposes in order that these laboratories may standardize their own calibration equipment. The standardization of such equipment for any particular radioactive nuclide, having radiations unique to itself and therefore giving rise to a definite efficiency for measurement, is then preserved in terms of a long-lived radioactive substance such as radium. Incoming shipments of radioactive materials can then be routinely calibrated and, provided that there is no change in the characteristics of the calibrating equipment, only an occasional recheck is necessary against a radioactivity standard of the nuclide under consideration. From the point of view of permanence and consistency of operation, ionization methods can be seen to be superior to counting methods that depend on both the life of the counter, if it is of the sealed variety, and the maintenance of the auxiliary electronic equipment.

Most of the radioactivity standards of the National Bureau of Standards, as can be seen from the list of table 1, are solution standards to which sufficient carrier has been added to render negli-

gible the adsorption of the active material to the container walls. With five exceptions, these are standards of relatively long life that are prepared in batches of several hundred ampoules at a time and kept in stock for distribution as required (figs. 26, 27, and 28). The standards of the short-lived nuclides sodium-24, phosphorus-32, potassium-42, iodine-131, and gold-198 were, up to 1957, each distributed twice a year. Some 6 weeks before each distribution, notification cards were sent to a mailing list consisting of some 350 potential users in North America. Normally between 50 and 100 orders were received, and the distribution planned accordingly, an appropriate number of glass ampoules being filled with the same solution (prepared by suitable dilution from a master solution) and flame-sealed. Several of these ampoules, taken at random, were then calibrated (fig. 29) and the distribution made either by air express (sodium-24, potassium-42, and gold-198) or railway express (phosphorus-32 and iodine-131).

In May of 1957, however, standard solutions of these short-lived nuclides were made available commercially and the National Bureau of Standards accordingly withdrew from the field. It continues, however, to maintain the primary standards of these nuclides and to compare them with those of other national standardizing laboratories.

In view of the difficulties that had to be surmounted in the distribution of the short-lived radioactivity standards, there have been a number of

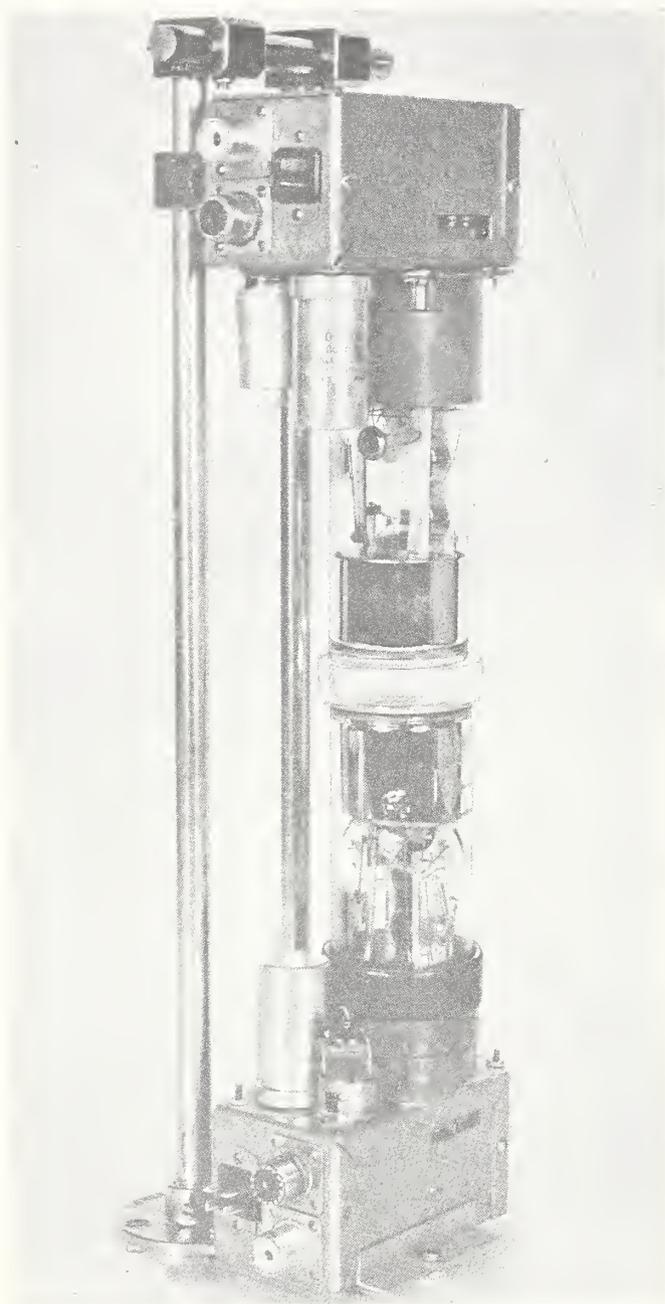


FIGURE 25. *Experimental arrangement for 4π -liquid-scintillation counting which is similar to that for 4π -sandwich-crystal counting shown in figure 21.*

attempts to provide substitute standards for these. Thus, in The Netherlands it has been shown that, using a beta counter with sodium-22, the absorption of the positrons, together with the gamma rays and annihilation radiation, in aluminum is essentially the same as that for the beta particles and gamma rays from iodine-131 up to about 90 mg/cm² of aluminum (Aten, 1954). The back-scattering from a thick layer of aluminum was practically the same for each nuclide. Thus, iodine-131 may be assayed in terms of a standardized sample of sodium-22. In the same way, thulium-170 has been shown to have the same absorption coefficient as gold-198 for beta counting, and potassium-40 is suggested as a good substitute



FIGURE 26. *A selection of radioactivity standards issued by the National Bureau of Standards.*

In the foreground is a radium D+E reference standard flanked by representative radioactivity solution standard samples. In the background are a number of radium rock ore standards and one uranium ore standard. The distribution of uranium and also of thorium ore standards has, however, been discontinued but these can currently be obtained from the Atomic Energy Commission's New Brunswick Laboratory.

standard, by beta counting, for sodium-24 (Aten, 1954). Recently a remarkably close gamma-ray analogue for iodine-131, whose principal gamma rays have energies of 335 and 608 keV, has been produced by mixing the correct proportions of barium-133 (82-, 300-, and 375-keV gamma rays) and cesium-137—barium-137 (662-keV gamma rays) (Brucer, 1955). When such a simulated standard is used to calibrate a true iodine-131 sample, its effect as measured by any kind of gamma-sensitive equipment will be approximately the same as for iodine-131. It may thus have uses for calibration to within say ± 3 to 5 percent when standards of short-lived iodine-131 itself do not happen to be available. A set of such "mock-iodine" standards consisting of two nominal 10-microcurie and two 50-microcurie ampoules was calibrated in the summer of 1955 at the National Bureau of Standards, the Max-Planck-Gesellschaft at Göttingen, and the National Physical Laboratory. The results, obtained by ionization chambers,¹ Geiger-Müller and scintillation counters at these three laboratories did not differ by more than ± 4 percent from the mean value.

At the National Physical Laboratory the problem of preserving short-lived radioactivity standardization outside the national standardizing laboratories is being approached by investigating the possibility of factory-producing a combined beta- and gamma-ionization chamber (Perry, Dale, and Pulfer, 1956). Four such combined chambers have already been tested at the National Physical Laboratory. It is hoped that when they are ultimately produced in quantity they will be so similar in construction that a separate calibration of each combined chamber for each nuclide will be unnecessary and that users of radioactive materials will always have available means for the calibration of individual nuclides to within ± 3 percent.

TABLE 1. Primary alpha, beta, and gamma radioactivity standards maintained by the National Bureau of Standards

Sample No.	Radiation	Nuclide	Nominal activity ^a	Volume	Chemical form of standard	Method of primary standardization	Method of secondary standardization
4900	α	Polonium-210 ^b	200 dps	(e)	Polonium plated from polonium-chloride solution.	2 $\pi\alpha$ -prop. counting	2 $\pi\alpha$ -prop. counting.
4901	α	Polonium-210 ^b	500 dps	(e)	do	do	Do.
4902	α	Polonium-210 ^b	1,000 dps	(e)	do	do	Do.
4903	α	U ₃ O ₈ ^d	15 dps	(e)	do	do	Do.
4910	$\beta(\alpha)$	RaD+E ^f	200 dps	(e)	Lead peroxide	Quantitative extraction from pitchblende.	Defined-geometry G.-M. counting.
4911	$\beta(\alpha)$	RaD+E ^f	500 dps	(e)	do	do	Do.
4912	$\beta(\alpha)$	RaD+E ^f	1,000 dps	(e)	do	do	Do.
4913	$\beta(\gamma)$	Cobalt-60	10 ⁴ dps/ml	(g)	Chloride in HCl solution	4 $\pi\beta$ -prop. counting; coinc. counting.	4 $\pi\gamma$ -ion. chamber; formamide counting.
4914	$\gamma(\beta)$	Cobalt-60	10 ⁵ dps	5.0 ml	do	do	Do.
4915	$\gamma(\beta)$	Cobalt-60	10 ⁶ dps	5.0 ml	do	do	Do.
4916	β	Phosphorus-32	10 ⁵ dps/ml	(g)	Phosphoric acid solution	4 $\pi\beta$ -prop. counting	2 $\pi\beta$ -ion. chamber; formamide counting.
4917	$\beta(\gamma)$	Iodine-131	10 ⁵ dps/ml	(g)	Sodium iodide solution	4 $\pi\beta$ -prop. counting; coinc. counting.	2 $\pi\beta$ -4 $\pi\gamma$ -ion. chambers; formamide counting.
4918	$\beta(\gamma)$	Gold-198	10 ⁵ dps/ml	(g)	Auric cyanide solution	do	Do.
4919	β	{Strontium-90 Yttrium-90}	10 ⁴ dps/ml	(g)	Chloride in HCl solution	4 $\pi\beta$ -prop. counting	{2 $\pi\beta$ -ion. chamber; formamide counting.
4920	β	Thallium-204	10 ⁴ dps/ml	(g)	Thallic nitrate in HNO ₃ solution.	do	Do.
4921	$\beta(\gamma)$	Sodium-22	10 ⁴ dps/ml	(g)	Chloride in HCl solution	4 $\pi\beta$ -prop. counting; coinc. counting; Co ⁶⁰ comparison.	4 $\pi\gamma$ -ion. chamber.
4922	$\gamma(\beta)$	Sodium-22	10 ⁶ dps	5.0 ml	do	do	Do.
4923	$\beta(\gamma)$	Sodium-24	10 ⁵ dps/ml	(g)	do	4 $\pi\beta$ -prop. counting; coinc. counting.	Do.
4924	β	Carbon-14	10 ³ dps/ml	25.0 ml	Sodium carbonate solution	Compensated gas counting	Formamide counting; gas ion. chamber; liq. scint. counting.
4925	β	Carbon-14 ^h	10 ⁴ dps/g	(e)	Benzoic acid in toluene	do	Do.
4926	β	Hydrogen-3	10 ⁴ dps/ml	25.0 ml	Tritiated water solution	Calorimeter; compensated gas counting.	Liq. scint. counting; gas ion. chamber.
4927	β	Hydrogen-3	10 ⁵ dps/ml	(g)	do	do	Do.
4928	β	Sulfur-35	10 ⁴ dps/ml	(g)	Sodium sulfate solution	Calorimeter; 4 $\pi\beta$ -prop. counting.	Liq. scint. counting; formamide counting.
4929	K	Iron-55	10 ⁵ dps/ml	(g,i)	Ferric chloride in HCl solution.	Liq. scint. counting	Liq. scint. counting; X-ray counting.
4930	K(γ)	Zinc-65	10 ⁵ dps/ml	(g)	Chloride in HCl solution	Liq. scint. counting; coinc. counting.	Do.
4931	$\gamma(\beta)$	{Cesium-137 Barium-137}	10 ⁴ dps	5.0 ml	do	4 $\pi\beta$ -prop. counting; coinc. counting.	4 $\pi\gamma$ -ion. chamber.
4932	$\gamma(\beta)$	Mercury-203	10 ⁶ dps	5.0 ml ⁱ	Mercurous nitrate solution	do	Do.
4933	$\gamma(\beta)$	Potassium-42	10 ⁵ dps/ml	(g)	Chloride in HCl solution	do	Do.
4934	$\gamma(\beta)$	Tantalum-182	10 ⁵ dps/ml	(g)	Fluoride in HCl solution	4 $\pi\beta$ -prop. counting	Do.
4935	β	Krypton-85	10 ⁴ dps	(i)	Gaseous krypton	Compensated gas counting	Gas ion. chamber; gas counting.

^a The disintegration rate as of the reference data is given on a certificate accompanying the standard.

^b Samples consist of a practically weightless deposit of polonium-210 on a silver disk 1 in. in diameter, 1/16 in. thick, and faced with 0.002 in. of palladium.

^c Deposited source.

^d Samples consist of U₃O₈ deposited on a 0.1-mm-thick platinum foil and mounted on an aluminum disk, 1 1/4 in. in diameter and 1/32 in. thick. The alpha-ray disintegration rate as of the date of calibration and in the forward hemisphere is indicated on the certificate accompanying the standard.

^e Evaporated source.

^f Standards consist of Pb-210 and Bi-210 in equilibrium, deposited on a silver disk 1 in. in diameter, 1/16 in. thick, and faced with 0.002 in. of palladium.

^g Approximately 3 ml of low-solids carrier solution containing the active nuclide in a flame-sealed ampoule.

^h Benzoic acid in toluene for use in liquid scintillation counters.

ⁱ In preparation.

^j A approximately 10 ml of krypton at STP in a break-seal ampoule.

Radium standards (for radon analysis)

Sample No.	Radium content (in grams)	Volume (in ml)
4950	10 ⁻⁹	100
4951	10 ⁻¹¹	100
4952	Blank solution	100

Samples are sealed in glass containers. They were prepared by determining the radium content of a purified sample of radium chloride by direct comparison with the U. S. Primary Radium Standards by means of the gold-leaf electroscop and radiation balance (Marin, Stockmann, Youden, Schwebel, Mullen, and Garfinkel, 1958). This radium chloride was then transferred quantitatively to a carrier solution consisting of 0.2 percent by weight BaCl₂·2H₂O in a 5 percent by weight solution of HCl.

Radium gamma-ray standards

Sample No.	Radium content (in grams)	Volume (in ml)
4955	0.1 × 10 ⁻⁶	5
4956	0.2	5
4957	0.5	5
4958	1.0	5
4959	2.0	5
4960	5.0	5
4961	10	5
4962	20	5
4963	50	5
4964	100	5

Samples are contained in flame-sealed glass ampoules. They were prepared by determining the radium content of a purified sample of radium bromide by direct comparison with the U. S. Primary Radium Standards by means of the gold-leaf electroscop. The radium bromide was then dissolved quantitatively in a 5 percent by weight solution of HNO₃.

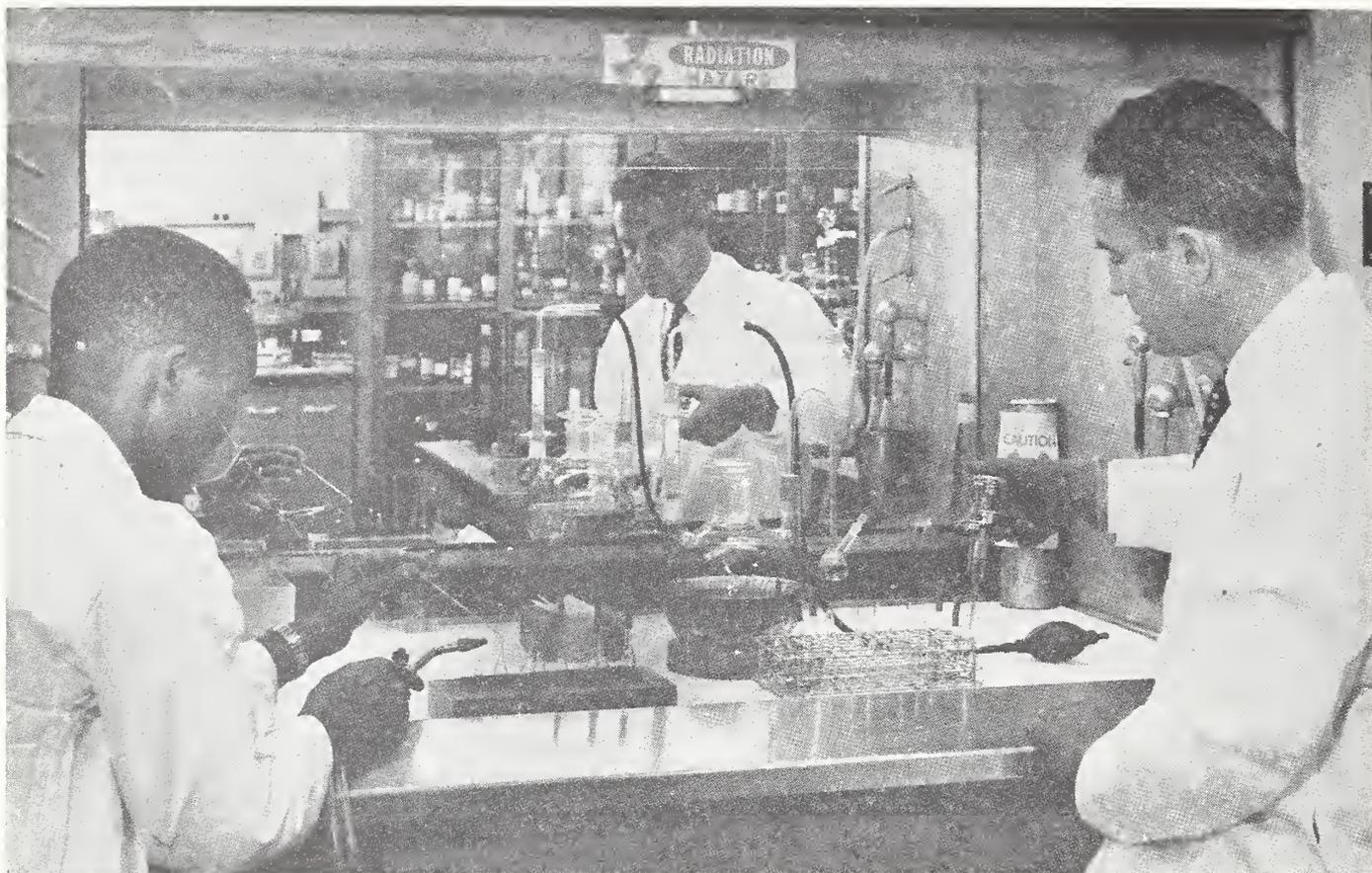


FIGURE 27. NBS Radiochemical Laboratory of the Radioactivity Section showing the experimental arrangement adopted for the dispensing of the carbon-14-labeled benzoic-acid-in-toluene standards for liquidscintillation counting.

The master solution is shown in the triple-necked flask from which it is transferred by air pressure to the modified 25-ml volumetric flask on the right. From this latter, 3-ml aliquets are transferred to the glass ampoules by means of a spring-loaded hypodermic syringe. The ampoules are then placed in a dry-ice-alcohol bath and are flame-sealed, the toluene having been cooled to a point where its vapor pressure is less than 1 mm of mercury.

TABLE 1. Radium rock standards—Continued

Sample No.	Rock	Average radium content (gram of radium per gram of rock)
4975	Dunite.....	$0.009 \pm 0.004 \times 10^{-12}$
4976	Carthage limestone.....	0.15 ± 0.03
4977	Berea sandstone.....	0.24 ± 0.02
4978	Columbia River basalt.....	0.33 ± 0.03
4979	Chelmsford granite.....	2.96 ± 0.08
4980	Quartzite.....	0.06 ± 0.01
4981	Graniteville granite.....	3.3 ± 0.2
4982	Gabro-diorite.....	0.18 ± 0.02
4983	Milford granite.....	0.23 ± 0.02
4984	Triassic liabase.....	0.18 ± 0.03
4985	Deccan trap.....	0.21 ± 0.04
4986	Kimberlite.....	0.59 ± 0.04

Each sample consists of 100 g of pulverized rock taken from bulk material analyzed for radium content. Petrographic data and approximate chemical analysis of a representative sample of each rock also given in a certificate accompanying each sample.

Contemporary standard for carbon-14-dating laboratories

Sample No.	Description	Quantity
4990	Oxalic acid.....	5 lb.



FIGURE 28. Radioactivity standards are stored in an outside brick room on shelves with brick and concrete lips having a thickness of 8 inches.

In this photograph carbon-14 and radium-226 standards can be seen reflected in the mirror at the back of such a shelf. The radium-226 ampoules are packaged in plastic containers with absorbent cotton.

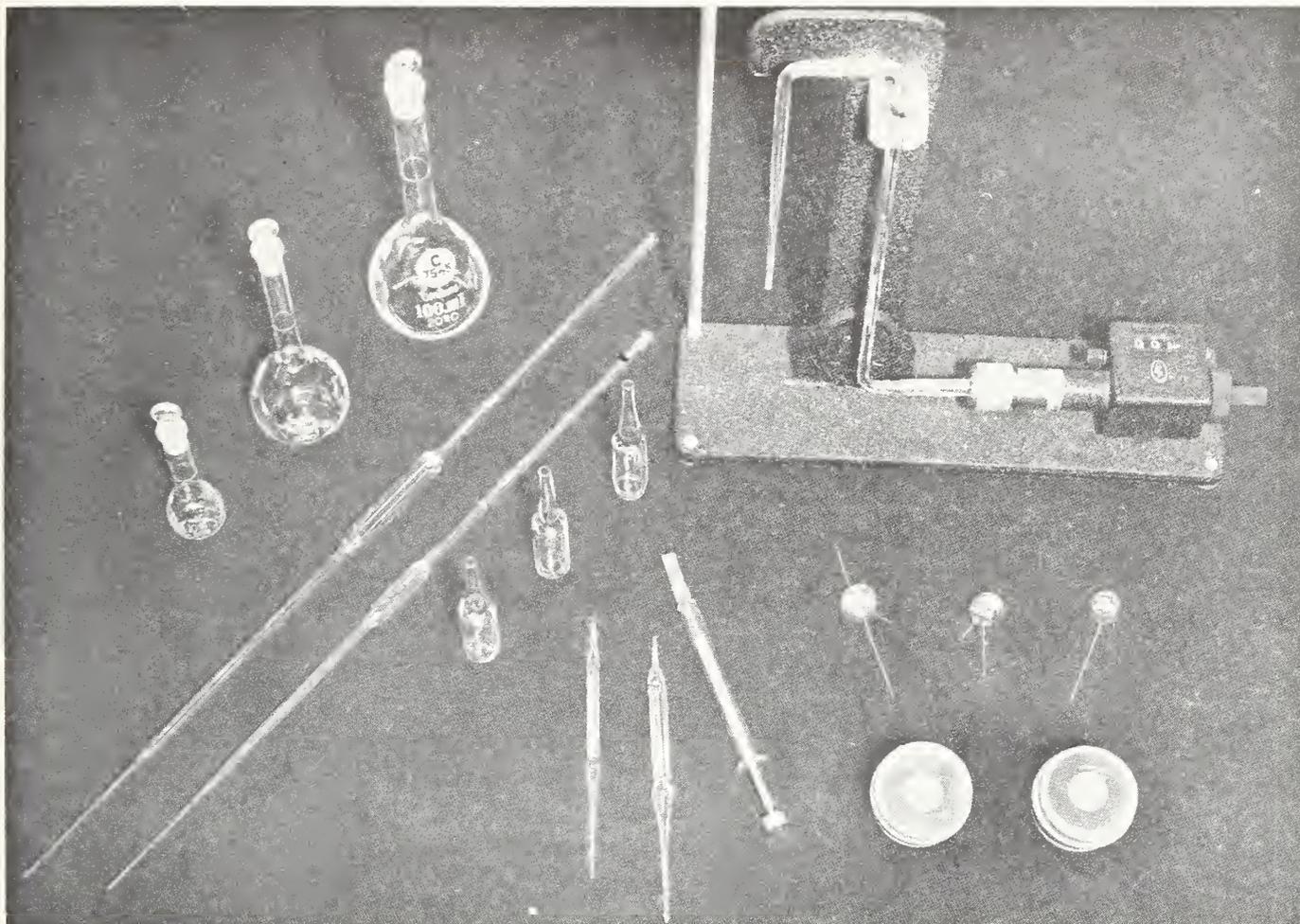


FIGURE 29. Apparatus used for preparation of sources for $4\pi\beta$ counting.

In the right rear is a mercury-displacement type ultramicroburet capable of delivering 0.1 ml in 1,000 divisions. The displacement of an accurately-machined stainless steel plunger is read directly on a micrometer dial. In the right center are extremely light pycnometers also used for delivering aliquots of solutions to $4\pi\beta$ -counter source mounts in the foreground. The fine wire by which the pycnometer is suspended from the balance arm can be seen attached to the one farthest to the left. The pycnometer is filled by first heating the body with the tip immersed in the active solution. A small soldering-iron tip, brought near the body while it is suspended from the balance arm can control the delivery of single droplets. The long capillary neck permits the pycnometer to be used for hours with negligible change of weight due to evaporation.

4. Use of Radioactivity Standards in the Laboratory

Determinations of the number of alpha particles emitted by radium per second per gram were first made by Rutherford and Geiger (1908), using their newly developed spark counter (fig. 30), and by Sir James Dewar (1910), who measured the rate of production of helium from about 70 mg of pure radium chloride. In the same year (1908), Regener attempted to detect alpha particles quantitatively by counting the scintillations produced in zinc sulfide and using the emission constant determined by Rutherford and Geiger, and by Dewar. The determination of the efficiency of the zinc-sulfide scintillation counter for alpha particles can therefore be considered to be the first direct application of a standard of radioactivity.

Also in 1908, Soddy (1908) measured the production of helium in purified uranium, leading to a value for the half-life of the latter element. This measurement had a direct application in answering one of the oldest questions of mankind, namely, how old is the earth? Radioactive mate-

rials and particularly uranium provided a process wherein the rate of progress of the radioactive decay was independent of external physical or chemical conditions and in which the element was sufficiently long lived so as not to have decayed appreciably during the entire process.

In 1912, Mosely (1912), using radium B+C, determined that within his experimental error there was only one electron emitted per beta disintegration. This result, while not a standardization *per se*, forms an essential link in the interpretation of the subsequent results of Ellis and Wooster (1927), who determined the mean energy per radium E beta disintegration. To do this they measured in a sensitive calorimeter the total rate of energy emission of a separated radium E sample and its subsequent polonium-210 daughter. From this they determined the ratio of the rate of energy emission of radium E to polonium-210, using the known decay constants. They also assumed the energy of the polonium-210 alpha particles to be 5.22 Mev.

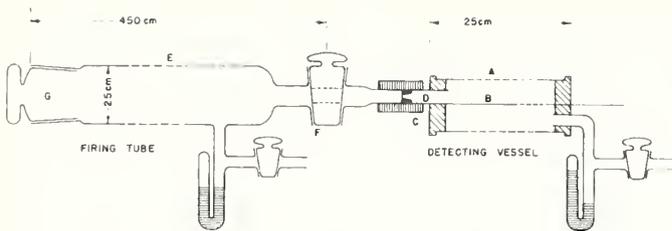


FIGURE 30. A schematic drawing of the original Geiger-Rutherford alpha-particle counter in which for the first time, in 1912, it was shown to be practicable to detect a single particle by means of its electrical effect.

A source of alpha particles on a thin film was placed in the large tube E which was then evacuated. When the stopcock F was opened a small fraction of the alpha particles entered the aperture into the counter which contained air at a few centimeters of mercury pressure. If the voltage was adjusted properly, the ionization by the alpha particle in the vicinity of the wire B caused an electrical breakdown of the air which was detected by means of an electrometer connected to ground through a high resistance.

From the primary applications of standards of radioactivity referred to above, namely (a) determination of absolute efficiencies of detectors, (b) determination of long half-lives, and (c) determination of mean energies, there have been derived today a number of related applications. There are measurements of absolute cross sections, activation analyses, and activity measurements for medical, industrial, and military uses and uranium prospecting, all based on (a); measurements of historical and geologic dates, based on (b); and measurements of W (the energy expended per ion pair), electron penetration, and depth-dose-rate for medical applications, based on (c). The use of the half-life of carbon-14 for historical dating has become an archaeological tool of extremely high resolving power, the latest and most important contribution being the accurate dating of the Dead Sea Scrolls.

It is important to distinguish between the applications of *radioactivity* and the applications of *standards of radioactivity*. Any report encompassing the former would be monumental, and would only mostly describe the same kind of precise relative measurements. However, even in these cases, for radiation safety, for planning experimental details, and for ordering and disposing of active material, a knowledge of the disintegration rates of the material is essential. The experimenter must account for all of the initial radioactivity in order to obtain a material balance in his experimental results. Thus the total efficiencies under differing experimental conditions of his various radiation detectors must be known. In the following a summary of the applications of

5. Methods of Measurement

Table 3 lists the most general methods available for the measurement of alpha, beta, and gamma emitters, together with a brief description of the counting conditions and the range of applicability of each method. In this connection the term Background Equivalent Activity (BEA) has been found to be a convenient method of expressing the intrinsic sensitivity of a detector (Seliger and Schwebel, 1954). In all measurements of radio-

standards of radioactivity will be presented. A large part will be devoted to suggested methods for the use of radioactivity standards for calibration purposes, and the various methods of measurement of radioactivity will be discussed. The remainder of the sections will be concerned with long-half-life measurements, historical and geologic dating, and measurements based on the mean energy of beta disintegration. The literature references are by no means exhaustive but are representative of the applications of standards of radioactivity.

The *total efficiency*, ϵ , of a detector for a source prepared from a radioactivity standard is defined as:

$$\epsilon = \frac{\text{counts per second observed for source}}{\text{disintegrations per second in source}} \quad (1)$$

The total efficiency is compounded of the *geometry* (i. e., the fractional solid angle subtended by the detector) and the *detector efficiency* (i. e., the probability of a measurable response from a radiation striking the detector).

Beta-ray solution standards distributed by the National Bureau of Standards are contained in flame-sealed glass ampoules and consist of approximately 3 ml of an aqueous carrier solution of the active nuclide. A certificate specifies the disintegration rate per second per milliliter as of the zero date. Ampoules intended for use as gamma-ray standards contain an accurately known volume of solution (5 ml), and in these cases the total disintegration rate and the exact volume of solution at a given temperature are also specified. Radium D+E beta-ray reference sources, radium F (polonium-210) alpha-ray standards, and uranium oxide (U_3O_8) alpha-ray standards consist of the active material already deposited on source mounts.

In table 2 are listed most of the important radionuclides, their decay properties, and the preferred methods to be used in the laboratory for the routine assay of samples. Nuclides now standardized or shortly to be standardized at the National Bureau of Standards are printed in *italic* type. The beta-ray energies available cover the range from the 18.9-keV spectrum of hydrogen-3 to the 3.58-MeV spectrum of potassium-42. Gamma-ray energies cover the range from the 87-keV line of cadmium-109 to the 2.758-MeV line of sodium-24, as well as the X-rays from the electron capturers such as iron-55 and zinc-65.

activity there is a background effect without the source which must be subtracted from the observed reading taken with the source present. The magnitude of this background, or more specifically the ratio of the source effect to background effect, may place practical time limitations upon the conduct of an experiment. It is for this reason that the term Background Equivalent Activity (BEA) was devised, this being the disintegration rate of the

TABLE 2. Preferred methods of measurement for selected radionuclides

Nuclide	$E_{\beta_{max}}$ (Mev)	Principal E_{γ} (Mev)	$T_{1/2}$	Suggested methods for routine assay ^a
H^3	0. 0189	-----	12. 3 y	Liquid scintillation counter; internal gas counter; internal gas ion chamber.
C^{14}	0. 155	-----	5600 y	$2\pi\beta$ windowless counter; liquid scintillation counter; internal gas counter; internal gas ion. chamber.
Na^{22}	0. 542	{ 0. 511 1. 28	2. 565 y	$2\pi\beta$ windowless counter; well-type scintillation counter.
Na^{24}	1. 39	{ 2. 758 1. 380	14. 97 h	Do.
P^{32}	1. 71	-----	14. 3 d	$2\pi\beta$ windowless counter; well-type scintillation counter (bremsstrahlung).
S^{38}	0. 167	-----	87. 1 d	$2\pi\beta$ windowless counter; liquid scintillation counter.
Cl^{36} A^{37}	0. 713 EC	----- $ClK\alpha$	4×10^4 y 35. 0 d	Do. Internal gas counter; internal gas ion. chambers.
K^{40}	1. 33 EC	1. 45, $A K\alpha$	1.3×10^6 y	$2\pi\beta$ windowless counter; well-type scintillation counter.
K^{42}	{ 2. 04 3. 58	1. 51	12. 44 h	Do.
Ca^{48}	0. 254	-----	152 d	$2\pi\beta$ windowless counter; liquid scintillation counter.
Sc^{46}	{ 0. 36 1. 2 0.5%	{ 1. 12, 0. 89, 0. 89, 0. 32	85 d	$2\pi\beta$ windowless counter; well-type scintillation counter.
Cr^{51}	EC	{ $V K\alpha$	27. 8 d	X-ray counter; well-type scintillation counter; liquid scintillation counter.
Mn^{54}	EC	0. 84 $Cr K\alpha$	291 d	Do.
Fe^{55}	EC	$Mn K\alpha$	2. 94 y	Do.
Fe^{59}	{ 0. 260 0. 460	{ 1. 3 1. 1	45. 1 d	$2\pi\beta$ windowless counter; well-type scintillation counter.
Co^{60}	0. 310	{ 1. 17 1. 33	5. 26 y	Do.
Ni^{60}	EC	$Co K\alpha$	8×10^4 y	X-ray counter; well-type scintillation counter.
Ni^{63}	0. 067	-----	85 y	$2\pi\beta$ windowless counter; liquid scintillation counter.
Zn^{68}	{ EC 0. 325 1. 5%	1. 11, $Cu K\alpha$ 0. 511	240 d	X-ray counter; well-type scintillation counter.
Ga^{67}	EC	{ Complex $Zn K\alpha$	77. 9 h	Do.
As^{74}	{ β^- 1. 36, 0. 69 β^+ 1. 53, 0. 92	0. 596	17. 5 d	$2\pi\beta$ windowless counter; well-type scintillation counter.
Br^{82}	0. 465	0. 547, 0. 787 Complex	35. 9 h	Do.
Kr^{85}	0. 695	0. 54 0. 6%	10. 2 y	Well-type scintillation counter; gas counter; internal gas ion. chamber.
Rb^{86}	{ 0. 716 1. 822	1. 081	19. 5 d	$2\pi\beta$ windowless counter; well-type scintillation counter.
Rb^{87}	0. 130	0. 034	6×10^{10} y	Do.
Sr^{88}	EC	0. 51	65 d	X-ray counter; well-type scintillation counter.
Sr^{90}	1. 46	-----	53 d	$2\pi\beta$ windowless counter; well-type scintillation counter (bremsstrahlung).
{ Sr^{90} Y^{90}	0 61	-----	19. 9 y	Do.
	2. 180	-----	61 h	Do.
Zr^{98}	{ 0. 371 0 84 isomer	0. 721	65 d	$2\pi\beta$ windowless counter; well-type scintillation counter.
Nb^{98}	0. 160	0. 745	35 d	Do.
Tc^{99}	0. 290	-----	2×10^6 y	Do.
Ru^{103}	{ 0. 217 0. 698	0. 498	39. 8 d	Do.
Ru^{106}	0. 039	0. 52 0. 62	1 y	$2\pi\beta$ windowless counter; liquid scintillation counter.
Ag^{110}	Complex	Complex	270 d	$2\pi\beta$ windowless counter; well-type scintillation counter.
Cd^{109}	EC	0. 087, $Ag K\alpha$	470 d	X-ray counter; well-type scintillation counter.
Sn^{113}	EC	$In K\alpha$	112 d	Do.
I^{128}	{ 0. 850 1. 27	0. 382	13 d	$2\pi\beta$ windowless counter; well-type scintillation counter.
I^{121}	{ 0. 255 0 600	0. 080 0. 364 0. 628	8. 08 d	Do.
Cs^{134}	{ 0. 090 0. 648	0. 602 0. 794	2. 3 y	Do.
{ Cs^{137} Ba^{137}	{ 0. 510 1. 20	-----	30 y 2. 6 m	Do. Do.
Ba^{133}	EC	0. 085, $Cs K\alpha$	10 y	X-ray counter; well-type scintillation counter.
{ Ce^{141} Ce^{144}	{ 0. 442 0. 581	0. 141	33. 1 d	$2\pi\beta$ windowless counter; well-type scintillation counter.
	0. 300, 0. 170	Complex	282 d	Do.
Pr^{144}	3. 0	1%	17 m	Do.

TABLE 2. Preferred methods of measurement for selected radionuclides—Continued

Nuclide	$E_{\beta, \max}$ (Mev)	Principal E_{γ} (Mev)	$T_{1/2}$	Suggested methods for routine assay ^a
Pm ¹⁴³	EC	0.067, Nd $K\alpha$	200 d	X-ray counter; well-type scintillation counter.
Eu ¹⁵⁴	0.300	0.778	16 y	2 $\pi\beta$ windowless counter; well-type scintillation counter.
	0.700	1.2		
	1.900	0.336		
Tm ¹⁷⁰	0.866	-----	129 d	Do.
	0.970			
Ta ¹⁸²	0.250	Complex	115 d	Do.
	0.530			
Ir ¹⁹²	0.670	Complex	74.4 d	Do.
Au ¹⁹⁸	0.970	0.411	2.698 d	Do.
Hg ²⁰³	0.208	0.279	47.9 d	Do.
Tl ²⁰⁴	0.783	-----	4.0 y	2 $\pi\beta$ windowless counter.
Pb ²¹⁰	0.026	0.007	22 y ?	2 $\pi\beta$ windowless counter; liquid scintillation counter.
Bi ²¹⁰	1.17	-----	5.0 d	2 $\pi\beta$ windowless counter; well-type scintillation counter (bremsstrahlung).
Po ²¹⁰	α 5.3	0.773 10 ⁻³ %	138 40 d	2 $\pi\beta$ windowless counter; ZnS α scintillation counter.
Ra ²²⁶	α 4.8	Complex	1620 y	4 $\pi\gamma$ ionization chamber; well-type scintillation counter.

^a The methods suggested are the most efficient. This does not preclude the use of the quartz-fiber electroscop or the stand and G-M counter for routine measurements.

particular isotope required to equal the background reading.

If we let A be the observed sample-plus-background counts observed in a given time and let B equal the background counts observed with no sample present, then the standard deviation of the net counting rate $A-B$ is given by

$$\sigma = (A+B)^{1/2}, \quad (2)$$

and the relative error of the net counting rate is

$$\frac{\sigma}{(A-B)} = \frac{(A+B)^{1/2}}{A-B}. \quad (3)$$

Thus the relative error of a measurement of a BEA is always $171/\sqrt{B}$ percent where \sqrt{B} is the standard deviation of the background count.

The 2 $\pi\beta$ windowless-gas-flow proportional counter is considered to be the most sensitive and versatile apparatus for the routine counting of all types of beta sources, followed closely by the 2 $\pi\beta$ -windowless electroscop (fig. 31). The well-type NaI (Tl) scintillation counter has the highest sensitivity for gamma rays of any detector now available (Baskin, Demorest, and Sandhaus, 1954; Haigh, 1954). With these instruments one can carry out almost any type of measurement involving radioactive materials. Where cost and trouble-free maintenance are overriding factors, the electroscop and the end-window G-M counter are efficient substitutes for the windowless flow counter, and a cylindrical arrangement of several gamma-ray G-M counters surrounding the source

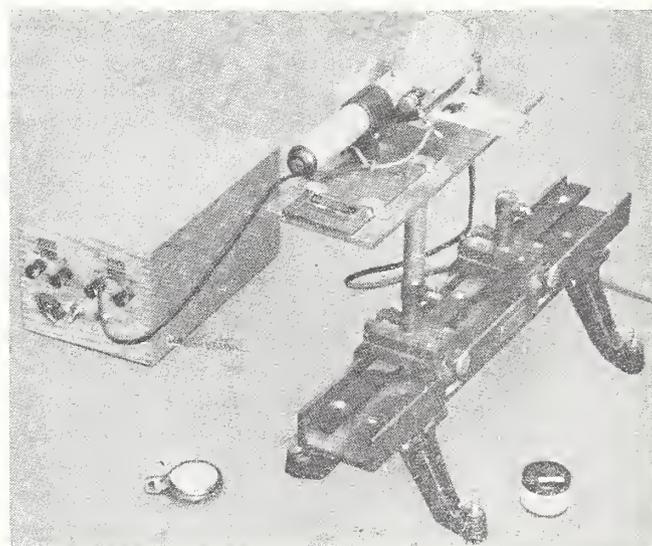


FIGURE 31. Photograph of 2 $\pi\beta$ windowless electroscop used for secondary standardization and for general relative counting.

The instrument is an adaptation of the Lauritsen electroscop as described by Hunter and Mann (1948). Inside the partly inserted sliding shelf is an NBS radium D+E reference source. If the same region of the scale is traversed for each timing interval such a high degree of precision can be obtained with a relatively simple and inexpensive instrument.

can substitute for the well-type scintillation counter. For these substitute methods the BEA is, of course, increased, especially so for the gamma-ray arrangement.

The liquid scintillation counter has recently come into limited use for the routine measurement of low-energy beta emitters. At the present time, under proper experimental conditions, it is possible to prepare a liquid scintillator which is more

TABLE 3. General methods of measurement for alpha, beta, and gamma emitters

Detector	Auxiliary equipment required ^a	Types of sources	Range of applicability and BEA ^b
1. $2\pi\beta$ -air-ionization chamber (recommended for secondary standardization at a large installation).	High-voltage supply, Vibrating-reed electrometer, or Lindemann - Ryerson electrometer Projector Calibrated condensers Voltmeter Stopwatch	Dried on planchet; liquid in glass or metal cup forming part of periphery of chamber.	Sensitive mainly to alpha and beta rays. Higher intensity gamma-ray sources can also be measured. Small self-absorption effects for low-energy beta emitters. BEA for P^{32} dried sources—0.001 μ c. BEA for P^{32} liquid sources—0.01 μ c.
2. $4\pi\gamma$ -air-ionization chamber (recommended for secondary standardization at a large installation).	High-voltage supply, Vibrating-reed electrometer, or Lindemann - Ryerson electrometer Projector Calibrated condensers Voltmeter Stopwatch	Liquid in ampoule; test-tube inserted into reentrant cylinder.	Beta rays are completely absorbed within the reentrant cylinder. Ionization is due to bremsstrahlung and to gamma rays. Big advantage is nondependence of reading on position or volume of source within the chamber. Can be used to compare high-energy beta emitters from bremsstrahlung measurements. BEA for I^{131} in solution—1.4 μ c. BEA for P^{32} in solution—20 μ c. High-pressure chambers are more sensitive.
3. $2\pi\beta$ - windowless-proportional flow counter (recommended for all beta-ray measurements on a routine basis).	High-voltage supply Non-overloading amplifier Discriminator Scaler 90% argon, 10% methane or helium isobutane, or 100% methane.	Dried on planchet; liquid in formamide or other low-vapor-pressure solvent, delivered to stainless-steel cup.	Most sensitive method for beta rays. Higher-intensity gamma-ray sources can also be measured. The efficiency for beta rays is practically 100% over the central region, becoming 90% at periphery. Simple to operate and to decontaminate. Alpha particles can be counted in presence of betas at lower voltages in the proportional region. BEA for P^{32} dried source—0.0001 μ c. BEA for P^{32} as thick source—0.001 μ c. BEA for C^{14} as liquid—0.001 μ c.
4. Well-type NaI scintillation counter (recommended for all gamma-ray measurements on a routine basis).	Precision high-voltage supply Nonoverloading linear amplifier Discriminator Multiplier Phototube Scaler Single-channel pulse-height analyzer	Liquid in ampoule; test-tube inserted into well. Source could also be external in conditions of "good" geometry.	Beta rays are completely absorbed in walls of crystal holder. Light output to phototube is due to bremsstrahlung and to gamma rays. Most sensitive method for gamma rays. Tremendous increase in sensitivity over air-ionization chamber. Light output is directly proportional to energy absorbed—permits identification of isotopes by gamma photopeaks. Window pulse-height analyzer permits counting one photopeak to exclusion of others in mixtures of nuclides. Relatively independent of source position or size inside well. BEA for I^{131} in 5-ml ampoule—0.0003 μ c. BEA for Cr^{51} in 5-ml ampoule—0.003 μ c. BEA for P^{32} in 5 ml ampoule—0.02 μ c.
5. Liquid scintillation counter (recommended for double beta-labeling experiments. Useful but not essential for low-energy beta emitters and electron capturers).	Precision high-voltage supply Non-overloading linear amplifier Discriminator Multiplier phototube Scaler Single-channel pulse-height analyzer	Liquid incorporated into solution of scintillator as water - alcohol - toluene, water-dioxane, or an organic tagged molecule such as C^{14} -benzoic acid.	Useful for measurement of low-energy beta-emitters such as H^3 , Ni^{63} and C^{14} and electron-capturing nuclides such as Cr^{51} , Fe^{65} , Ni^{58} —eliminates self-absorption effects. Useful for double- and triple-labeling experiments because of linear response to energy absorbed. Can discriminate between α 's and β 's. BEA for H^3O in 5-ml cell—0.01 μ c. BEA for C^{14} benzoic acid in 5-ml cell—0.004 μ c.
6. Mica end-window G-M counter (lower-priced substitute for $2\pi\beta$ -windowless counter).	High-voltage supply Scaler	Dried on planchet; liquid in glass or metal cup directly below window.	Almost as sensitive to betas as $2\pi\beta$ flow counter. 100% sensitive to electrons entering sensitive volume. Air and window absorption lower efficiency for low-energy beta emitters. Does not discriminate between α 's and β 's as in 2π flow counter. Efficiency for gamma rays same as $2\pi\beta$ -flow counter (1-2%). Halogen self-quenching tube gives long life, trouble-free service. Standard shelf arrangement gives reproducible geometry. BEA for P^{32} dried source in top shelf position—0.0001 μ c.

TABLE 3. General methods of measurement for alpha, beta, and gamma emitters—Continued

Detector	Auxiliary equipment required ^a	Types of sources	Range of applicability and BEA ^b
7. Thin-window argon, krypton, or xenon X-ray counter (specifically for use with electron-capturing nuclides).	High-voltage supply Linear amplifier Discriminator Scaler Single-channel pulse-height analyser	Dried on planchet; liquid in glass or metal cup directly below window.	Makes use of critical or near-critical absorption of gas for X-rays near critical absorption limit. Useful for pure electron-capturing nuclides. Window-absorption reduces efficiency for low-energy X-rays. Linear response makes <i>identification</i> and <i>differential</i> counting of mixtures of gamma-emitting nuclides possible. BEA for Fe ⁵⁵ dried source—0.0003 μ c.
8. Quartz-fiber electroscop (lower priced substitute for $2\pi\beta$ windowless counter).	Battery for charging Battery for pilot light Stopwatch	Dried on planchet, on slide mounted external or internal to electroscop. Internal liquid sources not recommended due to difficulty in decontamination.	Very-low-capacity system slightly more sensitive to internal beta sources than $2\pi\beta$ -air-ionization chamber. Low sensitivity to gamma rays. Displacement of fiber is slightly nonlinear. Excellent for relative measurements over same region of scale. Simple, trouble-free operation with care. Can be used for low-energy beta emitters with same precautions as $2\pi\beta$ -air-ionization chamber and for higher intensity external gamma sources. BEA for P ³² dried source—0.001 μ c.
9. Internal-gas-G-M counter.....	High-voltage supply Scaler Chemical conversion system	Nuclide converted to inert or counting gas; small amount present in vapor phase so as not to interfere with operation.	Useful in some cases for routine analyses of tritium or carbon-14 where material can be converted to gaseous phase. Can also be used for counting small amounts of THO as vapor, 100% efficient for beta particles. Small end and wall effects. BEA for C ¹⁴ as C ¹⁴ O ₂ —0.00003 μ c.
10. Internal-gas-ionization chamber.	High-voltage supply Vibrating-reed electrometer Chemical conversion system	Nuclide converted to gaseous phase.	Less sensitive than internal gas counter. However, the technique is less critical as to the type of gas present. Good G-M characteristics are not required so that larger amounts of the isotope as a gas may be counted. Sensitivity similar to $2\pi\beta$ -air-ionization chamber. BEA for C ¹⁴ as C ¹⁴ O ₂ —0.001 μ c.
11. ZnS alpha scintillation counter (used where β or γ counting rates interfere with α detection).	Precision high-voltage supply Linear amplifier Discriminator Multiplier phototube Scaler	Dried on planchet.....	Detects α 's in presence of high intensity β 's and γ 's. Efficiency is practically 100% for α particles striking ZnS film.

^a All equipment listed is available commercially in U.S. A.

^b BEA (Background Equivalent Activity) is a measure of the intrinsic sensitivity of a detector. It is the number of disintegrations per second of the particular nuclide that will produce an effect equal to the background effect.

efficient than anthracene (Seliger and Ziegler, 1956a, 1956b, and 1957). The most efficient liquid scintillator combinations reported to date are approximately 4 g/liter 2,5-diphenyloxazole (DPO) with 0.1 g/liter 1,4-di-(2(5-phenyloxazole))-benzene (POPOP) in toluene, and approximately 8 g/liter phenylbiphenyloxazadiazole (PBD) zene (POPOP) in toluene (Hayes, Orr, and Kerr, 1956). Under carefully controlled conditions of solution preparation and measurement it has recently been possible to intercompare tritiated-water standards received from Atomic Energy of Canada Limited, Chalk River, Canada, and Los Alamos Scientific Laboratory, Los Alamos, New Mexico, with the NBS tritiated-water standard with a precision of a few tenths of 1 percent (fig. 32). In this case a water: alcohol: xylene ratio of 1: 50: 250 was used in the system with 4 g/liter DPO as the scintillating solute. For certain other tracer applications involving lower activities of material and therefore requiring the measurement of larger volumes of water, the use of dioxane as a

solvent will permit a much larger percentage of water to be incorporated into the measuring cell (Langham, Eversole, Hayes, and Trujillo, 1956).

5.1. Considerations in Beta-Ray Calibrations

To use beta-ray solution standards it is necessary to open the standard ampoule and to prepare a standard source for comparison, or for calibration of a counting system. Assuming that the electronic equipment is suitable and the detector adequately stable, the following are the most important considerations:

a. Source Self-Absorption

Most beta-ray solution standards distributed by the National Bureau of Standards have a minimum of total solids present, consistent with chemical stability and freedom from adsorption effects. In some instances, however, where the

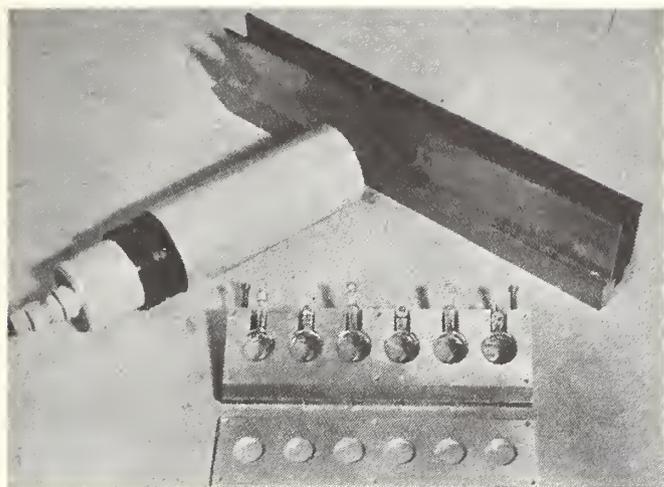


FIGURE 32. Sliding sample arrangement used for comparing liquid scintillating samples.

The slots on the bottom make for reproducibility of setting. The black felt on the sides of the slide holder prevents any but the light from the sample being viewed from reaching the photocathode of the phototube. The cells are 1-inch-diameter Pyrex absorption cells. The flame-sealed sample at the extreme right is a blank solution for determination of background. With this arrangement, a precision of 0.1 percent was obtained.

original material may be of low specific activity, as, for example, in the carbon-14 solution standards, the absorption of beta rays from a dried source may be quite appreciable on account of the amount of solids present. Because of the low energy in the case of the carbon-14 beta-ray spectrum ($E_{\max}=155$ kev), the observed counting rate will be a strong function of the amount of solids present as well as the method of source preparation. Even in the case of practically weightless deposits of cobalt-60 ($E_{\max}=310$ kev), Seliger and Schwebel (1954) have found as much as a 10-percent difference in observed counting rate in a $4\pi\beta$ counter, depending on the method of preparation of the source. This was observed independently by Smith and Wildblood (1953). It is therefore suggested in the cases of low-energy beta emitters that comparison sources be prepared from carrier solutions of nearly identical solid concentrations when low to medium solid sources are desired. Otherwise "infinitely" thick liquid or solid sources should be prepared. The $2\pi\beta$ -gas-flow proportional counter has almost 100 percent efficiency for a beta particle entering the sensitive volume. A method of counting, using this type of counter for infinitely thick liquid samples has been described by Schwebel, Isbell, and Karabinos (1951) and Schwebel, Isbell, and Moyer (1954), in which the radioactive material is dissolved in formamide, an organic solvent having a very low vapor pressure compared with water. In this method a diluting solution of formamide containing 1 part by volume of carrier solution to 99 parts of formamide is prepared. The radioactive solution to be standardized is then diluted by a factor of 100 with the formamide-carrier solution to reduce the water concentration to 1 percent (fig. 33). At this low concentration water vapor does not appear to change the count-

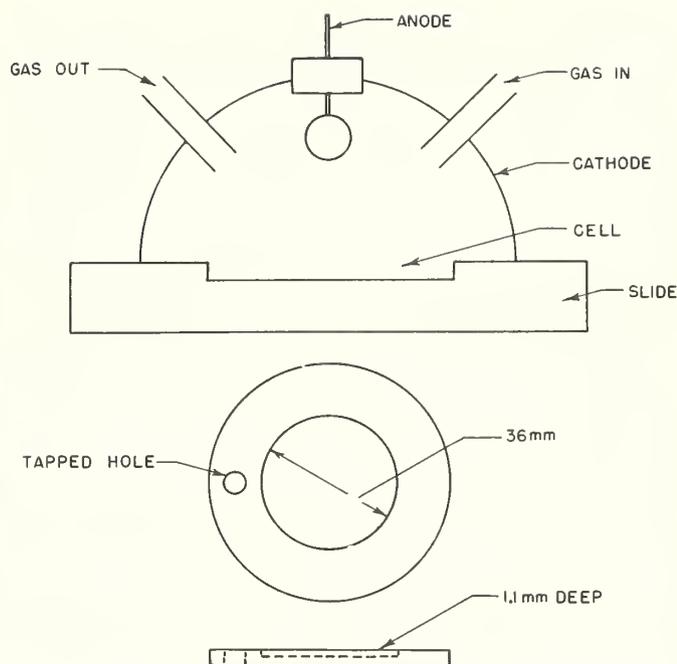


FIGURE 33. Schematic drawing of the NBS formamide counter (Schwebel, Isbell, and Karabinos, 1951; Schwebel, Isbell, and Moyer, 1954).

The cell, to which the active material in 99-percent formamide solution is added, is made of stainless steel. At 1-percent concentration in formamide, water vapor apparently does not change the counting characteristics of the $2\pi\beta$ -gas-flow counter.

ing characteristics of the $2\pi\beta$ flow counter. Usually 1 ml of the 1-percent solution is pipetted into a shallow stainless-steel cell. As the thickness of the solution is greater than the beta-particle saturation thickness, the counting rate is proportional only to the solution activity and to the area of the cell. If the volume and hence the depth of solution is always the same, any effects due to gamma rays will be constant. Thus the formamide method of liquid counting can also be used for beta emitters that have accompanying gamma rays.

"Infinitely" thick solid samples can be prepared uniformly from slurries of active material containing carrier. Care must be taken in the use of slurries or partial digestions or wet ashings because of slight differences in self-absorption and scattering due to changes in deposition of colloidal particles and possible settling. It may be necessary to stir the sample continuously or to use rotating planchets to average differences in concentration over the source. Even in the cases of the infinitely thick solid and liquid sources it must be remembered that the effective atomic number of the material will to some extent determine the amount of self-scattering and therefore the self-absorption (Nervik and Stevenson, 1952; Walton, Thompson, and Croall, 1953; Bowles and Walton, 1954; Suttle and Libby, 1955). In order to obtain reproducible measurements the chemical compositions of the samples being compared must be as alike as possible. For example, if identical areas of infinitely thick sources of carbon-14 as sodium carbonate and of carbon-14 as barium carbonate

are compared, the barium-carbonate carbon-14 will give the higher reading even though the sources have the same specific activity. For beta emitters of E_{\max} greater than 300 kev the problem of source self-absorption is not so important, and samples can be intercompared by drying aliquots on planchets and inserting these in position in the $2\pi\beta$ -flow counter. However, there is yet another important consideration.

b. Backscattering

For the routine analysis of dried samples of material various workers have used planchets or cups made of aluminum, glass, copper, stainless steel, palladium, or platinum, on which aliquots of solution were dried. The conditions of the experiment and the economic limitations of the cost of sample holders will determine the type of backings used. In any case, one must carry out all the measurements using the same type of backing, since the backscattering of beta rays is dependent on the atomic number of the backing material (fig. 34). This atomic-number dependence has been investigated for both electrons and positrons (Seliger, 1950 and 1952).

The effect of the self-scattering on observations made with end-window and 2π -geometry counters was shown very strikingly by Bowles and Walton (1954) and is reproduced in figure 35.

Under certain conditions the use of a nonconductor such as glass as a source backing can induce electric charges inside a counter. At the National Bureau of Standards sources are dried on palladium-faced silver disks. Palladium is less expensive than either gold or platinum, is close to silver in atomic number and therefore in backscattering properties and, unlike aluminum, copper, steel, and silver itself, is inert to most acids and bases.

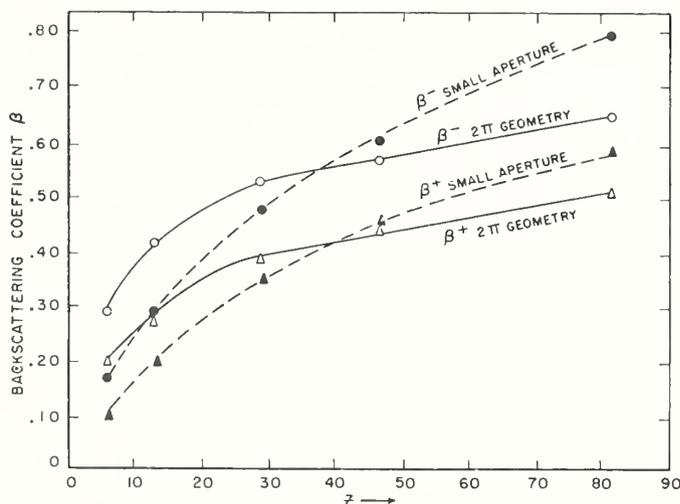


FIGURE 34. Backscattering coefficients for positrons and electrons as functions of the atomic number of the backing material for both end-window (low) geometry and 2π geometry (Seliger 1952).

The dependence of the backscattering coefficient upon the geometry of the measurement is due to the anisotropy in the angular distribution of the backscattered radiation. In the comparison of different samples, the source backings should be of the same atomic number.

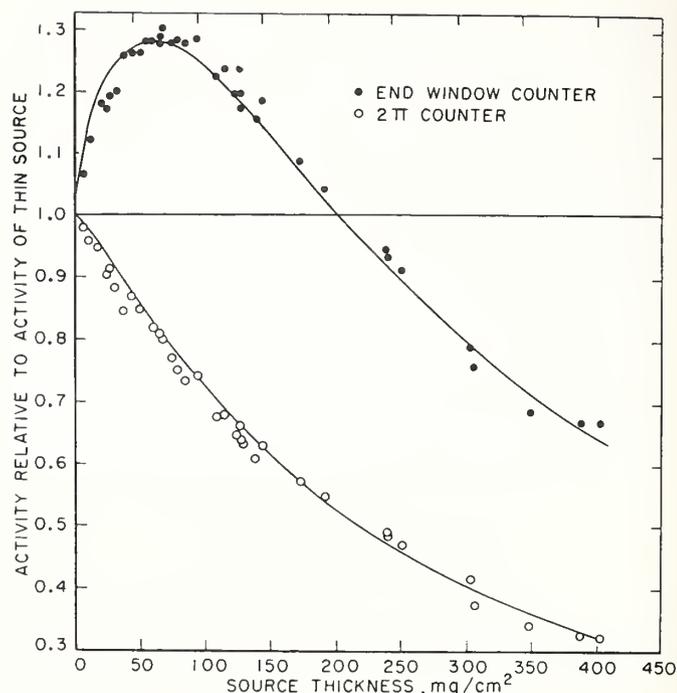


FIGURE 35. Results obtained by Bowles and Walton (1954) showing the effect of geometry and source self-scattering on the counting rates observed from thick sources.

The counting rate relative to that of a thin, almost weightless, source is plotted as a function of the thickness of the source, with the activity of the source remaining constant. From symmetry considerations the monotonic decreasing curve obtained with the $2\pi\beta$ counter is to be expected since an increase in solids can lead only to an increase in source self-absorption. However in the case of the end-window counter, with a low geometry, it is possible for beta rays to be anisotropically scattered into the forward direction, producing the anomalous effect shown in the figure. The same type of curve can be obtained for a thin source when an absorption curve in aluminum is plotted if the aluminum absorbers are placed on top of the source instead of directly in front of the counter window. Here again the self-focusing is initially more important than the absorption. Some examples of anisotropic angular distributions of backscattered beta rays have been given by Seliger (1952). (Reproduced, by courtesy, from AERA Report C/R 1463.)

c. Low-Solids Source Preparation

For the delivery of a small-volume source there is available a delivery-type mercury-displacement ultramicroburet that can deliver quickly and precisely volumes of solution as small as 0.01 ml with an accuracy of 0.5 percent. No particular problems are encountered in the preparation of most medium- and high-energy beta sources. The technique of air drying at room temperature has been quite satisfactory. For low-energy beta emitters and even in those cases of the higher-energy beta emitters such as Na^{22}Cl and $\text{Sr}^{90}\text{Cl}_2$, which form crystalline salts, it is necessary to prepare sources carefully to avoid self-absorption effects. Methods that are employed to obtain minimum self-absorption include freeze drying (Perry, 1954b); precipitation by addition of specific salts (such as AgNO_3 added to NaI^{131} solutions to precipitate AgI^{131}); slow evaporation in a saturated ammonia atmosphere (Seliger and Schwebel, 1954), in which ammonia induces precipitation in the form of an ammonium complex; vacuum distillation from organic chelate compounds (Pate and Yaffe, 1956); vacuum evaporation at higher temperatures (Stockendal and

Bergkvist, 1957); and adsorption of radioactive gases onto thin evaporated films (Pohm, Waddell, Powers, and Jensen, 1954).

5.2. Consideration in Gamma-Ray Calibrations

Except under special conditions, it is not intended that a gamma-ray solution standard be opened or destroyed. The following procedure is suggested for the application of a 5-ml gamma-ray standard. A stock solution of the same isotope is diluted so that 5 ml of the final dilution contains approximately the same activity as the standard ampoule. A "laboratory standard" ampoule of the same dimensions as the standard ampoule is prepared containing an accurately known volume close to 5 ml. The laboratory standard is then compared directly with the primary standard under identical conditions of geometry, e. g., in a well-type scintillation counter, thus standardizing the entire solution. In this way the radioactivity standard can be retained as a check on further solutions, or in case of any doubts as to chemical stability or losses from the solutions used for the actual experiments. The detector efficiencies can be determined again as in eq (1) from the newly-prepared "laboratory standard" or else other types of special sources can be prepared from it.

Some electron-capturing nuclides such as manganese-54, zinc-65, and cadmium-109 have nuclear gamma rays associated with their decay. These therefore can be used for standardization in the same manner as other gamma-ray emitters.

These characteristic X-radiations following the process of electron capture can be used as a source of low-energy monochromatic gamma rays for the calibration of detectors. In table 4 are given those X-ray emitters of suitable half-life for practical measurements, together with the *K* X-ray quantum energy of the daughter atom and *K*-branching ratio. Electron capturers, such as iron-55 and nickel-59, have only low-energy daughter-atom X-rays associated with their decay. In these latter cases the method of measurement may involve the opening of the ampoule for the preparation of dried sources on planchets for X-ray counting and $2\pi\beta$ counting, or for the preparation of liquid sources for liquid scintillation counting. The same precautions should be observed here as for the low-energy beta emitters.

5.3. Simulated Standards and Reference Sources

It is sometimes advantageous in the cases of the short-lived radioactive nuclides to have available a simulated standard with which intermediate checks can be made of the detectors and of the total efficiency during the course of an extended experiment. A radium D+E reference source (with a half-life of 22 years) serves this purpose adequately for beta-emitters in the medium- and high-energy range.

As, however, recent measurements at the National Bureau of Standards have cast some doubt on the half-life of radium D it is necessary to recalibrate a radium D+E reference source at intervals depending on the accuracy required.

TABLE 4. *Electron-capturing nuclides*

Nuclide	Half-life	<i>K</i> X-ray energy (kev)	% <i>K</i> -capture
A ³⁷	34.1 days	2.82	100
Ca ⁴¹	1.2×10 ⁶ years	2.61	100
Cr ⁵¹	27.8 days	5.47	92
Fe ⁵⁵	2.94 years	6.54	100
Ni ⁵⁹	7.5×10 ⁴ years	7.71	100
Zn ⁶⁵	245 days	8.98	98.5
Ge ⁷¹	11.4 days	10.39	100
As ⁷³	76 days	11.10	100
Se ⁷⁵	127 days	11.87	100
Y ⁸⁸	105 days	16.10	99.8
Tc ⁹⁵	60 days	20.00	99.6
Cd ¹⁰⁷	6.7 hours	25.54	99.3
Cd ¹⁰⁹	470 days	25.54	78
I ¹²⁶	13.1 days	31.82	58
Cs ¹³¹	9.6 days	34.55	100
Ba ¹³¹	12 days	35.97	100
Ba ¹³³	9.5 years	35.97	100
Ce ¹³⁴	72 hours	38.92	100
Ce ¹³⁹	140 days	38.92	100
Nd ¹⁴⁰	3.3 days	41.99	100
Pm ^{142, 143}	Approx. 250 days	43.57	100
Pm ^{143, 144}	Approx. 300 days	43.57	100
Sm ¹⁴⁵	410 days	45.2	100
Eu ¹⁴⁵	5 days	46.85	100
Eu ¹⁴⁷	24 days	46.85	99+
Eu ¹⁴⁸	59 days	46.85	100
Gd ¹⁴⁹	9 days	48.51	99+
Gd ¹⁵³	236 days	48.51	100
Tb ¹⁵³	5.1 days	50.2	100
Tb ¹⁵⁵	190 days	50.2	100
Tb ¹⁵⁷	4.7 days	50.2	100
Dy ¹⁵⁹	134 days	52.0	100
Ho ¹⁶²	65 days	53.8	85
Ho ¹⁶³	5.2 days	53.8	100
Tm ¹⁶⁷	9.6 days	57.5	100
Tm ¹⁶⁸	85 days	57.5	98
Yb ¹⁶⁹	31.8 days	59.4	100
Lu ¹⁷¹	600 days	61.3	100
Hf ¹⁷⁵	70 days	63.3	100
Ta ¹⁷⁹	600 days	65.4	100
W ¹⁷⁸	21.5 days	67.5	100
W ¹⁸¹	140 days	67.5	100
Re ¹⁸³	155 days	69.5	100
Re ¹⁸⁴	50 days	69.5	100
Os ¹⁸⁵	97 days	71.7	74
Ir ¹⁹⁰	12.6 days	73.9	100
Pt ¹⁹¹	3 days	76.1	100
Au ¹⁹⁵	180 days	78.4	100
Au ¹⁹⁶	5.6 days	78.4	95
Hg ¹⁹⁷	65 hours	80.7	100
Tl ²⁰²	12.5 days	83.1	100

Calibrated radium D+E sources from the National Bureau of Standards can also be used for standardization that is accurate to ± 5 to 10 percent for nuclides whose beta-ray spectra are not too dissimilar to the radium-E beta-ray spectrum. The procedure briefly is as follows: An unknown source is prepared from a stock solution on a palladium-faced silver disk similar to the standard disk, and an absorption curve in aluminum is obtained for each source with say, an end-window beta-ray G-M counter in a fixed geometry. Extrapolation of these curves to zero total absorber will correct to a first approximation for the differences in the beta energies

and for slight differences in geometry from one calibration to the next (figs. 36 and 37). Since the backscattering in the energy range 300 kev to 1.7 Mev is independent of the energy distribution, one may assume that the fractional backscattering for the unknown source and for the radium-E beta rays is the same. The disintegration rate of the unknown source is given by

$$A = \frac{c/s \text{ zero total absorber (unknown)}}{c/s \text{ zero total absorber (Ra D+E)}} \times (\text{Ra D+E disintegration rate}). \quad (4)$$

The radium D+E disintegration rate is given on a certificate accompanying the standard and represents the total number of radium-D disintegrations per second contained in the source. Thirty days after preparation of the standard, the radium E will be in equilibrium with the radium D. Therefore this number is also the disintegration rate of the radium E. The use of a minimum of 6 mg/cm² of total absorber between the source and the counter will insure that there is no contribution from either low-energy radium-D beta rays or from the radium-F alphas which are also present.

The method can be made exact by performing the above measurements using as an "unknown", a source prepared from a *standard solution* of the nuclide. In this case a reference number for the nuclide can be assigned to the radium D+E

Ra D+E reference number =
activity of standard source

$$\times \frac{(c/s) \text{ extrapolated (Ra D+E)}}{(c/s) \text{ extrapolated (std. source)}}. \quad (5)$$

These reference numbers may be slightly different for each nuclide. Once more it should be emphasized that the very important considerations of stability of electronic equipment, technique of source measurement, and statistical design are always present, although they are not discussed in this Circular.

In some cases there exist long-lived beta emitters whose beta spectra are nearly identical to those of the short-lived nuclides. Sources prepared from these long-lived beta emitters can be assigned reference numbers as in eq (5), with the simplification that the absorption curves in aluminum are parallel, i. e., the ratio at only one thickness of absorber is necessary. Aten (1954), using end-window G-M counters, has found that the absorption of sodium-22 positrons is the same as that of iodine-131 electrons up to 90 mg/cm² of aluminum absorber, and that thulium-170 electrons are absorbed in the same manner as gold-198 electrons between 20 mg/cm² and 100 mg/cm² of aluminum absorber. At the present time there is a sufficient range of energies of beta-ray standards available for use as substitute standards, so

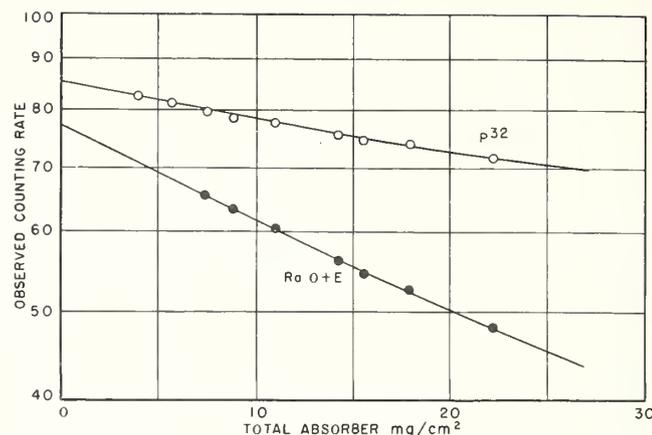


FIGURE 36. Approximate method for determining the disintegration rate of a phosphorus-32 sample by comparison with an NBS radium D+E reference source.

Sources are mounted on palladium-faced silver disks and absorption curves in aluminum are measured using an end-window Geiger counter. The window thickness can range from 1.4 to 4 mg/cm². The geometry is shown in figures 38 and 39. Since the beta-ray energies are different, it is necessary to extrapolate back to zero total absorber. There is a slight curvature below about 20 mg/cm².

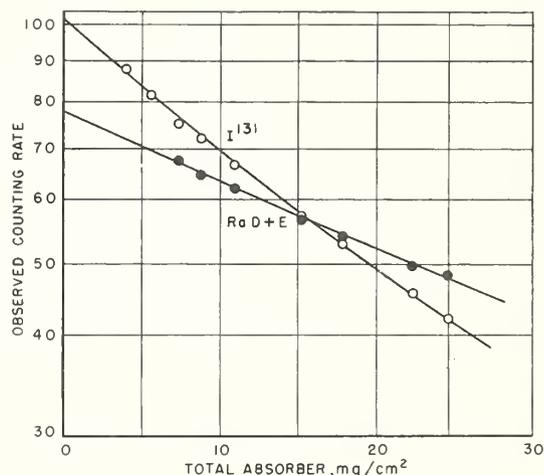


FIGURE 37. Approximate method for determining the disintegration rate of an iodine-131 sample by comparison with an NBS radium D+E reference source.

Sources are mounted on palladium-faced silver disks and absorption curves in aluminum are measured using an end-window Geiger counter. The window thickness can range from 1.4 to 4 mg/cm². The geometry is shown in figures 38 and 39. Since the beta-ray energies are different, it is necessary to extrapolate back to zero total absorber. There is a slight curvature below about 20 mg/cm².

that practically any beta emitter can be standardized to ± 10 percent routinely in the laboratory. Some representative transmission curves in aluminum, using end-window geometry are shown in figures 38 and 39. Mixtures of fission-product nuclides can be assayed, using thallium-204 as a substitute standard (Reynolds and Brooksbank, 1953; Whalen, Reynolds, and Brooksbank, 1954).

There are several simulated gamma-ray standards available, the most notable of which is the "mock iodine-131" standard developed by Brucer (1955; see also Brucer, Oddie, and Eldridge, 1956). In this case an empirically determined mixture of barium-133 and cesium-137-barium-137 produces, with proper filtration, a gamma-ray spec-

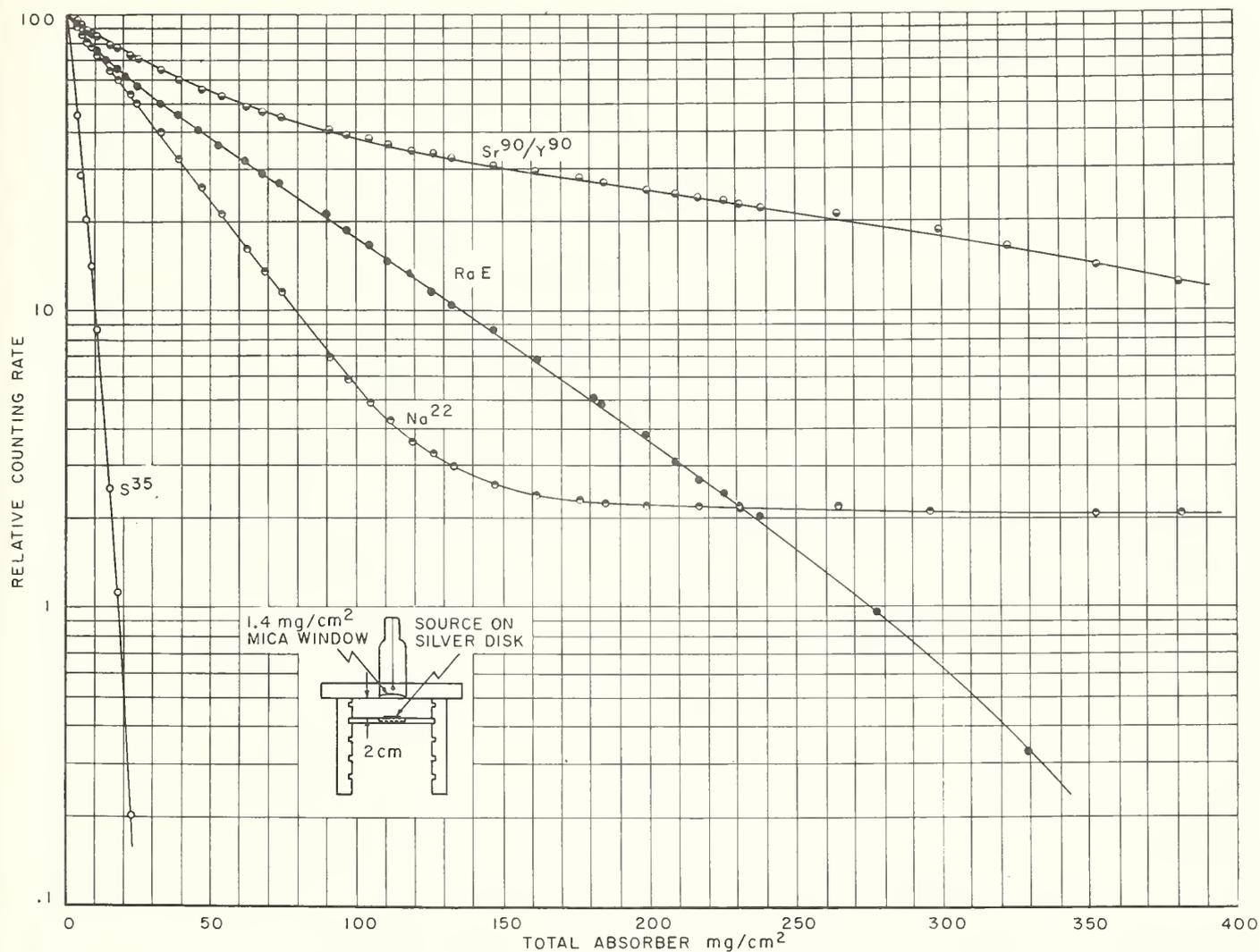


FIGURE 38. Transmission of beta rays of the radionuclides sodium-22, sulfur-35, strontium-90/yttrium-90, and radium E through aluminum.

The geometry is shown in the figure. These curves can be used as qualitative checks on the radioactive purity of a sample. The shapes of the curves are very similar. There is a slight concave-upwards portion at the beginning, a straight-line (exponential) portion over most of the range and then a tailing off into a gamma-ray portion as in the case of sodium-22 or to negligible transmission for pure beta emitters. All sources used were mounted on palladium-faced silver disks. Absorbers were placed as close to the counter as practicable to minimize self-focusing effects similar to those illustrated in figure 35.

trum almost identical with that of iodine-131. Two sets of such "standards" have been "standardized" in a $4\pi\gamma$ -ionization chamber by direct comparison with iodine-131 previously standardized in the $4\pi\beta$ counter. The long half-lives of the barium-133 and the cesium-137 will permit the simulated standards to be used for approximately 10 years before there is much change in the spectrum due to the differences in decay. Not only is a long-lived simulated standard advantageous as a check on calibrations during the course of an experiment, but the material can be incorporated into actual phantoms and into various organ shapes in order to serve as a reference in, say, iodine-uptake studies in human beings. At the present time such simulated standards of iodine-131 are being used throughout the United States in intercomparisons of iodine-131 uptake measurements, in an effort to make diagnoses more precise and to standardize the method of measurement.

Another example of the use of a substitute standard for gamma rays is that of cobalt-60 whose gamma rays are suitable for the standardization of sodium-22, iron-59, and zinc-65. Since the response of a NaI(Tl) crystal, and of an ionization chamber to some extent, is dependent upon the energy of the gamma rays being detected, it is possible, using previously standardized nuclides having widely separated gamma-ray lines associated with their decay, to determine empirically a smooth curve of response per unit activity versus energy for a given NaI(Tl) scintillation counter (Kahn and Lyon, 1953) or with a given ionization chamber. With this smooth curve it should then be possible to standardize, by interpolation or extrapolation, gamma emitters of practically any energy. This will be of special interest to nuclear spectroscopists and those interested in determining absolute gamma-ray yields from nuclear reactions.

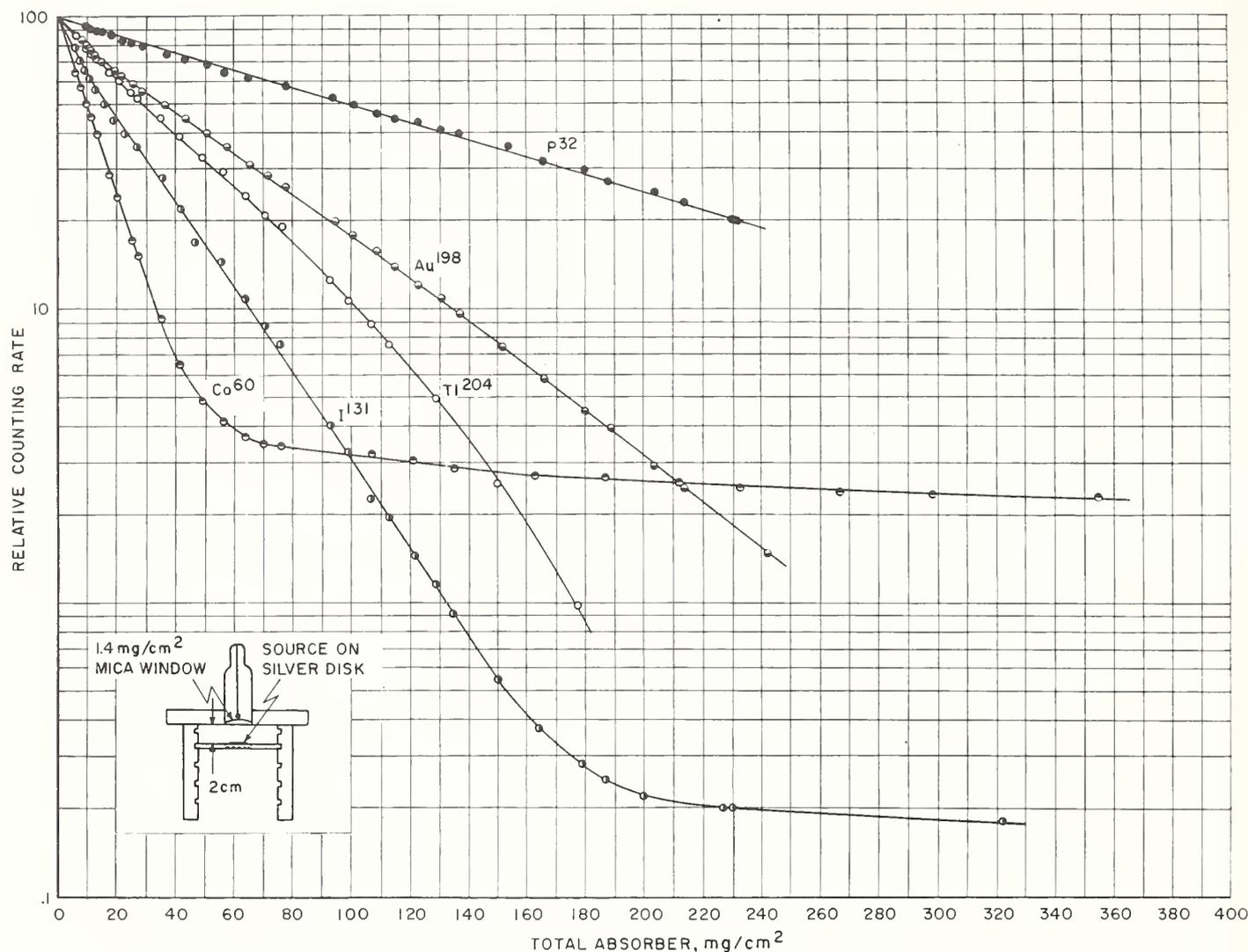


FIGURE 39. Transmission of beta rays of the radionuclides phosphorus-32, cobalt-60, iodine-131, gold 198, and thallium-204 through aluminum.

The geometry is shown in the figure. These curves can be used as qualitative checks on the radioactive purity of a sample. The shapes of the curves are very similar. There is a slight concave-upwards portion at the beginning, a straight-line (exponential) portion over most of the range and then a tailing off into a gamma-ray portion as in the case of cobalt-60 and iodine-131 or to negligible transmission for pure beta emitters. All sources used were mounted on palladium-faced silver disks. Absorbers were placed as close to the counter as practicable to minimize self-focusing effects similar to those illustrated in figure 35.

The currents per unit activity as functions of energy are given in figures 40 and 41 for the National Bureau of Standards $2\pi\beta$ - and $4\pi\gamma$ -ionization chambers, respectively. This technique, in principle, can be applied to other types of detectors.

5.4. Complex Standardization Procedures

There arise many cases in which standardizations are to be made of mixtures of radionuclides. Fission products, for which thallium-204 serves as a substitute standard (Reynolds and Brooksbank, 1953; Whalen, Reynolds, and Brooksbank, 1954) are an example of a mixture being standardized by means of a single average standard. In other cases, mainly in medical and biological research, the simultaneous use of two or more different

tracers is of great assistance to the researcher. In this way each tracer nuclide, whose effects are to be observed separately, will have had the same host under exactly the same experimental conditions. Double-labeling experiments involving hydrogen-3 and carbon-14 make use of the difference in the beta spectra of these two nuclides. The method of measurement involves (a) incorporating the samples in a liquid-scintillator solution and (b) a two-channel pulse-height analysis of the observed pulses. The relative positions and widths of the two windows of the two-channel pulse-height analyser are adjusted, using standard solutions of the nuclides, so that most of the hydrogen-3 counts occur in the lower window and most of the carbon-14 counts occur in the upper window. With the hydrogen-3 and carbon-14 standard solutions the channels and the response can there-

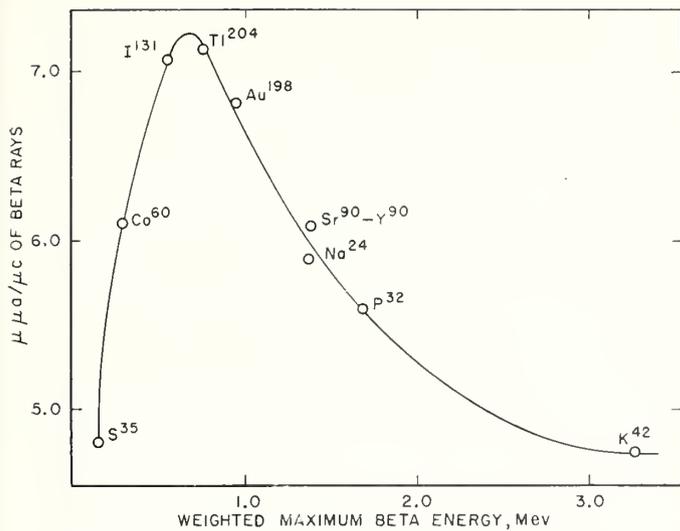


FIGURE 40. Curve showing the energy response of the NBS $2\pi\beta$ -ionization chamber.

The current per unit activity has been plotted as a function of the maximum beta-ray energy. Where there is beta-ray branching this energy value is the weighted average of the energies of the branches. The response curve appears to be a smooth curve on both sides of a peak at a maximum energy of approximately 0.7 Mev. The existence of a peak and the anomalous decrease above $E_{\max}=0.7$ Mev are due solely to the finite dimensions of the chamber. The high-energy beta rays of potassium-42 with their consequently smaller specific ionization will strike the walls and be absorbed before producing as many ion pairs as the lower-energy beta rays of gold-198. Most of the sulfur-35 beta rays are absorbed in the gas of the $2\pi\beta$ -ionization chamber. However, these are quite low in energy and thus produce relatively few ion pairs.

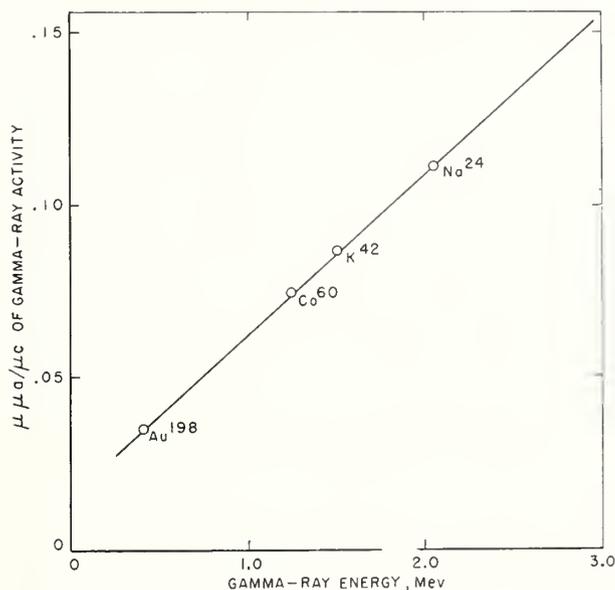


FIGURE 41. Curve showing the energy response of the NBS $4\pi\gamma$ -ionization chamber.

The current per-unit activity has been plotted as a function of the gamma-ray energy. Where two gamma rays appear in cascade, the energy values have been averaged. Since most of the ionization current measured is the result of Compton scattering, one would expect the energy dependence shown. It should therefore be possible, knowing the decay scheme of a particular radionuclide, to interpolate and to standardize the disintegration rate of a sample of the nuclide by measurement of the current only.

fore be calibrated and the overlap reduced to a minimum. A description of the simultaneous use of sodium-24 and potassium-42 as tracers is given by Tait and Williams (1952). One method of separating the components of a mixture of

beta emitters is the graphical analysis of beta-absorption curves (Harley and Hallden, 1955). Harley and Hallden (1955) give a table of the semi-logarithmic slopes of a large number of beta emitters relative to phosphorus-32 and calcium-45.

A number of nuclides emit several gamma rays with different energies. In these cases the various gamma rays can be separated, using the well-type scintillation counter with pulse-height analysis. Similarly, mixtures of gamma-emitting nuclides can be separated by counting under the photopeaks corresponding to the most suitable gamma rays. By means of an efficiency curve obtained with gamma-ray standards the components of the mixture can be analyzed quantitatively. The simultaneous use of Cr^{51} - Fe^{59} and Na^{24} - K^{42} samples is described by Hine, Burrows, Apt, Poelycave, Ross, and Sarkes (1955). A similar application of the energy dependence of the light output of the $\text{NaI}(\text{Tl})$ crystal was made by Upson, Connally, and Leboeuf (1955) in counting plutonium-239 gamma rays in the presence of fission-product gamma rays.

5.5. Bremsstrahlung

In addition to its versatility and high efficiency for nuclear gamma rays, the well-type scintillation counter can be used to compare beta sources by measurement of the internal and external bremsstrahlung. Loevinger and Feitelberg (1955) have recently described a method of counting phosphorus-32 routinely by means of the bremsstrahlung from solutions of this nuclide in a well-type $\text{NaI}(\text{Tl})$ crystal. The efficiency is given as 0.8 count per 100 disintegrations. For a beta emitter an approximate expression for the energy radiated as bremsstrahlung (Wyand, 1955) measured in million electron volts per beta ray, is

$$B = 1.23 \times 10^{-4} (\bar{Z} + 3) E_{\beta}^2 \quad (6)$$

when \bar{Z} is the effective atomic number of the material in which the beta particles are brought to rest and E_{β} is the maximum energy of beta disintegration. As a rule of thumb, 85 percent of the total intensity of bremsstrahlung from a beta emitter is emitted in the range 0 to $0.3 E_{\beta}$, in the ratio 45:25:15 for 0 to $0.1 E_{\beta}$, 0.1 to $0.2 E_{\beta}$, and 0.2 to $0.3 E_{\beta}$, respectively. From eq (6) the energy lost per disintegration from sulfur-35 ($E_{\beta}=166$ kev) would be one-hundredth that of phosphorus-32. In addition, 45 percent of the bremsstrahlung would have energies less than 16 kev, and would therefore be highly absorbed in the housing of the scintillation crystal. The technique should therefore be used for the higher-energy beta emitters. The only disadvantage of the method of measurement is its extreme sensitivity to gamma-emitting impurities, although this can be turned into an asset in the determination of radioactive purity.

6. Determination of Absolute Efficiencies of Detectors

A number of examples of the use of beta- and gamma-ray standards for the determination of the absolute efficiencies of detectors has been reported. Smith, Seliger, and Steyn (1956) have used standardized solutions of phosphorus-32, cobalt-60, strontium-90—yttrium-90, iodine-131, and thallium-204 to determine the absolute efficiency of the 4π -crystal-scintillation-counting technique for beta rays of varying end-point energies.

Gamma-ray standards have been applied to the absolute calibration of experimental equipment used to investigate Coulomb excitation of nuclei. Heydenberg and Temmer (1954), Temmer and Heydenberg (1955), and Fagg, Wolicki, Bondelid, Dunning, and Snyder (1955) have made use of the gamma rays from standards of sodium-22, iodine-131, cerium-141, and gold-198 in order to calibrate their equipment and so determine absolute gamma-ray yields. In the Coulomb-excitation experiment a very thin layer of the target material

is laid on top of a Na I(Tl) crystal and is bombarded with alpha particles or protons. The gamma rays emitted from any low-lying excited levels are then detected by means of a scintillation spectrometer. To determine the absolute efficiency, a standardized gamma-ray source of the same size as the beam cross section is delivered to the target material. The ratio of source strength to area under the photopeak then yields one point of an absolute photopeak efficiency curve. A similar application has been made by Swann and Metzger (1955) for the absolute calibration of their gamma-ray detectors. These were used in investigating the cross sections for the production of isomeric states excited in nuclei by inelastic scattering of monoenergetic neutrons. Heft and Libby (1955) have measured absolute cross sections for deuterons on beryllium from absolute measurements of the disintegration rate of the tritium produced in the reaction.

7. Determination of Long Half-Lives

In determining the half-life of a radioactive nuclide by counting measurements for several half-lives, the familiar relationship

$$N = N_0 e^{-\lambda t} \quad (7)$$

is reduced to

$$\ln \lambda N = \ln \lambda N_0 - \lambda t \quad (8)$$

and the disintegration constant λ is found from the slope of a linear plot, whence the half-life T is given by

$$T = \frac{\ln 2}{\lambda} \quad (9)$$

When it is impracticable to measure for a sufficiently long period, the disintegration constant can be obtained by using the more fundamental decay law

$$dN/dt = -\lambda N. \quad (10)$$

If the half-life is so long that the disintegration rate dN/dt of a sample does not change appreciably during a measurement, λ can be deduced from a knowledge of N , the number of nuclei present at the beginning (or end) of the experiment, and of dN/dt .

For isotopically pure elements such as radium the determination of N can be made by gravimetric means. For isotopically pure elements that can be converted to the gas phase a measurement of pressure and temperature in a defined volume will yield N . In most cases, however, due to the presence of other isotopes of the same element, isotopic analysis by means of the gas-density balance or mass spectrometer is necessary.

The determination of N in the measurement of the half-life of carbon-14 is an example of these

latter procedures. The carbon-14 half-life is of particular importance in archaeology for the accurate dating of organic relics. In principle the procedure for the determination of the half-life is as follows:

A sample of, say, carbon-14-tagged carbonate is converted to carbon dioxide. A small but accurately-known fraction of this gas is mixed thoroughly with an appropriate counter gas at a definite pressure, and the sample is counted with nearly 100-percent efficiency. Appropriate end and wall corrections are made as discussed previously. The sensitive volume of the counter together with the pressure and temperature give the total number of molecules of carbon dioxide within the sensitive volume of the counter but do not differentiate among carbon-12, carbon-13, and carbon-14. The isotopic abundance ratio of the carbon-dioxide gas must also be determined in the mass spectrometer or in the gas density balance. Since the quantitative count in the gas counters is due only to carbon-14 and the isotopic abundance ratio yields $C^{14}/(C^{12} + C^{13} + C^{14})$ we have dN/dt (C^{14}) and $N(C^{14})$ as required by eq (10).

At the present time the reported values of the carbon-14 half-life range from 5,360 years (Manov and Curtiss, 1951) to 6,360 years (Hawkings, Hunter, Mann, and Stevens, 1949).

In an analogous manner to the carbon-14 half-life determination, Jones (1951) has used a disintegration-rate standard of tritium to determine the half-life of tritium. In this case the isotopic abundance ratio measurement can be quite accurate because of the large relative differences in the atomic weights of the hydrogen isotopes. As has already been mentioned in this Circular, Jones later (1955) also determined dN/dt mass spec-

tiometrically by assaying the helium-3 collected in a given time.

A determination of the half-life of potassium-40 has recently been made by Suttle and Libby (1957). In this case the gamma rays from a standardized sample of cobalt-60 were used to determine the absolute gamma efficiency of the

experimental arrangement, a known amount of cobalt being homogeneously incorporated into the sample. A small correction was necessary for the differences in gamma-ray efficiencies of their counters between the 1.17- and 1.33-Mev gamma rays of cobalt-60 and the 1.46-Mev gamma rays from argon-40.

8. Historical and Geological Dating From Half-Lives

The constancy of the disintegration constant of any group of nuclei, independent of external physical or chemical environment, forms the basic assumption of all age determinations based on radioactivity decay. From investigations of pleochroic haloes in mica it has been shown that the ranges of alpha particles from a number of natural radioactive species have always remained constant. Since it has also been shown, both empirically (Geiger and Nuttall, 1911) and theoretically (Gamow, 1928; Gurney and Condon, 1928), that the decay constant is uniquely related to the energy and therefore to the range of the alpha particles ($\log \lambda = a + bE$), it follows that the decay constant has not changed. In particular, it is 1.54×10^{-10} year⁻¹ for uranium-238 corresponding to a half-life of 4.49×10^9 years.

With this constancy definitely established, the half-life becomes a unit of time measurement. For example, in the naturally occurring uranium series there are eight successive alpha transformations from uranium-238 to lead-206. Each alpha particle at the end of its range becomes a helium atom, and since 1 cm³ of helium contains 2.687×10^{19} atoms, it can readily be shown that 1 g of uranium-238 in secular equilibrium with all daughter products produces 1.16×10^{-7} cm³ of helium per year.

Assuming no loss of daughter products, the age of the rock is given by

$$8.6 \times \frac{\text{Volume of He}^4 \text{ in cm}^3}{\text{Mass of U}^{238} \text{ in grams}} \text{ million years.}$$

Alternatively, the age can be determined from the $\text{Pb}^{206}/\text{U}^{238}$ ratio, the $\text{Pb}^{207}/\text{Th}^{232}$ ratio, or the $\text{Pb}^{206}/\text{Pb}^{204}$ ratio (Nier, 1939). In each of these cases no loss of daughter products must have occurred between the time of formation of the rock and the time of measurement. A review of this field and a fairly complete reference list can be found in articles by Burling (1952) and Kulp, Broecker, and Eckelmann (1953). A more recent summary of the field and of the use of separated isotopes for the determination of geologic ages by means of the $\text{K}^{40}\text{-Ar}^{40}$ and the $\text{Rb}^{87}\text{-Sr}^{87}$ ratios has been given by Aldrich (1956).

Near the other end of the dating scale is the carbon-14 method developed by Libby (1955).

In this case the time unit is approximately 5,600 years instead of 4.49×10^9 years. There is an important further qualification to the fundamental assumptions of constancy of decay, however. In the naturally radioactive series the material is incorporated into the rock and the quantitative amount of decay is measured. Carbon-14 on the other hand is a relatively short-lived nuclide produced by cosmic-ray bombardment in the reaction $\text{N}^{14}(n,p)\text{C}^{14}$. Only under the further assumption that the intensity of cosmic rays has been constant throughout the dating period is the carbon-14 dating method applicable. Living organic matter exchanges carbon dioxide with the atmosphere which is in equilibrium with the C^{14}O_2 produced in the atmosphere by cosmic rays. When the organism dies, the C^{14}O_2 already present is trapped, and loss of carbon-14 by decay is no longer compensated for by exchange of carbon dioxide with the atmosphere. The ratio of the amount of carbon-14 per gram present in any dead organic matter to the amount of carbon-14 per gram present today in living organic matter, together with the knowledge of the half-life of carbon-14, permits the determination of the "age" of the specimen. The sensitivity of the carbon-14 method has been extended to 40,000 years by means of large-volume liquid-scintillator counting (Arnold, 1954), where the carbon is incorporated into the liquid scintillator, and by high-pressure gas-phase counting of acetylene (Suess, 1954) and of carbon dioxide (Fergusson, 1955).

The limitations of available isotopes with appropriate half-lives and in proper chemical composition leaves a wide gap in the dating scale between 40 thousand and 40 million years ago. In this intermediate range, ages have been determined from both thorium 230 (half-life, 83,000 years) and chlorine-36 (half-life 4.4×10^5 years) measurements, the former for deep-sea sediments and the latter for relatively young surface rocks less than 1 million years old. The thorium-230 method makes use of the thorium-uranium ratio, assuming that the rate of adsorption of uranium by the sediment has been constant. The chlorine-36 in surface rocks is built up by the $\text{Cl}^{35}(n\gamma)\text{Cl}^{36}$ reaction due to slow neutrons from cosmic-ray showers. Both of these methods suffer from difficulties due to leaching and erosion.

9. Determination of Mean Energies and Energy Per Ion Pair

When the beta disintegration-rate standardization is combined with a measurement of the total energy-emission rate, one obtains the average energy \bar{E} defined by

$$\bar{E} = \frac{\int_0^{E_{\max}} N(E)E dE}{\int_0^{E_{\max}} N(E) dE}, \quad (11)$$

where $N(E) dE$ is the number of beta particles emitted in unit time having energies between E and $E+dE$. The only direct experimental method for the determination of the quantity

$$\int_0^{E_{\max}} N(E)E dE$$

is by calorimetry. The quantity $\int_0^{E_{\max}} N(E) dE$ is the disintegration rate of the source.

Generally it is easier to measure the disintegration rate of a source than to measure directly the total energy-emission rate. Thus for biological applications where one is interested in energy delivered to a particular volume, the quantity $\lambda N\bar{E}$ is of interest, \bar{E} having been determined from eq (11).

In actual practice, calorimetric measurements of the rate of energy absorption in any medium are difficult to make. However, with a knowledge of the stopping-power ratio for the medium in question to air, use can be made of the ionization produced in air by the radiation, which is related to the rate of energy absorption in air by the ratio W_{air} , the mean energy in electron volts expended in the production of an ion pair in air.

Since the roentgen, in X- and gamma-ray dosage, is defined in terms of ionization in air, and since most dosimeters, with the exception of the chemical dosimeters, make use of this ionizing property, it has been of considerable interest to obtain accurate values of W_{air} for electrons. Experimentally one obtains

$$W_{\text{air}} = \frac{\int_0^{E_{\max}} N(E)E dE}{J_{\text{air}}}, \quad (12)$$

where J_{air} is the total current of ion pairs produced by the source and $\int_0^{E_{\max}} N(E)E dE$ is determined with a calorimeter. W_{air} for sulfur-35 beta rays has recently been determined using eq (12). The rate of energy emission of an approximately 400-mc source was measured in a Peltier microcalorimeter (Mann, 1954b) and the total ionization current from specially prepared sources was measured in a large cylindrical ionization chamber (Bay, Mann, Seliger, and Wyckoff, 1957). The value for W_{air} 33.7 ± 0.3 ev per ion pair agrees quite well with the value of 33.6 ev per ion pair, based on measurements made in an extrapolation ionization chamber by Gross, Wingate, and Failla (1957) and the most recent value of 33.9 ev per ion pair reported by Jesse and Sadaukis (1957). A review of the entire field of the measurement of W for other gases as well as for air and also for alpha particles has been given by Binks (1954).

Standards of radioactivity form an essential link between the measurement and the interpretation of physical and chemical phenomena in both the biological and the physical sciences. Their importance will undoubtedly grow as the increasing availability of radioactive materials makes their use more widespread.

10. International Comparisons of Radioactivity Standards

Within the last 5 years a great many intercomparisons of radioactivity standards have been carried out between the standardizing laboratories of Canada, the United Kingdom, and the United States (Astin, Bullard, and Lewis, 1956a and 1956b). The results that are available of these intercomparisons are summarized as follows:

10.1. Radium-226

In the summer of 1952 one of the two United States Hönigschmid radium standards (No. 5440) was taken to the National Physical Laboratory and compared with the British Hönigschmid radium standard (No. 5432) (Perry, 1955). In February 1954 the British Hönigschmid standard (No. 5432) was brought to the National Bureau of Standards and compared with the United States

Hönigschmid standards (Nos. 5437 and 5440) (Davenport, McCraven, Mann, and Smith, 1954; Connor and Youden, 1954). In November and December 1955 the Hönigschmid standard of the Federal Republic of Germany (No. 5426) and the Canadian Hönigschmid standard (No. 5425) (Garrett and Geiger, 1956) were brought to the National Bureau of Standards for comparison with the United States standards (Loftus, Mann, Paoletta, Stockmann, and Youden, 1957) (fig. 42); and between these last two measurements the Federal Republic of Germany standard was compared at the National Research Council's laboratories in Ottawa with the Canadian standard. The measurements of these standards carried out at the National Bureau of Standards using the electroscopes and calorimeter agreed to within 0.3 percent with the results of Hönig-

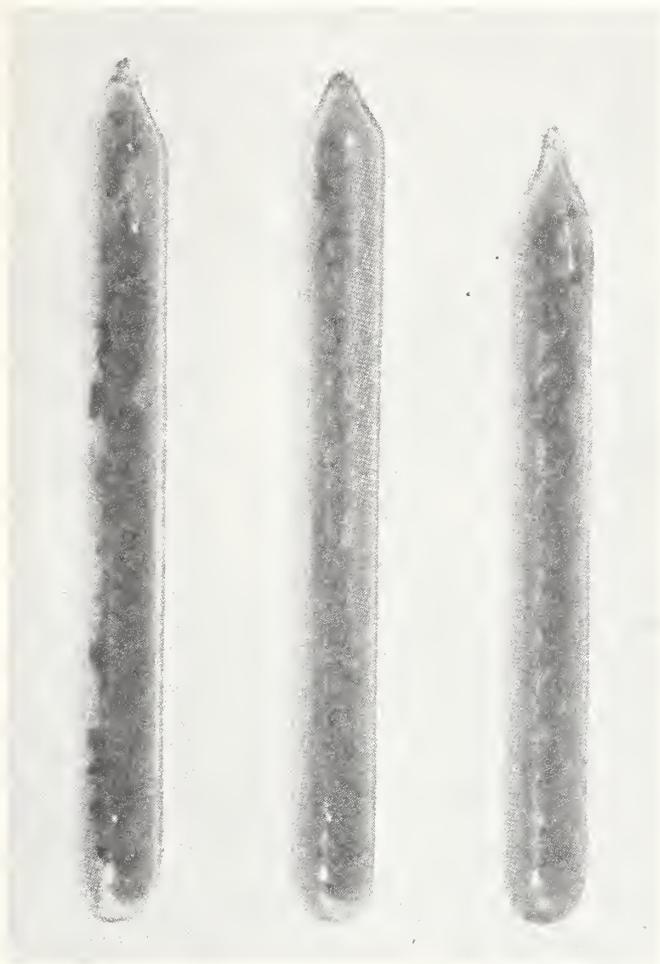


FIGURE 42. Three primary national radium standards that were prepared by Hönigschmid in 1934.

The standards are, from left to right, United States Primary Radium Standards numbers XIV (Hönigschmid No. 5437) and XV (Hönigschmid No. 5440) and the Primary Radium Standard of the Federal Republic of Germany (Hönigschmid No. 5426). The standards are shown with the grains of radium chloride distributed along the lengths of the glass vials as they would normally be arranged for measurement by the National Bureau of Standards standard electroscope.

schmid's weighings. By combining all three determinations at the National Bureau of Standards with Hönigschmid's own weighings, "best estimates" of the masses of radium have been made (Loftus, Mann, Paoletta, Stockmann, and Youden, 1957).

In the autumn of 1952 the United States Hönigschmid radium standard (No. 5440) was taken to Ottawa for comparison, by the method of gamma-ray ionization measurement, at the National Research Council's laboratories with the old Canadian National Radium Standard (XIII), which was not one of the original Hönigschmid standards (Michel and Whyte, 1955). As the Hönigschmid standards are loosely packed and the old Canadian standard tightly packed, it was necessary to calculate absorption corrections for each of the two standards. In the description of this intercomparison by Michel and Whyte (1955) the results of all previous measurements of the Canadian standard are quoted and the mean given for all values determined by the gamma-ray

ionization method. None of the individual ionization values differs from this mean by more than ± 0.1 percent, but the calorimetric determination at the National Bureau of Standards is noted to have given a value for the radium content that was 0.4-percent higher. Subsequently, in 1957, the United States standard No. 5440 was compared with the new Canadian standard in Ottawa (Geiger, 1957).

A number of very precise comparisons of Hönigschmid standards, including the new Canadian standard (No. 5425), with the 1911 Paris (Marie Curie) and Vienna standards and with the new international standard (Hönigschmid No. 5430) has also recently been carried out at Brussels (Kipfer, 1956) by the balanced-ionization-chamber method. The precision of the results obtained, of a few parts in 10,000, takes one, however, at least one order of magnitude beyond the uncertainties involved in the self-absorption of the sources themselves.

10.2. Hydrogen-3

Hydrogen-3 standards, in the form of tritiated water, were distributed to a number of laboratories for intercomparison by Atomic Energy of Canada Limited in 1953. At the National Bureau of Standards the Canadian and United States standards were compared by the method of liquid scintillation counting and found to agree to within 1.3 percent. The Canadian standard had been calibrated by the method of gas counting (Hawkins and Merritt, 1954) and that of the United States calorimetrically.

10.3. Carbon-14

Solution standards of this nuclide have been distributed internationally, but the general situation leaves much to be desired. The appropriate method of primary standardization would appear to be that of gas counting, but the disagreements obtained can be matched only with those still extant for its half-life; to which latter disagreement, however, the contributions of mass spectrometry would appear to be not wholly trivial. At the National Bureau of Standards, new and carefully designed compensated gas counters of copper and of stainless steel (figs. 15, 16, 17, and 18) have been constructed with a view to recalibrating its carbon-14 standards (Mann, 1958).

10.4. Sodium-22

Standard samples of sodium-22 have been distributed on an international basis, but to date no intercomparison results are available.

10.5. Sodium-24

Since the inception of the half-yearly distribution of standard solutions of this nuclide by the National Bureau of Standards in the autumn of 1954, samples have been sent regularly to Canada and the United Kingdom for intercomparison. In

spite of its short half-life of only 15 hours, comparisons have been carried out by the methods of $4\pi\beta$ counting and $\beta\gamma$ -coincidence counting, with an over-all agreement of ± 2 percent from the mean value for each distribution. The cooperation of the United Kingdom Supply and Treasury Delegation and Scientific Mission in getting these solution standards to the National Physical Laboratory on the day after dispatch from Washington, D. C., has been an essential factor in the success of these intercomparisons. Normally, zero dates for measurement are set at definite times on the second, third, and fourth days after dispatch from Washington. This is the shortest-lived nuclide used for international comparisons, and it is doubtful whether standards of much shorter-lived nuclides could be conveniently prepared for international or domestic distribution. For individual or special requirements, however, standardizations could probably be carried out with nuclides having half-lives of an order of magnitude less than that of sodium-24, but such standardizations would probably call for simultaneous measurements a day or so after despatch of a sample that is some 10^7 or 10^8 times stronger than is ultimately required for measurement.

10.6. Phosphorus-32

Semiannual intercomparisons of this nuclide with Canada and the United Kingdom have been carried out since February 1950 (Seliger and Schwebel, 1954). Most measurements have been by the method of $4\pi\beta$ counting (in the proportional or Geiger regions), and over-all agreement appears to be settling down to about ± 1 percent from the mean value for each distribution.

10.7. Cobalt-60

Early in 1952, solution standards of this nuclide were made available for international-comparison measurements by the National Bureau of Standards. The results obtained by Canada, the United Kingdom, and the United States by $\beta\gamma$ - and $\gamma\gamma$ -coincidence counting agreed to within ± 1 percent from the mean value (Seliger and Schwebel, 1954). A somewhat larger spread arose, however, in the results obtained by $4\pi\beta$ counting (both proportional and Geiger), and it was subsequently shown (Seliger and Schwebel, 1954) that source self-absorption could cause a decrease in the observed counting rate by as much as 10 percent. By taking the most extreme precautions in source preparation and precipitating the cobalt from solution in the 0.05-ml drop that was placed on the formvar-polystyrene film by means of ammonia, the $4\pi\beta$ -proportional counter gave results that were in agreement with those obtained by coincidence counting (Seliger and Schwebel, 1954). The National Physical Laboratory has also shown, by $4\pi\beta\gamma$ -coincidence counting, that the losses due to source self-absorption in the $4\pi\beta$ counting

of cobalt-60 can be of the order of 5 percent (Perry, 1953). For beta counting, therefore, the solids in solution must be kept low and a minimum of carrier used. It would appear that $4\pi\beta$ counting is not generally to be recommended for radioactive nuclides with beta spectra having maximum energies below 300 kev.

10.8. Zinc-65

A standard solution of this electron-capturing nuclide was sent by the National Bureau of Standards to the Atomic Energy Research Establishment, Harwell, in 1957 for comparison, and agreement was obtained to within 1.5 percent.

10.9. Strontium-90 in Equilibrium With Yttrium-90

Standard solutions of this nuclide were distributed by the Atomic Energy Research Establishment, Harwell, in 1953. By means of $4\pi\beta$ counting, over-all agreement within the limits of ± 2 percent from the mean value was obtained (Seliger and Schwebel, 1954). It was shown, however, at the National Bureau of Standards that in sources prepared for $4\pi\beta$ -proportional counting the source self-absorption for evaporated sources could be as much as 6-percent higher than that for precipitated sources of this nuclide. As the maximum energy of the yttrium-90 beta rays is 2.2 Mev, this is surprising, and emphasizes the precautions that must be taken in source preparation.

10.10. Iodine-131

Semiannual intercomparisons of this nuclide have been made with Canada, or the United Kingdom, or both, ever since December 1949, using the standards prepared for the regular distributions of the National Bureau of Standards (Seliger and Schwebel, 1954). Agreement has been obtained in the determinations of the basic counting rates to within ± 1 percent from the mean value for each distribution. The National Bureau of Standards has, however, made a calculation for the source self-absorption based on shadow electron micrographs of silver-iodide source deposits (Seliger and Schwebel, 1954) (fig. 14). These calculations give a source self-absorption of about 2 percent, and accordingly all National Bureau of Standards calibrations by $4\pi\beta$ -proportional counting are corrected by this amount for source self-absorption. The standardized activity is thus 2-percent higher than that determined by $4\pi\beta$ -proportional counting.

10.11. Gold-198

The first intercomparison of gold-198 was carried out in May 1953 on samples distributed to Canada and the United States by the Atomic Energy Research Establishment, Harwell. Over-

all agreement was not better than ± 4 percent from the mean value, the calibrations being carried out by $4\pi\beta$ -proportional and $4\pi\beta$ -Geiger counting, $\beta\gamma$ -coincidence counting, γ -reference chamber, and β -reference chamber. Subsequently, this agreement has been improved to about ± 2 percent, using the solution standards made available semiannually by the National Bureau of Standards, but in December 1956 an intercomparison was carried out between Atomic Energy of Canada Limited, the National Physical Laboratory (United Kingdom), the National Physical Research Laboratory (Union of South Africa) and the National Bureau of Standards, which supplied the sources, and results were obtained none of which differed by more than ± 1 percent from the mean value.

10.12. Thallium-204

Standard solutions of this nuclide, calibrated by $4\pi\beta$ -proportional counting, have been distributed by the National Bureau of Standards for intercomparison. Some difficulties that may possibly be ascribed to adsorption on the glass or to exchange have, however, been experienced, and work is proceeding currently on the subject. These difficulties may arise from the same source as gives rise to the two groups of quite different half-lives for thallium-204 that appear in the literature.

10.13. Summary

It will thus be seen that, of the original nine nuclides that were proposed for intercomparison at the 1951 Harwell conference, satisfactory agreement has been achieved in the case of six. Two others, namely hydrogen-3 and zinc-65, have also been satisfactorily intercompared.

11. International Organization

As has been noted already, the first meeting of the Subcommittee on Radioactivity Standards of the International Commission on Radiological Units was held in Frankfurt-am-Main on 5 and 6 August 1955. At this meeting it was recognized that, in the field of radioactivity standardization, there were three general groupings of the countries that were then, or were in the future likely to be, interested. These groupings were: (1) Those countries that were producing regularly primary or secondary standards of radioactivity and were in a position to supply other countries with such standards; (2) those countries that, while being in a position to produce their own standards of radioactivity, might yet find it more convenient to obtain standards from countries in group (1); and (3) those countries that would find it difficult to produce primary or secondary standards and would prefer to meet their needs by relying wholly on relative measurements using standards supplied by countries in group (1). It was recognized that the National Bureau of Standards (U. S. A.) and

In addition to the three nuclides still outstanding, the difficult field of electron-capturing nuclide standardization remains relatively untouched from an international point of view. Even on a purely national basis, the attempts to produce radioactivity standards of such nuclides have been somewhat sporadic, and it yet remains to achieve the kind of accuracies and reproducibility of results that have been aimed at in international comparisons. The methods available for calibrating electron-capturing nuclides have recently been reviewed by Allen (1957) who has himself used, as described in his review, high-pressure 4π -Geiger-Müller and 4π -proportional counters to measure the rate of emission of X-rays from electron-capturing nuclides. He did not, however, obtain complete absorption of the X-rays. The use of a proportional counter for the assay of such X-ray-emitting nuclides has also been described by Lyon and Reynolds (1957) while Campion and Merritt (1957) have used a 4π -proportional counter operated with argon and methane up to pressures of approximately 7 atmospheres to measure the rate of X-ray emission from manganese-55 in the decay of iron-55.

Another problem that is presently engaging the attention of the National Bureau of Standards is the production of gamma-ray standards kits with a variety of standards to cover, in suitable intervals, the energy range from 0.1 to 1.0 Mev. These standards, which are required for nuclear-physics research, will probably be issued in the form of disks calibrated in terms of the gamma-ray emission in a 2π geometry, from the surface of the disk on which the source is deposited. It is anticipated, from the interest already shown in them, that these standards will also, in the future, be considered for international comparison.

the National Physical Laboratory (U. K.) were the only laboratories at present preparing and distributing radioactivity standards in quantity, and it was accordingly agreed that the Subcommittee should prepare a combined catalogue of the radioactivity standards available from these two standardizing organizations. This combined catalogue has now been published as appendix III to the National Bureau of Standards Handbook 62 (Report of the International Commission on Radiological Units and Measurements 1956).

It is eventually hoped that other countries will participate in international comparisons of radioactivity standards, and accordingly the directors of the three standardizing laboratories concerned, in the United States, United Kingdom, and in Canada, have, in the closing paragraph of a joint statement (Astin, Bullard, and Lewis, 1956a and 1956b) extended an invitation to those who are interested in such intercomparisons to write to the most conveniently situated of the three directors.

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WASHINGTON, April 1, 1958.

ERRATA TO ACCOMPANY NBS CIRCULAR 594, PREPARATION,
MAINTENANCE, AND APPLICATION OF STANDARDS OF RADIOACTIVITY

Page 28, Column 1, The following should be inserted between lines 7 and
8: with 0.1 g/liter 1,4-di-(2(5-phenyloxazole))-ben-

February 3, 1959
USCOMM-NBS-DC

Comparison of Four National Radium Standards

Part 1. Experimental Procedures and Results

T. I. Davenport, W. B. Mann, C. C. McCraven, and C. C. Smith

Part 2. Statistical Procedures and Survey

W. S. Connor and W. J. Youden

Part 1

The two United States primary radium standards have been compared with the British primary radium standard and the Canadian national radium standard (1) by an ionization method, using the NBS standard electroscop, (2) calorimetrically, using the Peltier-cooling radiation balance, (3) by means of a Geiger-Müller counter, and (4) using a scintillation counter. Where there is little or no difference in gamma-ray source self-absorption, the four methods should, and in fact do, give good agreement. In the case of the Canadian national radium standard the difference in the results obtained is an indication of a difference in source self-absorption.

1. Introduction

During January and February 1954 the British primary radium standard and the Canadian national radium standard were at the National Bureau of Standards for the purpose of comparing these standards with the two United States primary radium standards at the Bureau. The intercomparisons were conducted over a period of 12 days and were made as exhaustive as possible, using the NBS electroscop, a Peltier radiation balance, and Geiger-Müller and scintillation counters.

2. Historical Background

In August 1911 Mme. Pierre Curie prepared, in Paris, a primary radium standard consisting of 21.99 mg of the pure anhydrous radium chloride that had been used to determine the atomic weight of radium as 226.0. This 21.99 mg of radium chloride was sealed into a glass tube 32 mm long, having an internal diameter of 1.45 mm and a wall thickness of 0.27 mm.

At the same time Professor Otto Hönigschmid, in Vienna, made three radium-standard preparations from very pure radium chloride consisting of 10.11, 31.17, and 40.43 mg of radium chloride sealed in glass tubes about 32 mm long, having internal diameters of 3.0 mm and wall thicknesses of 0.27 mm, each tube having a platinum wire sealed in one end. This wire was presumably to prevent the accumulation of static charge within the tubes. The purity of the radium chloride was defined by a radium atomic-weight determination, resulting in a value of 225.97. Of these the 31.17-mg preparation was chosen as a secondary standard. Mme. Curie's 21.99-mg primary standard and Professor Hönigschmid's secondary standard are generally and respectively referred to as the 1911 Paris and Vienna radium standards.

In 1934, after 23 years had elapsed, some concern was felt lest the Paris primary standard, together with a number of secondary radium standards, might explode on account of the accumulation of helium and chlorine and possible devitrification of the containing tubes. Hönigschmid was at that time carrying out, in Munich, a further determination of the atomic weight of radium, and accordingly the International Radium Standards Commission asked him to prepare new standards, using the same salt as for the atomic-weight determination.

For his atomic-weight determination, which was carried out in the early part of 1934, Hönigschmid used approximately 4 g of radium chloride, containing 3 g of radium element, that had been placed at his disposal by the Union Minière du Haut Katanga. This salt was purified by Hönigschmid to a point where spectroscopic analysis by Gerlach showed a maximum of 0.002 to 0.003 percent of barium atoms. A value was obtained for the atomic weight of radium equal to 226.05, which is currently accepted.

Hönigschmid then used some 817 mg of this highly purified anhydrous radium chloride to prepare 20 new standards of radium. Exactly who asked him to do this is not now quite clear. According to Mlle. Chamié [1],¹ the International Radium Standards Commission, at the suggestion of Stefan Meyer, "entrusted Mr. O. Hönigschmid with the preparation of 20 standards, using the salt he had purified and used in measuring the atomic weight of radium." According to Hönigschmid himself, however, in a paper [2] presented after his death by Stefan Meyer, the 20 standards were prepared "at the wish of the Belgian radium company." These two versions are, however, not irreconcilable if one assumes that the suggestion of the Belgian company was made known to the International Radium Standards Commission, which then gave it its official sanction.

Figures in brackets indicate the literature references on page 272.

The 20 new Hönigschmid standards were sealed into glass tubes on June 2, 1934, the glass tubing being similar to that used to seal the 1911 Vienna standard and having an internal diameter of 3.0 mm and a wall thickness of 0.27 mm. A platinum wire was sealed into the end of each standard.

One of the new Hönigschmid standards that was 42 mm long and contained 22.23 mg of radium chloride was selected as the new international standard, and its value was carefully compared with the 1911 Paris standard by gamma-ray measurements over a period of 4 years [1]. The Hönigschmid reference number for this standard is 5430. Hönigschmid states [2] that the error of a single weighing was not more than 0.02 mg. The gamma-ray comparison with the 1911 Paris standard showed a discrepancy, however, of 0.2 percent, corresponding to a weight of 22.27 mg as of June 2, 1934.

The first United States radium standard was brought to America in 1913 by Mme. Curie. This source contained 20.28 mg of radium chloride and was designated by the International Radium Standards Commission number IV (Vienna No. 6).

In 1936 two of the twenty Hönigschmid preparations were acquired as the United States primary radium standards. They are each designated by two numbers, namely, 5437, XIV and 5440, XV. The arabic numerals are those given by Hönigschmid, and the roman numerals are those assigned by the International Radium Standards Commission and imply that the standards have undergone gamma-ray comparison with the 1911 Paris and Vienna standards. The lengths of these two United States standards are 36 and 37 mm, and they contained 50.22 and 26.86 mg, respectively, of radium chloride as weighed by Hönigschmid on June 2, 1934. These weights correspond to 38.23 and 20.45 mg of radium element. The weights derived from a comparison with the Paris and Vienna 1911 standards corresponded, however, to only 38.13 and 20.38 mg, respectively, of radium element, as of June 1934.

The British primary radium standard is designated by one number only, namely, 5432. It is solely a standard by weight and was not compared with the 1911 Paris and Vienna standards. It is, however, one of the original Hönigschmid preparations sealed on June 2, 1934. Its length is 38.8 mm, and its salt content corresponds to 15.60 mg of radium element, as of that date. This standard replaced the first British radium standard, which had been in the custody of the National Physical Laboratory since 1913. This earlier standard was designated by the International Radium Standards Commission number III (Vienna No. 3).

The United States and British primary radium standards, as can be seen from figure 1, have low ratios of volume of salt to volume of tube. It is therefore to be expected that with the standards in a horizontal position and the grains of radium chloride distributed evenly along the tube their gamma-ray source self-absorption would be very nearly the same.

The Canadian national radium standard is however shorter and of smaller diameter than the Hönig-

schmid preparations, and it is tightly packed (fig. 1). It was sealed in June 1930 by the Union Minière du Haut Katanga, its contents, and that of six other sources in the custody of the National Research Council, having been taken from two tubes of radium chloride that had been prepared by the Union Minière in June 1924. Its weight was derived by gamma-ray comparison in 1933, in Paris and Vienna, with the 1911 standards, and it is designated by the number XIII. It is understood that no corrections for possible differences in self-absorption were made in these gamma-ray comparisons. Its length is 10.5 mm, its internal diameter 1.5 mm, and its salt content corresponds, according to the gamma-ray comparison with the 1911 radium standards, to 24.23 mg of radium element, as of June 1934. Information on all four national standards is summarized in table 1.

TABLE 1. Description of four national radium standards

	A ¹ U. S. primary radium standard	B British primary radium standard	C Canadian national radium standard	D U. S. primary radium standard
Reference numbers.....	5437, XIV	5432	XIII	5440, XV
Radium content as given by:				
1. Hönigschmid's weighings.	38.23 mg	15.60 mg	-----	20.45 mg
2. Comparison with Paris and Vi- enna 1911 stand- ards, as of June 1934.....	38.13	-----	24.23 mg	20.38 mg
Length of glass tube...	36 mm	38.8 mm	10.5 mm	37 mm
Internal diameter of tube.....	3 mm	3 mm	1.5 mm	3 mm
Tube wall thickness...	0.27 mm	0.27 mm	0.25 mm	0.27 mm

¹ For convenience, A, B, C, and D are used here and elsewhere in this paper to identify these radium standards.

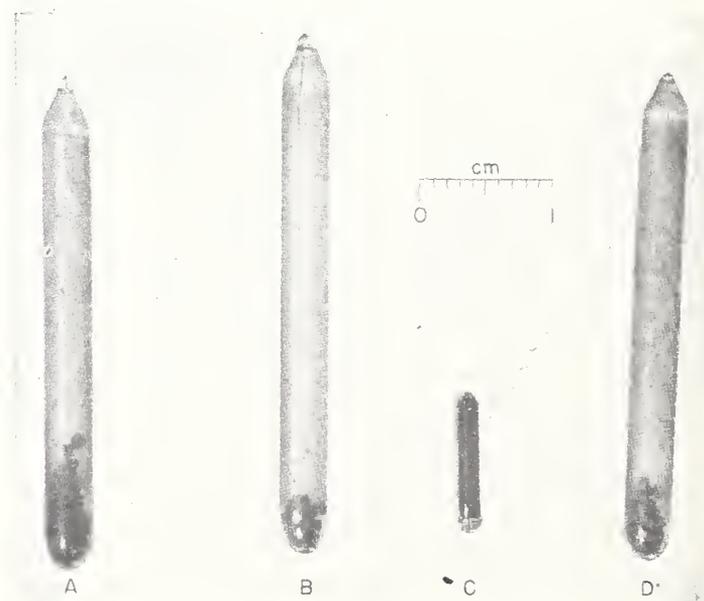


FIGURE 1. Four national radium standards.

A, American; B, British; C, Canadian; and D, American.

In view of the uncertainties that exist and the differences between the Hönigschmid weights and their weights as derived by comparison with the 1911 Paris and Vienna standards [3], it has recently been suggested that new radium standards be prepared from about 1 g remaining of Hönigschmid's original "atomic-weight" material. Another possibility lay in a recheck of the present standards. With this end in view, United States primary radium standard 5440, XV was taken to the United Kingdom in the summer of 1952 and to Canada in the autumn of the same year. At the National Physical Laboratory, in Teddington, and at the National Research Council Laboratories, in Ottawa, it was compared, by gamma radiation, with the British primary radium standard, 5432 [4] and the Canadian national radium standard, XIII [5]. The results obtained by these laboratories are discussed later in connection with the data given in table 3.

The question also arises as to what is desired in a radium standard. In order to derive the mass of any radium preparation in terms of the standard by gamma-ray measurements it is necessary to know both the absorption of the containers of the preparation and standard and also the self-absorption of the radium salts themselves. In NBS certificates the results are stated in terms of milligrams of radium when contained in a Thüringen glass tube having a wall thickness of 0.27 mm, together with an empirical absorption correction for the container in question. Only calorimetric measurements can give the ratios of the true radium contents, irrespective of absorption but in this case it is necessary to know the date of sealing of the preparation in order that correction may be made for the growth of polonium. A small fraction of the gamma-ray energy is absorbed and measured by the calorimeter, but any difference in absorption between two sources will represent only a small correction to the already small contribution of gamma-ray energy emission (about 7%) to the total energy emission.

3. Measurements With the NBS Standard Electroscopie

The NBS standard electroscopie [6] and measuring system were used, without modification, for this comparison of four national radium standards. The ionization chamber consists of a 10-cm cube free-air volume, with walls made of 1 cm of lead and a ½-cm aluminum inner lining. A gold leaf is suspended near the center of the chamber. A 10- μ quartz fiber at the free end of the leaf provides a fine line for projection. The fiber image is magnified approximately 100 times and projected onto a metric scale. The discharge of the electroscopie is measured by timing the transit of the image between two fixed points on the scale 6 cm apart.

The source indexing system consists of a V-shaped trough of ½-in. Lucite on an aluminum stand. The stand can be moved along a line perpendicular to the face of the ionization chamber or rotated about its own vertical axis. Preparations are centered in the trough opposite the center of the

chamber, so that measurements are made perpendicular to the axes of symmetry of the preparations.

The four standards were measured relative to each other by comparison of each of the six possible combinations of pairs. Independent measurements were made on each pair by each of three different observers at source distances of 66.5 cm and 74.1 cm from the chamber. The entire series of measurements was repeated twice.

The following procedure was adopted for comparing each pair of standards:

1. The trough was placed at the distance selected and parallel to the chamber face.
2. A standard was held horizontally and tapped lightly until the salt was distributed uniformly along the length of the capsule, as in figure 2.
3. The standard was placed in the trough and centered.
4. Three observations of the discharge time were made and recorded.
5. The trough was rotated 180 degrees, and three more observations were made.
6. Procedures 1 to 5 were repeated with the second standard of the pair.
7. Procedures 1 to 6 were repeated for both members of the pair at the second distance from the electroscopie.

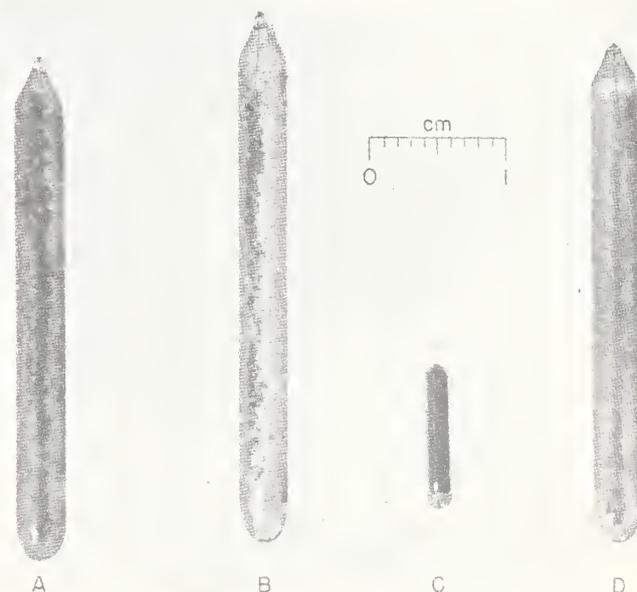


FIGURE 2. Four national radium standards, with the grains of salt in the three Hönigschmid standards distributed along the length of the tubes.

A, American; B, British; C, Canadian; and D, American.

4. Comparison by Geiger-Müller Counter

The Geiger-Müller counter used for this comparison was a neon-halogen-filled tube. The tube itself was surrounded by a sheath of lead ¼ inch thick so that the soft gamma rays, the spectrum of which might be varied by source absorption to a greater extent than that of the higher-energy gamma rays, would not be counted. The resolving time of the counter was determined by the two-source method to be 211 μ sec \pm 5 percent. The correction for re-

solving time applied to the data ranged from 1.1 to 2.7 percent.

The source holder of the NBS standard electro-scope was used to position each standard in turn in these measurements, and the standards were tapped so that, in the case of the more loosely packed Hönigschmid standards, the grains would be distributed uniformly along the tube.

In order to eliminate any possible effects due to drift a series of measurements was carried out on each pair of international standards. Thus, in the comparison of *A* and *B*, measurements were carried out with *A* and *B* arranged in "packages" in the following order: *A, B, B, A; B, A, A, B; A, B, B, A; and finally, B, A, A, B*. Similar package measurements were made on each of the other five pairings of the four international standards.

A total of about 80,000 counts was taken on each of the 16 members of the 4 packages comprising a pair comparison. Thus in the comparison of *A* and *B* a total of some 640,000 counts were made with *A* in position and 640,000 with *B*.

5. Comparison by Scintillation Counter

The scintillation counter consisted of a thallium-activated sodium-iodide crystal mounted on the face of a photomultiplier tube. The resolving time of the counter and amplifier was $5 \mu\text{sec} \pm 10$ percent, and the corrections applied to the data varied from 0.3 to 0.8 percent. The discriminator was set to accept pulses corresponding to gamma-ray energies greater than 1 Mev. Thus, as for the NBS standard electro-scope and the Geiger-Müller counter, the effect of source self-absorption of the lower-energy gamma rays should not be apparent. The sodium-iodide crystal and photomultiplier were mounted adjacent to the Geiger-Müller counter so that counts on each source could proceed concurrently with both counting systems. Exactly the same pairing and packaging order of sources as was used for the Geiger-Müller counter comparison was, ipso facto, also used in the scintillation-counter measurements. The counts for each source in position were of the order of 400,000 compared with 80,000 in the case of the Geiger-Müller counter.

6. Measurements With the Radiation Balance

A modification of the radio-balance originally designed by Callendar [7] for the measurement primarily of radiant energy has recently been described [8], which is suitable for the measurement of the energy emission from radioactive materials. This modification of the radio-balance has been renamed the radiation balance, its most important feature being the ability to balance the energy emission from a radioactive source either against Peltier cooling or the energy emission from another radioactive source, or both.

None of the radiation balances constructed previously was large enough to accommodate the large Hönigschmid standards, and, accordingly, a new one was constructed for this purpose. This balance is described in detail separately in this issue [9]. It

differed essentially from the first one, however, in that its larger cups were made from gold instead of copper.

7. Radiations Measured

The radiation from radium in equilibrium with all its products consists of five energetic alpha-particle groups, including that of polonium; three main groups of beta particles, the most energetic being that from the transition of radium *E* to radium *F* with a maximum energy of 1.17 Mev; and a complexity of gamma rays, the most energetic being from the excited levels of radium *C'*.

Three of the methods described here and used to compare the radium contents of the four national radium standards were essentially gamma-ray comparisons. With the thicknesses of lead used, or the discriminator setting, the chief contribution to the gamma-ray effect would be from the energetic radium *C'* gamma rays (above 0.6 Mev in the case of the electro-scope and Geiger-Müller counter and above 1 Mev in the case of the scintillation counter).

In contrast, the radiation balance measures primarily the energy emitted in corpuscular form. Some 93 percent of the energy produced by radium and its daughters down to radium *D* is associated with particulate emission, the remaining 7 percent of the energy produced being associated with the gamma radiation. The wall thickness of the gold cups was such as to absorb completely the most energetic beta particles from radium *E*. Some 12 percent of the energy associated with the gamma rays is also absorbed. Of the 7 percent of the total energy produced that is associated with the gamma-ray emission, another 1 percent (for the Canadian standard) or 1.5 percent (for the Hönigschmid standards), corresponding, respectively, to 0.07 and 0.1 percent of the total energy produced, will be absorbed in the sources themselves. The difference of 0.5 percent between the source self-absorption of the Canadian and Hönigschmid standards corresponds therefore to a difference of only 0.035 percent of the total energy produced, which is negligible. Any smaller differences in gamma-ray source self-absorption of the three Hönigschmid standards are also therefore negligible so far as the measurements in the radiation balance are concerned. The alpha-particle and beta-particle absorption is complete; a correction must be made, however, for the growth of radium *E* and polonium, which will not be in equilibrium with the radium.

8. Results

The results obtained with the radiation balance, measuring the sources singly and in every combination of pairs, are summarized in table 2. In this table the order of measurement is represented by reading from left to right and down the table.

From the results in table 2 the following best estimates for the energy absorbed (in microwatts) from sources *A, B, C, and D* have been deduced:

<i>A</i>	<i>B</i>	<i>C</i>	<i>D</i>
6293.4	2569.8	4131.0	3360.7

TABLE 2. Energy absorption, in microwatts

Source	Energy absorbed						
B.....	2571.0	A-B..	3727.1	D-B..	788.6	C-B..	1561.2
C.....	4127.1	C-D..	776.0				
D.....	3371.6	A-D..	2935.6				
A.....	6285.2	A-C..	2164.2				

In table 3 are shown the complete results for the six pairs of standards, using the NBS standard electroscop, Geiger-Müller counter, scintillation counter, and radiation balance. In the last line of the table are shown the weight ratios for the same six pairs. The weight of the Canadian standard (C) is, however, only a derived weight, and for this reason, any ratio involving this derived weight is shown in quotation marks. The ratios A/B, A/D, and B/D are, however, the ratios of Hönigschmid's own weighings.

TABLE 3. Adjusted results for the ratios of the four international standards

Method	A/B	A/C	A/D	B/C	B/D	C/D
NBS standard electroscop.....	2.441	1.570	1.870	0.6429	0.7661	1.192
Geiger-Müller counter.....	2.461	1.582	1.885	.6430	.7659	1.191
Scintillation counter.....	2.478	1.579	1.889	.6370	.7624	1.197
Radiation Balance.....	2.449	1.523	1.873	.6220	.7647	1.229
Weighing.....	2.451	"1.578"	1.869	"0.6438"	.7628	"1.185"

For comparison with these values the ratios obtained by Perry [4], using the NPL standard ionization chamber with gold-leaf electroscop, an ionization chamber with a Lindemann electrometer, and a Geiger-Müller counter for B/D were, respectively, 0.7669, 0.7657, and 0.7669. The result obtained for the gamma-ray ratio C/D by Michel [5], using the NRC precision ion chamber and Lindemann electrometer was 1.192. Michel, from geometrical considerations, then calculated the source absorption of each standard and corrected the gamma-ray ratio to give a weight, or content, ratio of C/D equal to 1.185. The direct gamma-ray ratios obtained both by Perry and Michel are in excellent agreement with the results shown in table 3.

A check on the internal consistency of the results shown in table 3 can be provided by assuming that A, B, and D are so much alike that there are negligible differences in source absorption for high-energy gamma rays, and none at all in the case of the calorimeter, where 93 percent of the energy absorbed is particulate, so that any change due to absorption of the 7 percent of gamma rays and secondary electrons would be even more negligible. A check can then be run on the results for A, B, and D by dividing the quantity characteristic of each in each determination by the Hönigschmid weight of each standard. This characteristic quantity is scale divisions per second for the NBS standard electroscop, counts per second for the counters, and microwatts

for the radiation balance. In each case the characteristic quantity is as of February 1954, and the mass of radium element is as of June 1934. It is not necessary for this check to correct for the 20-year decay of radium as this is the same constant for each standard.

The results of this internal precision check are shown in table 4, in which the figures quoted are the characteristic quantity, divisions, or counts per second or microwatts, divided by the mass of radium element present and normalized to make the "best average" equal to 100.00 in each case.

TABLE 4. Radioactive effect per milligram of radium element (Normalized to make the best average equal to 100.00)

Method	A	B	D	Best average	Standard deviation of the individual results
NBS standard electroscop.....	99.93	100.31	99.88	100.00	0.23
Geiger-Müller counter.....	100.31	99.90	99.51	100.00	.40
Scintillation counter.....	100.52	99.42	99.47	100.00	.62
Radiation balance.....	100.03	100.10	99.86	100.00	.13

The values of the best average should, in turn, enable one to form an estimate of the precision of Hönigschmid's weight determinations, in which, according to Hönigschmid himself [2], the error of a single weighing was not more than 0.02 mg. A statistical survey of the results was carried out with the cooperation of W. S. Connor and W. J. Youden, and resulted in the best estimates of the mass of radium element in A, B, and D given in table 5. The methods adopted to arrive at these best estimates, and also the best estimates given in table 3 for the ratios of pairs of standards, are described by Connor and Youden in part 2 of this paper.

TABLE 5. Best estimates, in milligrams, of the masses of the Hönigschmid radium standards, as of June 2, 1934

Standard	A	B	D
Hönigschmid's mass.....	38.23	15.60	20.45
Mass derived from NBS standard electroscop.....	38.227	15.611	20.446
Mass derived from Geiger-Müller counter.....	38.235	15.598	20.443
Mass derived from scintillation counter.....	38.235	15.595	20.444
Mass derived from the radiation balance.....	38.235	15.608	20.435

9. Mass of Radium Element in the Canadian National Standard

By comparing the calorimetric ratios given in table 3 with the "weight" ratios, it is clear that the derived weight of the Canadian national radium standard (C) is low by about 3 percent. However, this does not allow for the difference in sealing date,

which involves a compensating polonium-growth correction of about 1.8 percent. By comparison of the "weight" ratios with the NBS standard electro-scope ratios, it is also confirmed that no source self-absorption correction could have been made in deriving the certified weight of radium in the Canadian standard. However, from the data available it is possible to derive a value for this mass of radium.

The experimentally determined ratios of the energy absorbed in the radiation-balance cups per unit mass of radium element for *A*, *B*, and *D* are 164.62, 164.73, and 164.34 $\mu\text{w}/\text{mg}$, respectively. Taking the best average value of 164.58 $\mu\text{w}/\text{mg}$ of radium element, the mass of the radium in the Canadian national radium standard is found to be equal to 25.10 mg, as of June 1934, uncorrected for the growth of polonium or of radium E.

Using the Curie-Yovanovitch equation, as corrected for new values of the decay constants by Jordan [8, 10], the energy increments due to growth of polonium-210 in *A*, *B*, and *D*, on the one hand, and in *C*, on the other, are found to be equal, respectively, to 12.2 and 16.2 $\text{cal g}^{-1} \text{hr}^{-1}$ inclusive, of nuclear recoil energy, the separation and sealing dates being, respectively, May 25, 1934, and June 2, 1934, for the Hönigschmid standards, and June 1924 and June 1930 for the Canadian national standard. The growth of radium E will contribute, in proportion, another 0.8 and 1.0 $\text{cal g}^{-1} \text{hr}^{-1}$. Subtracting the contributions of polonium-210 and nuclear recoils and of radium E from the energy absorbed from *A*, *B*, and *D* in the radiation-balance cups gives a total energy absorption for all three sources equal to 11103.0 instead of 12223.9 μw (as of February 1954).

In the case of the Canadian national standard, an energy production of 17.2 $\text{cal g}^{-1} \text{hr}^{-1}$ by polonium-210 and radium E corresponds to 20.0 $\mu\text{w}/\text{mg}$ of radium element, which, by a second approximation, is found to be equivalent to 489.7 $\mu\text{w}/24.48_5$ mg of radium element (the mass of radium as of June 1924). The corrected energy absorption from the Canadian national radium standard is therefore 3641.3 instead of 4131.0 μw , as of February 1954. The radium content of the Canadian national radium standard, as of June 1934, is then obtained by multiplying the total weight of the Hönigschmid standards (76.28 mg as of June 1934) by the ratio of the corrected energy absorptions of February 1954. This gives the result that there were 24.36 mg of radium element in the Canadian national standard, as of June 1934. This value will, if anything, be on the low side, however, as some radium D on the walls of the original two tubes may have been lost on transfer when the Canadian standard was resealed in June 1930. In this event, the polonium-210 correction will have been too great.

10. Summary of Results

As a result of this intercomparison of national radium standards, the ratios of the weights ascribed to three of them by Hönigschmid have been confirmed. It would appear that the weights derived

from the comparison of the two United States standards with the 1911 Paris and Vienna standards are, therefore, too low; unless it were assumed that all of Hönigschmid's mass determinations were low in the same ratio. However, this is to be discounted because the Berlin standard was, by comparison with the 1911 standards, found to have a greater weight than that determined by Hönigschmid [1].

Relative to the Hönigschmid weights, the Canadian national radium standard is found to have a mass of radium element equal to 24.36 mg, which indicates that no correction for difference in source self-absorption was made in its comparison with the 1911 Paris and Vienna standards. The difference between this value and that obtained by comparison with the 1911 Paris and Vienna standards (24.23 mg as of June 1934) would indicate a self-absorption correction of 0.53 percent. The absorption correction determined by Michel [4] was 0.94 percent; the difference between these two values could be a measure of the loss of radium D and polonium-210 in the transfer of June 1930.

Most grateful acknowledgments are made to the following: The Director of the National Physical Laboratory and the President of the National Research Council, for the loan of the British primary radium standard and the Canadian national standard; to W. E. Perry and W. S. Michel, respectively, for their helpful cooperation and for the transportation to Washington, D. C., of these standards; to W. J. Youden, for many helpful and most valuable discussions on the planning of the experiments, and to him and W. S. Connor, Jr., for discussion of the final results; to H. H. Seliger for providing the Geiger-Müller and scintillation counter equipment and for advice on its operation; and to L. F. Paoletta for valuable assistance in carrying out the readings on the NBS standard electro-scope and the Geiger-Müller and scintillation counters.

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Part 2. Statistical Procedures and Survey

W. S. Connor and W. J. Youden

The statistical analysis of the observations on the four national radium standards is discussed. The readings made with the electroscopes, Geiger-Müller counter, and scintillation counter were adjusted by one formula, and the readings made with the radiation balance by a different formula. In each case the adjusted values of the standards satisfy a consistency criterion. Finally, the adjusted values were improved by making use of the proportional relationship between the masses and the radioactive effects of the standards.

1. Introduction

Four national radium standards were recently compared at the National Bureau of Standards, as described in part 1 of this paper. The unusual opportunity associated with the presence of four standards in one laboratory directed attention to certain statistical aspects of the intercomparison. The experimental procedures and results are described in part 1. Part 2 discusses the statistical analysis.

When two standards are compared, careful measurements provide an estimate for the value of one standard in terms of the other. A standard error may be calculated for this estimate. A third standard makes possible the additional experimental evaluation of each of the first two standards in terms of the third.

Suppose that three standards A , B , and C are available. The experimental ratios a/b , b/c , c/a may each be determined by using exactly the procedure that would have been employed if just two standards had been available. None of the measurements made on A in estimating a/b are used in the estimation of c/a . Additional data for A are taken to determine c/a . There is a considerable advantage in this method because the precision of the comparison is improved by alternating the readings on the two standards under comparison. This alternation reduces the effects of drift in the instruments and changes in the environment. As soon as the ratios a/b , b/c , c/a have been determined there is a simple test for the consistency of the three ratios. The product of the three ratios should be unity. The discrepancy between this product and unity provides a measure of the errors in these ratios.

A similar consistency criterion was applied to the six ratios determined by the electroscopes, Geiger-Müller counter, and scintillation counter. Because a different statistical treatment was required for the measurements made with the radiation balance, those measurements are discussed separately.

The last section describes how the masses of the standards were used further to improve the estimates of the standards.

2. Comparison of the Standards by Means of Electroscopes, Geiger-Müller Counter, and Scintillation Counter

Using these methods, environmental conditions common to paired measurements introduce a common multiplicative error in the measurements. It is ad-

vantageous to express the results of paired measurements as ratios to eliminate this error.

There were four standards, A , B , C , and D . Therefore, the following six ratios could be determined experimentally:

$$a/b \quad a/c \quad a/d \quad b/c \quad b/d \quad c/d.$$

These provide opportunities to test the consistency of the data. For example, the products

$$a/b \times b/c \times c/a$$

$$a/b \times b/d \times d/a$$

$$a/c \times c/d \times d/a$$

$$b/c \times c/d \times d/b$$

should all be equal to unity. The discrepancies between these products and 1.0000 reveal the errors of the measurements. It is proper to make use of the information that the products should be exactly equal to one. The measured ratios may be adjusted by a least-squares technique to obtain new ratios \hat{A}/\hat{B} , \hat{A}/\hat{C} , etc., which do in fact multiply out to unity for all combinations that should give unity. This includes not only three factor combinations such as

$$\hat{A}/\hat{B} \times \hat{B}/\hat{C} \times \hat{C}/\hat{A}$$

but also four factor products

$$\hat{A}/\hat{B} \times \hat{B}/\hat{C} \times \hat{C}/\hat{D} \times \hat{D}/\hat{A}.$$

The adjustment formula used on the data shown in table I is of the form.

$$\frac{\hat{A}}{\hat{B}} = \sqrt[4]{\left(\frac{a}{b}\right)^2 \left(\frac{a}{c} \times \frac{c}{b}\right) \left(\frac{a}{d} \times \frac{d}{b}\right)},$$

where the lower case letters indicate the measured ratios.¹ The adjusted values (see table 3 in part 1)

¹ This adjustment formula is related to the adjustment formula for the difference between the estimates of two treatment effects in a balanced incomplete block (BIB) design, see R. L. Anderson and T. A. Bancroft, *Statistical theory in research*, p. 252 (McGraw-Hill Book Co., Inc., New York, N. Y., 1952). Since the two measurements in a pair, as a and b or c and d , are subject to a common multiplicative error, the logarithms of the two measurements in the pair are subject to a common additive error. Hence, the BIB design formula applies for the difference between the logarithms of the adjusted values, as $\log \hat{A} - \log \hat{B}$, and by taking antilogarithms, the above formula is obtained.

have the property that

$$\frac{\hat{A}}{\hat{B}} = \frac{\hat{A}}{\hat{C}} \times \frac{\hat{C}}{\hat{B}} = \frac{\hat{A}}{\hat{D}} \times \frac{\hat{D}}{\hat{B}}$$

The observed values do not meet this consistency requirement. The reconciliation among the results effected by the above least-squares technique introduces each standard symmetrically in the computation pattern and does not single out any one standard as a superstandard. After the relative values have been established, one standard may be given an agreed value, whereupon all other standards are determined without changing the relative values.

TABLE 1. *Experimental results for the ratios of four standards*

Method	a/b	a/c	a/d	b/c	b/d	c/d
Electroscope.....	2.4438	1.5675	1.8703	0.64246	0.76650	1.1918
Geiger-Müller counter..	2.4746	1.5785	1.8784	.64489	.76789	1.1920
Scintillation.....	2.4847	1.5710	1.8930	.63921	.76186	1.1953

The above least-squares adjustment has long been used for other comparisons. Recently, it has been found that certain subsets of pairs selected from all possible pairs lead to convenient least-squares estimates.² Given that a reasonably small number of pairs will suffice to interrelate all the standards, there would appear to be some chance of success for an international program of comparison. Once a properly selected subset of pairings was obtained, the various national standards could be tied together with values that would give consistent comparisons among the standards.

3. Radiation-Balance Measurements

The radiation balance used in this work was suitable for measuring either a proportion of the energy emitted by one standard or the same proportion of the difference in energies emitted from two standards. This difference is determined by one measurement. The schedule of measurements included separate measurement on the four standards as well as the six possible differences between them. The precision of measurement of a difference was the same as the precision of measurement of a single standard.

Typical formulas for the least-squares estimates³ for the 10 quantities follow:

$$\hat{A} = \frac{2}{3}a + \frac{1}{3}[(a-b) + b] + \frac{1}{3}[(a-c) + c] + \frac{1}{3}[(a-d) + d]$$

$$\begin{aligned} (\hat{A} - \hat{B}) = & \frac{2}{3}(a-b) + \frac{1}{3}[(a-c) + (c-b)] \\ & + \frac{1}{3}[(a-d) + (d-b)] + \frac{1}{3}a - \frac{1}{3}b. \end{aligned}$$

² W. J. Youden and W. S. Connor, Making one measurement do the work of two, *Chem. Eng. Progr.* **49**, 549 (1953); and W. J. Youden and W. S. Connor, New experimental designs for paired observations, *J. Research NBS* **53**, (1954) RP2532.

³ For a discussion of the method of least squares, see R. L. Anderson and T. A. Bancroft, *Statistical theory in research*, p. 155 (McGraw-Hill Book Co., Inc., New York, N. Y., 1952).

The quantities a , b , $(a-b)$, etc., are measured quantities. The value for $(\hat{A} - \hat{B})$ given by the above formula will agree exactly with the result obtained by subtracting the adjusted estimate \hat{B} from the adjusted estimate \hat{A} . This was not true for the recorded values. The total amount of energy measured for the standards is left unaltered by the adjustment. Slight shifts take place in a , b , c , d , $(a-b)$, etc., to achieve consistency among the results. The discrepancies between the measured quantities and the corresponding adjusted values afford a measure of the precision of the measurements. The calculation is shown in table 2. It should be noted that no quantity was measured twice. The replication is concealed. There are, of course, only four standards; that is, four quantities to be determined from the ten observations. This leaves six contrasts, i. e., six degrees of freedom, available for estimating the standard deviation.

TABLE 2. *Calculation of standard deviation, in microwatts, for radiation balance*

Standard	Observed	Adjusted	Difference	(Difference) ²
	μw	μw	μw	μw
A-----	6285.2	6293.4	8.2	67.24
B-----	2571.0	2569.8	1.2	1.44
C-----	4127.1	4131.0	3.9	15.21
D-----	3371.6	3360.7	10.9	118.81
A-B----	3727.1	3723.6	3.5	12.25
A-C----	2164.2	2162.4	1.8	3.24
A-D----	2935.6	2932.7	2.9	8.41
C-B----	1561.2	1561.2	0.0	0.00
C-D----	776.0	770.3	5.7	32.49
D-B----	788.6	790.9	2.3	5.29

Standard deviation = $\sqrt{\frac{264.38}{6}} = 6.6\mu w$.

4. Masses of the Radium Standards

Standards A , B , and D were made from the same supply of radium salt. The weighings were made in the same day by Hönigschmid and are considered to have a maximum error of 0.02 mg. The various properties of the three Hönigschmid standards measured by the several methods used in this intercomparison are believed to be directly proportional to the masses of the standards. All the methods give relative values for the standards. In addition, the radiation balance measures the difference between any two standards directly. Standard D was arbitrarily given the value of unity and the values for A and B expressed relative to it. Table 3 contains some of the adjusted ratios from table 3 of part 1, including the ratios derived from Hönigschmid's weighings.

TABLE 3. Value of standard, when D equals 1.000

Method	A	B	D
Scintillation counter	1. 889	0. 7624	1. 000
Geiger-Müller counter	1. 885	. 7659	1. 000
NBS standard electro- scope	1. 870	. 7661	1. 000
Radiation balance	1. 873	. 7647	1. 000
Weighing	1. 869	. 7628	1. 000

For each method of measurement a plot may be made of the values of the standards against the corresponding masses. The resulting points should lie along a straight line that passes through the origin. Let m denote the mass and r the radioactive effect given by any one method of measurement in the relative units of table 3. If the error in r is k times as large as the error in m (as measured by the standard deviations), the slope b may be computed from the quadratic

$$b^2 + \frac{k^2 \sum m_i^2 - \sum r_i^2}{\sum m_i r_i} b - k^2 = 0,$$

where m_i and r_i ($i=A, B, D$) are the masses and the corresponding radioactive effects for standards A, B , and D from table 3.⁴ This method of determining b has the property that the sum of the squares of the perpendicular distances of the points (km_i, r_i) from the line $r=b(km)$ is minimized.

⁴ For a discussion of this method, see W. Edwards Deming, Statistical adjustment of data, Exercise 6, 184 (John Wiley & Sons, Inc., New York, N. Y., Nov. 1944).

In general, any particular plotted point will not be located exactly on the fitted line. The plotted points are subject to errors of observation. The "best" estimates of the coordinates for the point are taken to be the coordinates of the point on the line nearest to the plotted point. These coordinates, m'_i and r'_i , are

$$m'_i = \frac{m_i + br_i}{1 + b^2} \quad \text{and} \quad r'_i = b \frac{m_i + br_i}{1 + b^2}.$$

This procedure for fitting lines was followed for each of the lines relating the measured radioactive property to the mass. For the electro-
scope, Geiger-Müller counter, scintillation counter, and radiation balance, k was taken as 2, 4, 5, and 1, respectively. These values correspond to the errors given by table 4 of part 1, except for the scintillation counter, for which 5 was used instead of 6.

To obtain estimates for the masses, each value of m'_i was multiplied by Hönigschmid's value for D , i. e., 20.45 mg. These estimates are recorded in table 5 of part 1. In every case the result agreed with the assigned mass within the claimed weighing error. It is particularly interesting to observe that the estimates obtained from the line, using the radiation balance results, confirm the assigned masses. For this line the errors in m and r were taken to be the same, and therefore any displacement of the point to bring it on the line required equal changes in the experimental values for mass and energy.

WASHINGTON, May 27, 1954.

Comparisons of National Radium Standards

T. P. Loftus, W. B. Mann, L. F. Paoella, L. L. Stockmann, and W. J. Youden

The national primary radium standards of the Federal Republic of Germany and the Dominion of Canada have recently been compared with the two United States national primary radium standards at the National Bureau of Standards. The comparisons were made using the standard electroscope and radiation balance, and the results obtained for these four Hönigschmid standards have been compared with those obtained in an earlier comparison of the United States primary radium standards with that of the United Kingdom, which is also a Hönigschmid standard. In every case, agreement with Hönigschmid's values to within 0.2 percent has been obtained.

1. Introduction

During January and February 1954 the British primary radium standard and the Canadian national radium standard were compared with the United States primary radium standards at the National Bureau of Standards [1,2].¹ The British and United States standards were Hönigschmid standards, whereas the Canadian standard consisted of radium chloride sealed in a glass tube of considerably smaller dimensions than those of the Hönigschmid standards and therefore considerably more closely packed [1]. In any gamma-ray comparison between this Canadian standard and a Hönigschmid standard, it is therefore necessary to apply corrections for self-absorption of the source [3]. In 1955 the National Research Council of Canada procured a Hönigschmid standard (No. 5425), to replace the older preparation, as the primary radium standard of Canada [4]. This new Hönigschmid standard was compared during the summer of 1955 with the German (No. 5426) and British (No. 5432) Hönigschmid standards in Braunschweig and Teddington, respectively, and was then brought, in early December 1955, to Washington, D. C., for comparison with the United States Hönigschmid standards (Nos. 5437 and 5440) at the Bureau.

In November 1955 the Hönigschmid standard of the Physikalisch-Technische Bundesanstalt was also compared with the two United States standards.

The results of the measurements carried out on the Canadian, German, and United States Hönigschmid standards are given in this paper, together with a reassessment of the measurements previously carried out at the Bureau on the British and United States Hönigschmid standards [1,2].

2. Methods of Measurement

For the comparisons of the Canadian and the German standards with the United States standards, only the NBS standard electroscope [5] and Peltier-effect microcalorimeter, or radiation balance [6,7], were used. These have already been demonstrated to give precise and reproducible results and consequently the counting methods, previously utilized [1], were not again employed to supplement the measurements of the electroscope and microcalorimeter. The procedures of the earlier comparison for

both these methods have already been fully described [1] and were again followed precisely.

A new and very careful determination of the thermoelectric power (dE/dT) of the Peltier couples of the radiation balance was carried out, using a pair of copper-sheathed heating and compensating resistance coils whose difference in resistance was precisely measured. The results of a great many measurements gave a new average value at 25° C of dE/dT equal to 58.71 $\mu\text{V}/\text{deg}$, as compared with 58.78 $\mu\text{V}/\text{deg}$ used in the earlier work [2]. This new value will not affect the ratios of the standards as then determined, but will give a different value for the absolute rates of energy emission for the different radium standards. For the purpose of comparison with the results obtained in the Canadian and German intercomparison, those for the British intercomparison have therefore

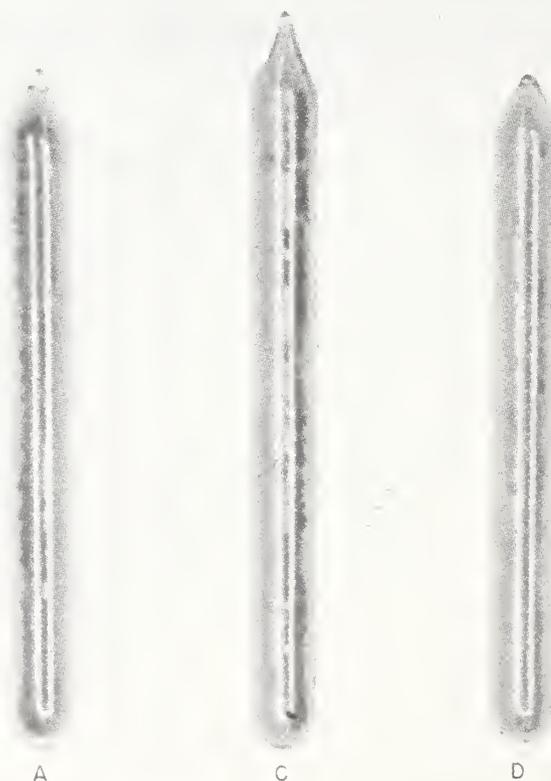


FIGURE 1. Three Hönigschmid national radium standards with the grains of salt distributed along the length of the tubes.

A, American; C Canadian D, American.

¹ Figures in brackets indicate the literature references at the end of this paper.

TABLE 1.—Masses of five Hönigschmid radium standards, in Hönigschmid milligrams

	A U. S. primary radium standard (5437, XIV)	B British primary radium standard (5432)	C Canadian primary radium standard (5425)	D U. S. primary radium standard (5440, XV)	G German primary radium standard (5426)
Mass of radium chloride as determined by Hönigschmid on June 2, 1934 ^a	50.22	20.50	31.73	26.86	19.19
Radium content, as of June 1934, as given by:					
1. Hönigschmid's weighings ^a	38.23	15.60	24.15	20.45	14.61
2. By comparison with Paris and Vienna 1911 standards ^b	38.12	-----	-----	20.37	-----

^a Hönigschmid stated [8] that his weights were calibrated against each other but not in relation to the standard kilogram. He also found [8] that the calibration corrections for his weights were greatly in excess of the weighing errors. The masses of radium have therefore been designated throughout this paper in terms of "Hönigschmid milligrams." This practice should also have been adopted in the previous paper [1, 2].

^b Corrected from "the end of 1936 or beginning of 1937", using a half-life of 1,620 years. It is uncertain what relation these masses in milligrams bear to Hönigschmid's later weighings [8].

been recalculated. Values for the rates of energy emission for all five standards (British, Canadian, German, and both United States) have also been corrected back to their date of sealing by Hönigschmid, namely, June 2, 1934, in order to eliminate the correction for the decay of radium and the considerably larger correction for the growth of radium E and polonium-210 between February 1954, when the British intercomparison was carried out, and November and December 1955, when the German and Canadian standards, respectively, were intercompared with the United States standards. In the recalculation of the British results the best estimates have been derived, incorporating the results obtained for the old Canadian standard.

In addition, the sensitivity of the radiation balance has been increased by a factor of some 6.7, to give a scale deflection of 2.8 mm/ μ w instead of 0.42 mm/ μ w. This was done primarily to calibrate a preparation of some 4.8 curies of tritium in the preparation of the Bureau's tritium standard. The radiation balance has also been placed in a temperature-attenuating enclosure to minimize the effects of fluctuations in the room temperature. This enclosure consists of a box of about 3/4-in.-thick dural, with internal dimensions of approximately 10 by 10 by 10 in., surrounded by about a 2-in. thickness of balsa wood. The radiation balance is placed on a balsa-wood block in the middle of this enclosure. A small opening in the top surface of the enclosure provides for access to the balance. An over-all improvement in the operating characteristics of the radiation balance was obtained.

In the subsequent discussion of the results, the United States Hönigschmid standards will again be designated *A* and *D*, the British standard *B*, the new Canadian standard *C*, and the German standard *G*. A summary of their essential data is given in table 1, and the two United States standards with the Canadian and German standards are shown in figures 1 and 2. The tubes containing each of the four standards are of Thüringen glass of 3-mm internal diameter and 0.27-mm wall thickness, and their lengths vary from 36 to 40 mm.²

² *A* was described as 36 mm long and *D* as 37 mm long in table 1 of the earlier paper [1], and they are indeed thus described in the certificates issued by the International Radium Standards Commission. Measurement of the photographs of *A* and *D* reveal, however, that *A* is the longer standard, being about 36.7 mm long, whereas *D* is only some 36.3 mm in length.



FIGURE 2. Three Hönigschmid national radium standards, with the grains of salt distributed along the length of the tubes.

A, American; *D*, American; *G*, German.

3. Results

In table 2 are summarized the results of all the measurements of energy-absorption rates for all five standards, both singly and balanced in pairs, at the different times of measurement, uncorrected for either the decay of radium or the growth of its daughter products.

The best estimates of the rates of energy absorption for *A*, *B*, *C*, *D*, and *G*, derived by the method previously described [2], are given in table 3, together with the values of these rates of energy absorption corrected to June 2, 1934. Two additional values of *A*

TABLE 2. Rate of energy absorption, in microwatts

Date	Source	Energy absorbed	Source	Energy absorbed
February 1954	A	6271.0	A-D	2930.9
Do.	B	2566.3	A-B	3721.2
Do.	D	3364.2	D-B	786.5
November 1955	A	6294.4	A-D	2925.3
Do.	G	2416.6	A-G	3895.1
Do.	D	3368.5	D-G	956.8
December 1955	A	6295.7	A-D	2942.8
Do.	C	3981.2	A-C	2323.6
Do.	D	3362.2	C-D	619.7

TABLE 3. Best estimates of rates of energy absorption, in microwatts

Date	Source	Energy absorbed	Energy absorbed as of June 2, 1934
February 1954	A	6280.6	5747.5
Do.	B	2563.8	2346.2
Do.	D	3353.0	3067.7
November 1955	A	6298.6	5729.3
Do.	G	2411.1	2193.7
Do.	D	3369.9	3065.4
December 1955	A	6300.3	5731.0
Do.	C	3979.1	3619.4
Do.	D	3359.7	3055.1
December 1955	A	6304.1	5734.9
February 1956	A	6304.4	5730.8
Do.	D	3364.7	3057.8

and one additional value for *D*, obtained in the course of calibrating two other radium preparations for use at the Bureau, are also included.

In the comparisons carried out with the NBS standard electroscop, the method of adjustment of the ratios previously described [2] was again used. The results for the adjusted ratios obtained by the electroscop and also for the ratios obtained from the best estimates of the rates of energy absorption given by the radiation balance are summarized in table 4, together with the corresponding ratios obtained from Hönigschmid's weighings. Slight differences in the last figure from the ratios previously reported [1] are due mainly to the rounding off of the last figure in the calculations and are insignificant. In table 4 it will be noted that the electroscop ratios are almost

always lower than those obtained by the radiation balance by an amount varying from about 0.1 to 0.4 percent. This would, however, be consistent with a slight gamma-ray source-self-absorption in the larger standard, which, in the ratios given, always appears in the numerator.

For complete internal consistency the ratios shown in columns 3, 4, and 5 of table 4 should be the same for any given pair of standards, apart from the effect of gamma-ray source-self-absorption just noted. It is interesting therefore to compare the percentage or fractional divergencies of these ratios one from another. This can best be done by dividing one ratio into another, whereupon any systematic error should immediately become apparent. The results of such a comparison are shown in columns 6, 7, and 8 of table 4, and the effect of gamma-ray source-self-absorption immediately becomes apparent from column 6 by the systematic trend of 2 or 3 parts in a thousand (except in the case of the ratio of *C* to *D*, which are of nearly the same mass).

Column 7 shows the divergence of the radiation-balance ratios relative to Hönigschmid-weighing ratios to be fairly random. Neither of these methods of measurement involves any gamma-ray effect.

Once again, however, a systematic trend of 3 or 4 parts in a thousand is apparent in column 8. This is consistent with the effect of gamma-ray source-self-absorption decreasing the electroscop readings for the larger standards. Here again the ratios for *C* to *D* would be expected to be higher due to their near equality in mass.

These trends are only slightly significantly greater, however, than the experimental errors involved, but a comparison of the figures of columns 7 and 8 does tend to confirm that the radiation balance is more nearly measuring the ratios as determined by Hönigschmid's own weighings.

As in the earlier intercomparison of the United States and British radium standards [1], it is of interest to compare the radioactive effect per milligram of radium element for each of the standards in order to check the combined internal precision of the electroscop or radiation-balance results, on the one hand, and of Hönigschmid's weighings, on the other.

TABLE 4. Adjusted ratios for five international radium standards

Date	Standards	Electroscop	Radiation balance	Hönigschmid	Ratio of radiation balance ratio to electroscop ratio	Ratio of radiation balance ratio to Hönigschmid ratio	Ratio of electroscop ratio to Hönigschmid ratio
February 1954	A/B	2.441	2.450	2.450	1.003	1.000	0.997
Do.	A/D	1.870	1.873	1.870	-----	-----	-----
Do.	D/B	1.305	1.308	1.310	1.002	0.998	.996
November 1955	A/G	2.608	2.612	2.617	1.002	.998	.996
Do.	A/D	1.870	1.869	1.870	-----	-----	-----
Do.	D/G	1.395	1.398	1.400	1.002	.999	.996
December 1955	A/C	1.578	1.583	1.583	1.003	1.000	.997
Do.	A/D	1.870	1.875	1.870	-----	-----	-----
Do.	C/D	1.185	1.184	1.181	1.000	1.003	1.003
February 1956	A/D	-----	1.874	1.870	-----	-----	-----
Average	A/D	1.870	1.873	1.870	^a 1.002	^a 1.002	^a 1.000

^a Ratio of averages.

The results of such an internal precision check are given in tables 5, 6, and 7, for the electroscopes and for the radiation-balance measurements. As before, the radioactive effect per Hönigschmid milligram, namely, divisions per second for the electroscopes and microwatts for the radiation balance, has been normalized to make the "best average" in each case equal to 100.00. This best average has again been determined by dividing the sum of Hönigschmid's masses into the sum of the radioactive effects for each series of three standards.

In table 8 are shown the values for *B*, *C*, and *G* that are obtained by assuming Hönigschmid's values for *A* and *D* and multiplying by the appropriate adjusted ratios, the values for which are given in table 4. In table 4 the results have been rounded off to four significant figures.

TABLE 5. Radioactive effect per Hönigschmid milligram of radium element, normalized to make the best average equal to 100.00

British intercomparison, February 1954

Method	A	B	D	Best average	Standard deviation (2 degrees of freedom)
Electroscope.....	99.93	100.31	99.88	100.00	0.24
Radiation balance.....	100.05	100.08	99.85	100.00	.13

TABLE 6. Radioactive effect per Hönigschmid milligram of radium element, normalized to make the best average equal to 100.00

German intercomparison, November 1955

Method	A	D	G	Best average	Standard deviation (2 degrees of freedom)
Electroscope.....	99.94	99.92	100.28	100.00	0.21
Radiation balance.....	99.96	99.98	100.13	100.00	.09

TABLE 7. Radioactive effect per Hönigschmid milligram of radium element, normalized to make the best average equal to 100.00

Canadian intercomparison, December 1955

Method	A	C	D	Best average	Standard deviation (2 degrees of freedom)
Electroscope.....	99.92	100.22	99.90	100.00	0.18
Radiation balance.....	100.08	100.06	99.72	100.00	.21

TABLE 8. Values of *B*, *C*, and *G*, in Hönigschmid milligrams of radium element as of June 2, 1934, derived from *A* and *D* by means of radiation-balance and electroscopes measurements

Standard	Radiation balance through—		Electroscope through—		Average all values
	A	D	A	D	
<i>B</i>	15.60 ₈	15.63 ₇	15.65 ₈	15.66 ₇	15.64 ₂
<i>C</i>	24.14 ₅	24.22 ₈	24.22 ₃	24.22 ₈	24.20 ₄
<i>G</i>	14.63 ₄	14.63 ₂	14.66 ₁	14.66 ₄	14.64 ₅

4. Discussion of Results

4.1. Radiation-Balance Measurements

The radiation balance permits measurements for each standard separately or for a comparison of any pair of standards. The practice followed in these experiments has, as previously, been that of measuring each standard separately and of making all possible comparisons. There are $n(n-1)/2$ possible pairs that can be formed from n standards. These $n(n-1)/2$ measurements, together with the n measurements on the standards alone, give a total of $n(n+1)/2$ observations for the estimation of n quantities. Section 3 in the preceding study [2] lists the formulas from which the least-squares estimates for values of the radium standards have been derived. These estimates are the so-called "adjusted values," which have been used in all subsequent numerical calculations.

The differences between the adjusted values and the original observations provide an estimate of the standard deviation of the measurements. Table 9 shows a recalculation for the comparison of the United States standards with the British standard and the old Canadian standard. The calculations are also shown for the comparison with the German standard and with the new Canadian standard. The three estimates for the standard deviation of a single measurement are 7.2, 6.4, and 3.8 μw and are in

TABLE 9. Calculation of standard deviation, in microwatts, for each comparison of the national radium standards

Standard ^a	Observed	Adjusted	Difference	(Difference) ²
A	6271.0	6280.6	9.6	92.16
B	2566.3	2563.8	2.5	6.25
C*	4117.5	4121.7	4.2	17.64
D	3364.2	3353.0	11.2	125.44
A-B	3721.2	3716.8	4.4	19.36
A-C*	2160.9	2158.9	2.0	4.00
A-D	2930.9	2927.7	3.2	10.24
C*-B	1558.6	1557.9	0.7	0.49
D-B	786.5	789.2	2.7	7.29
C*-D	774.2	768.7	5.5	30.25
Total.....				313.12
A	6294.4	6298.6	4.2	17.64
D	3368.5	3369.9	1.4	1.96
G	2416.6	2411.1	5.5	30.25
A-D	2925.3	2928.7	3.4	11.56
A-G	3895.1	3887.5	7.6	57.76
D-G	956.8	958.8	2.0	4.00
Total.....				123.17
A	6295.7	6300.3	4.6	21.16
C	3981.2	3979.1	2.1	4.41
D	3362.2	3359.7	2.5	6.25
A-C	2323.6	2321.2	2.4	5.76
A-D	2942.8	2940.6	2.2	4.84
C-D	619.7	619.4	0.3	0.09
Total.....				42.51

Comparison	Total of (Difference) ²	Degrees of freedom	Quotient	Standard deviation	
				Single measurement	Adjusted value
British.....	313.12	6	52.19	7.2	4.5
German.....	123.17	3	41.08	6.4	4.5
Canadian.....	42.51	3	14.17	3.8	2.7

^a C* designates the former Canadian standard [1,2], which was not a Hönigschmid standard.

unusually close agreement, considering the limited number of degrees of freedom available for each estimate. In the course of calibrating another radium preparation against *A* and *D* at the Bureau, a fourth estimate of the standard deviation was also found to be 3.8 μ w.

The standard deviation for the adjusted estimates is obtained by dividing the calculated standard deviation shown in table 9 by the square root of $(n+1)/2$, where *n* standards have furnished $n(n+1)/2$ measurements. In the present work, with three standards the divisor is $\sqrt{2}$, and with four standards the divisor is $\sqrt{2.5}$.

4.2. Electroscopical Measurements

Comparison of two radium standards by the electroscopical method is best effected by alternating the two standards in a support at a fixed distance from the electroscopical leaf and taking the ratio of the times (corrected for background) for the electroscopical leaf to fall through a specified angle. Three standards are intercompared by determining the ratios for all three possible pairs of standards, a complete set of readings being taken by each of two operators. These measurements were repeated twice, using two fixed distances from the electroscopical leaf for the new Canadian standard, whereas four different distances were used in the work with the German standards. In the earlier work [1] with the British standard and the old Canadian standard, three operators and two distances were used and a complete repetition made, so that 12 measurements were available.

Statistical examination of the results showed them independent of both distance and operator. The standard deviation for any particular ratio was determined from the 8, 6, or 12 individual determinations available. The standard deviations calculated for the various ratios are shown in table 10, both as calculated and when expressed as parts per thousand of the average value for the ratio. The standard deviation for a result obtained by one operator working at one distance is 2.1 parts per thousand. The standard deviation for the average

TABLE 10. Standard deviations for electroscopical measurements

Radium standards compared ^a	Average ratio, unadjusted	Number of measurements ^b	Standard deviation	
			Absolute	Parts per thousand
<i>A/D</i>	1.8684	8	0.0043	2.3
<i>A/G</i>	2.6095	8	.0058	2.2
<i>D/G</i>	1.3936	8	.0033	2.6
<i>A/D</i>	1.8709	6	.0041	2.2
<i>A/C</i>	1.5771	6	.0026	1.7
<i>D/C</i>	0.84454	6	.0013	1.6
<i>A/C</i>	1.8703	12	.0043	2.3
<i>A/B</i>	2.4438	12	.0083	3.4
<i>A/C*</i>	1.5675	12	.0048	3.1
<i>B/C*</i>	0.64246	12	.0022	3.4
<i>B/D</i>	0.76650	12	.0024	3.1
<i>C*/D</i>	1.1918	12	.0027	2.3

^a C* designates the former Canadian standard [1, 2], which was not a Hönigschmid standard.

^b Degrees of freedom equals number of measurements minus 1.

ratio is obtained by dividing by the square root of the number of measurements, giving 0.75, 0.85, and 0.85 parts per thousand as the standard deviation for the average ratios for the German, Canadian, and British studies, respectively.

5. Best Estimates for the Hönigschmid Standards

The data tabulated in table 4 have been used to fit straight lines to points that have been plotted using the radiation-balance ratio as ordinate and the weight ratio as abscissa. Only the ratios using *D* as the denominator are used. The three points plotted for the February 1954 data have the coordinates (1.869, 1.873), (0.7628, 0.7648), (1.000, 1.000). The point (1.000, 1.000) establishes a unit scale where one unit is set equal to 20.45 Hönigschmid milligrams. The line is determined by minimizing the sum of the squares of the perpendicular distances from the plotted points to the fitted line. The formulas are given in [2]. A perpendicular from each plotted point to the fitted line determines the coordinates of matching points on the line. The abscissa for each point is then converted back to Hönigschmid milligrams by multiplying by 20.45. The weights corresponding to the points on the line are compared with Hönigschmid's masses in table 11.

TABLE 11. Estimates, in Hönigschmid milligrams, of the masses of the Hönigschmid radium standards as of June 2, 1934

Standard	<i>A</i>	<i>B</i>	<i>C</i>	<i>D</i>	<i>G</i>
Hönigschmid's mass	38.23	15.60	24.15	20.45	14.61
Mass derived from radiation balance and Hönigschmid	{ 38.23; 38.24; 38.22;	{ 15.60; ----- -----	{ 24.15; ----- -----	{ 20.43; 20.42; 20.45 ₀	{ ----- ----- 14.62 ₂
Mass derived from electroscopical and Hönigschmid	{ ^a 38.21 ₆ 38.22 ₃	{ ^a 15.62 ₈ -----	{ 24.17 ₇ -----	{ ^a 20.44 ₂ 20.44 ₁ 20.44 ₅	{ ----- ----- 14.63 ₃

^a These results differ slightly from those given in table 5 of reference [1], the computations having been repeated giving greater weight ($k=1$) to the electroscopical ratios than in that paper ($k=2$).

Three similar lines were calculated for the ratios obtained with the electroscopical method. In every instance the derived masses in Hönigschmid milligrams agree with Hönigschmid's masses within his stated weighing errors. In order to be assured that no preference was given to *D*, in taking it as unity in the estimates of the best values, the calculation was run again in the *BAD* and *GAD* series, respectively, taking *B* and *G*, the smallest standards, as unity. No greater differences than 2 or 3 parts in the third decimal place were, however, observed.

It should be emphasized that these derived masses are the best estimates of the values of the Hönigschmid standards so far as the National Bureau of Standards results are concerned. Internationally recommended values must await the correlation of the results from all other laboratories.

We gratefully acknowledge the courtesy of the President of the National Research Council of Canada and of the President of the Physikalisch-Technische Bundesanstalt of the Federal Republic of Germany for making their respective Hönigschmid radium standards available to us for measurement; and we express our thanks to K. W. Geiger and G. v. Droste, respectively, for bringing the standards personally to Washington, D. C. We also extend grateful acknowledgments to W. E. Perry of the National Physical Laboratory for many helpful discussions and especially for drawing our attention to Hönigschmid's statement that his weights were not compared with the standard kilogram.

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advice on the temperature-attenuating enclosure for the radiation balance that was installed some time prior to the investigations here described.

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Preparation of New Solution Standards of Radium

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New radium-solution standards have been prepared in the ranges of 10 micrograms and also 10^{-9} and 10^{-11} gram of radium element. These have been compared with the National Bureau of Standards' 1940 and 1947 series of radium-solution standards and, as a result of these comparisons, it has been found that the 1940 10^{-9} and 10^{-11} -gram solution standards contained some 2 to 3 percent more radium element than certified. It has been shown that this difference probably arose in the dilution of the 1940 standards.

1. Introduction

Radium-solution standards have previously been prepared at the National Bureau of Standards in 1940 and 1947. The 1940 series consisted of standards in the microgram range, ranging from 0.1 to 100.0 μg of radium element in 5 ml of solution, and standards for radon calibration consisting of 10^{-9} and 10^{-11} g of radium element in 100 ml of the radium-salt and carrier solution. The 1947 series consisted only of microgram standards ranging from 0.1 to 100.0 μg of radium element in 5 ml of solution of the radium bromide and nitric acid acting as carrier.

Recently the stock of 10^{-9} -g radium-solution standards became so depleted that it was necessary to prepare a new set of standards which has been designated as the 1957 series of standards and which consists chiefly of 10^{-9} and 10^{-11} -g standards with a few microgram standards which were prepared for comparison purposes. A new set of "blank solutions" was also prepared consisting of 100-ml samples containing 0.2 percent by weight of $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$.

2. Radium Calibration

A sample of radium chloride containing approximately 10 mg of radium element was returned to the Radium Chemical Company for a re-separation from radium D and its products and for recrystallization. It was requested that the radium salt should be crystallized in such a manner that the grain size would be of the same approximate dimensions as those in the Hönigschmid radium standards (which were also radium chloride) and that the radium salt should be enclosed in a glass tube of about the same dimensions (length, diameter, and wall thickness) as the tubes used by Hönigschmid. It would then be possible to compare this radium source with the two United States primary radium standards [1,2],¹ using the NBS gold-leaf electroscope [3], without making any absorption corrections. In such a comparison the sources are supported horizontally and then gently tapped so as to spread the grains of salt uniformly along the glass tubes.

While the radium source was compared in this manner with the two primary standards, this comparison was only treated as confirmation for a series of microcalorimetric comparisons which were carried

out using the NBS radiation balance [4, 5]. These measurements consisted of three measurements of the rate of energy emission from the new radium source alone and also one triad of measurements [1, 2] of the new source relative to both primary standards (No. XIV and XV). The results of these measurements are shown in table 1.

TABLE 1. Radiation-balance results for comparison of new radium source with the United States primary radium standards

Date of measurement	Rate of energy emission in micro-watts from—			Milligrams of radium element in new source
	No. XIV	No. XV	New source	
October 23, 1956.....	-----	-----	914.8	6.108
November 1, 1956.....	-----	-----	914.8	6.108
November 8, 1956.....	-----	-----	914.6	6.107
November 28, 1956.....	5728.0	3060.3	914.1	6.104

In calculating the values shown in table 1 a correction was made for the growth of polonium-210 in the national standards since June 1934. The mean value of the rate of energy emission from the new source is 914.6 μw which corresponds to 6.107 mg of radium element as of November 1956. The gamma-ray comparison, carried out with the gold-leaf electroscope, gave an average value from twelve measurements equal to 6.08 mg of radium element.

3. Preparation of the New Radium-Solution Standards

The 6.107-mg radium source was now completely shattered at the bottom of a 5-liter thick-bottomed glass bottle under 3.052 liters of carrier solution, determined by weighing and consisting of 0.2-percent $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ plus 5-percent HCl, by imparting a sharp blow to the glass tube by means of a specially constructed glass rod with a thickened and elongated end which was struck at its other end with a hammer. By this procedure the master solution of radium and carrier, with a concentration of radium element of 2.001×10^{-6} g/ml, was prepared.

The dilutions that were made from this master solution are shown diagrammatically in figure 1. These dilutions were carried by two independent routes, designated as A and B, in order to check the accuracy of dilution. The master solution as well as all subsequent dilutions thereof were thoroughly

¹ Figures in brackets indicate the literature references at the end of this paper.

"10- μg " radium-solution standards are 10 μg as of August 1940 and May 1947, respectively.

In terms of the 1940 10- μg radium standards the 1957 "10- μg " standards were found to contain $10.150 \pm 0.0014 \mu\text{g}$ of radium in August 1957. This value is in terms of different 1940 ampoules and the agreement is well within the almost ± 1 percent uncertainty of the values of the 1940 standards.

In terms of the 1947 10- μg radium standards the 1957 "10- μg " standards were found, in the August 1957 measurements in the NBS $4\pi\gamma$ -ionization chamber, to have a radium content of $10.230 \mu\text{g}$ in contrast to the calibrated value of $10.16_3 \mu\text{g}$. This value is again within the almost ± 1 percent uncertainty of the 1947 standards.

Finally a number of the 20- μg and 50- μg standards of the 1947 series were compared with those of the 1940 series. The complete series of $4\pi\gamma$ -ionization-chamber measurements is summarized in figure 2 where, on the left-hand side the results of the comparisons of the 1940 to 1947 standards are shown. In this case both the 1940 and 1947 standards are certified as 10, 20, and 50 μg . The small errors indicated are those to be associated with the $4\pi\gamma$ -ionization-chamber measurements while the larger errors are those inherent in both the 1940 and 1947 standards. Within these latter limits there is no deviation from unity.

On the right-hand side the ratio of the four 1957 to seven 1947 10- μg standards is shown, *after correcting* for the volume of the 5.079-ml pipet (used in the 1957 series) to the equivalent of the 5 ml (i. e., correcting to 10.000 μg instead of $10.16_3 \mu\text{g}$ for the 1957 series). After this correction has been made the ratio of the 1947 series (certified simply as 10 μg) to the 1957 series should be unity. Once again the deviation from unity (1.0054 ± 0.0004 , the error of the $4\pi\gamma$ -ionization-chamber measurement) is well within the 0.8 percent "uncertainty" certified in the case of the 1947 series of radium-solution standards above, without even considering the errors inherent in the 1957 series which are estimated to total about ± 0.1 percent or 0.2 percent. The actual precision in

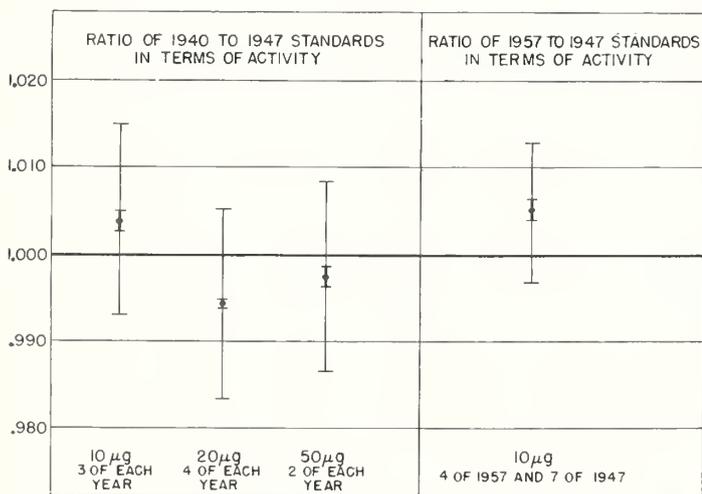


FIGURE 2. Comparisons of the activities of the 1940, 1947, and 1957 radium gamma-ray standards.

four microcalometric comparisons of the 6-mg radium preparation with the national radium standards was such as to give a standard deviation of the average of 0.02 percent.

4. Comparison of the 1940 and 1957 10^{-9} -g Radium-Solution Standards by the Method of Radon Analysis

The method of radon analysis in use at the National Bureau of Standards is essentially the method described by Curtiss and Davis [8, 9], but with the reflux condensers for de-emanation of the radium solution standards replaced by wash bottles with sintered-glass filters as described by Harding, Schwebel, and Stockmann [10]. The radon is removed from these solutions by means of a fine stream of nitrogen bubbles passing through the solutions from the sintered-glass filters. In order to confirm the results obtained with this method of de-emanation the reflux condensers were, however, reinstalled for a final set of comparisons.

Because of the somewhat lower precision of the radon method of analysis, as compared with the gamma-ray measurements already described, a most exhaustive series of intercomparisons between the 1940 and 1957 series of standards was carried out by the radon method.

As indicated in figure 1 ampoules 1A, 25A, 50A, 1B, 25B, and 50B were selected from the 1957 dilutions for comparative measurements.

Four 100-ml ampoules, designated as A, B, C, and D, were available from the 1940 10^{-9} -g radium-solution standards and were used to compare with the six 1957 ampoules.

The 1940 10^{-9} -g standards were certified just as 10^{-9} g of radium element. The certified value for the 1957 standards is 0.999×10^{-9} g of radium. The ratio of activities of the 1940 and 1957 standards was now determined by using each in turn to calibrate eight NBS alpha-particle-pulse-ionization chambers [8, 9] that are routinely used for radon assays. The results were expressed in terms of the ratio of the activity of the 1957 radium-solution standards, as determined by the calibration (in terms of counts per unit of radon) of any given chamber to that of the 1940 radium-solution standards corrected for radium decay, using the same chamber, and are shown in table 2 (a).

TABLE 2 (a). Comparison of 1957 and 1940 10^{-9} -g standards (The entries are the result of dividing the activities of the 1957 standard by those of the 1940 standards)

1940 Standards	1957 Standards					
	1A	25A	50A	1B	25B	50B
A-----	0.970	0.964	0.971	0.970	0.975	0.974
B-----	.987	.958	.980	.974	.987	.964
C-----	.982	.969	.983	.980	.962	.964
D-----	.983	.977	.965	.980	.976	.980
Average ratio 1957/1940=0.9740						
Standard deviation of the average of 24 ratios=0.0018						

The average ratio of 0.9740 derived from the results of table 2 (a) for the 1957 and 1940 standards indicates that the certified radium content of the 1940 standards was low by 2.6 percent.

Subsequently eight reflux condensers were re-installed for de-emanation of the standard solutions by boiling. The radon was fed from two of these reflux condensers into two of the eight alpha-particle-ionization chambers that had been used to obtain the results in table 2 (a). The values for the ratio obtained for the 1957/1940 activities (again obtained from the chamber calibrations) using the reflux condensers, and again corrected for decay, are shown in table 2 (b). These last results were not, however, used in calculating the average ratio of 0.9740 as they were not systematically determined for every combination of the 1957 standards (1A, 25A, 50A, 1B, 25B, and 50B) on the one hand and the 1940 standards (A, B, C, and D) on the other. The reflux-condenser measurements were merely confirmatory.

TABLE 2 (b). Comparison of 1957 and 1940 10^{-9} -g standards using reflux condensers for de-emanation

Ratio of activities of 1957 to 1940 standards			
1940 Standards	1957 Standards		
	50A	25B	50B
A-----	0.978	0.977	-----
B-----	.978	.975	0.984
C-----	.987	.986	-----
D-----	.986	.985	.989
	.983	.980	-----
Average ratio 1957/1940=0.9823			

This discrepancy of 2.6 percent was so large that it was considered desirable to check the 1940 and 1957 standards against the 1947 standards to try to determine which was the more likely to be in error.

Unfortunately, however, the 1947 series consists only of standards in the range of microgram quantities of radium element. It was, therefore, necessary to carry out a dilution of 1947-microgram standards to the 10^{-9} -g level. This was, however, accepted as an additional check on the accuracy of our dilution. The dilution scheme is shown in figure 3, the initial master solution consisting of six 10^{-6} -g radium-solution standards from the 1947 series. By taking six standards, each consisting of 5 ml of solution, the total error arising from the individual errors in volume of each of these standards should be proportionately lower. Once again dilutions were carried out by two alternate routes. Five samples were taken from each route and these were numbered as shown in figure 3. Again all glassware used was carefully calibrated. The nominal values of the dilutions were: 2×10^{-7} g/ml, 2×10^{-9} g/ml, and 1×10^{-11} g/ml.

The ratios of activities were now determined for the 1947 and 1940 10^{-9} -g samples and standards

MASTER SOLUTION CONSISTING OF SIX 10^{-6} -GRAM RADIUM GAMMA-RAY STANDARDS EACH CONTAINING 5 ml OF SOLUTION

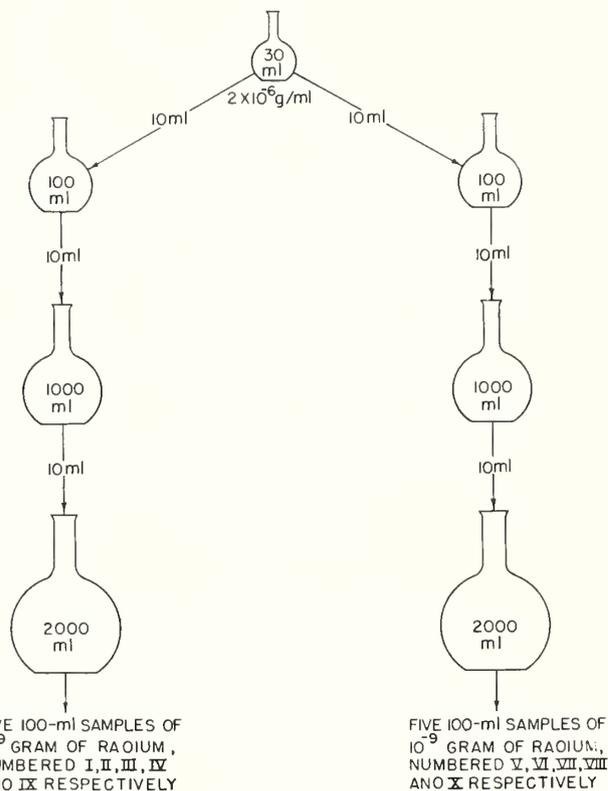


FIGURE 3. Dilution scheme for the preparation of 10^{-9} -g samples from 1947 radium gamma-ray standards.

and for the 1947 and 1957 10^{-9} -g samples and standards with the results shown in tables 3 (a) and 4 (a), the appropriate corrections again being made for radium decay. The sintered-glass-filter method of de-emanation was used in these measurements and also the same eight alpha-particle-ionization chambers as were used to obtain the 1957/1940 ratios shown in table 2 (a). In view, however, of the greater numbers of 1940 and 1957 10^{-9} -g standards and 1947 10^{-9} -g samples involved it was not possible to compare every standard solution with every 1947 10^{-9} -g sample. A pattern of comparison was devised, as indicated in tables 3 (a) and 4 (a) to give a maximum number of interchecks without taking every possible combination. Once again a number of spot comparisons was carried out using the same alpha-particle-ionization chambers and reflux condensers as were used to make the measurements in table 2 (b) and the results of these spot comparisons, again corrected for radium decay, are shown in tables 3 (b) and 4 (b). These results, as they were less systematic, were not used in the deriving of the 1947/1940 and 1947/1957 averages of 0.9722 and 0.9878, respectively.

These results of 0.9722 (1947/1940) and 0.9878 (1947/1957), as compared with 0.9740 (1957/1940), do tend to show a weight of evidence against the 1940 series of 10^{-9} -g radium-solution standards.

TABLE 3 (a). Comparison of 1947 10^{-9} -g samples and 1940 10^{-9} -g standards

(The entries are the result of dividing the activities of the 1947 samples by those of the 1940 standards)

1940 Standards	10 ⁻⁹ -g solutions made from six 1947 10-μg gamma-ray standards							
	I	II	III	IV	V	VI	VII	VIII
A-----	0.976	0.966	-----	-----	-----	-----	0.967	0.956
B-----	-----	-----	0.970	0.980	0.967	0.973	-----	-----
C-----	.967	.972	-----	-----	.981	.970	-----	-----
D-----	-----	-----	.978	.972	-----	-----	.981	.980

Average ratio 1947/1940=0.9722
Standard deviation of the average of 16 ratios=.0018

TABLE 3 (b). Comparison of 1947 10^{-9} -g samples and 1940 10^{-9} -g standards using reflux condensers for de-emanation

Ratio of activities of 1947 to 1940 standards				
1940 Standards	10 ⁻⁹ -g solutions made from six 1947 gamma-ray standards			
	I	II	V	VIII
A-----	{ 0.966	0.960	0.962	0.965
	{ .072	.968	.964	-----
B-----	{ .981	.975	.976	.968
	{ -----	-----	-----	.978
D-----	{ .974	.968	.970	.967
	{ .977	.973	.969	.970

Average ratio 1947/1940=0.9702

TABLE 4 (a).—Comparison of 1947 10^{-9} -g samples and 1957 10^{-9} -g standards (the entries are the result of dividing the activities of the 1947 samples by those of the 1957 standards)

1957 Standards	10 ⁻⁹ -g solutions made from six 1947 gamma-ray standards							
	I	II	III	IV	V	VI	VII	VIII
1A-----	0.990	0.979	0.991	0.984	-----	-----	-----	-----
25A-----	-----	-----	.987	1.008	1.007	0.987	-----	-----
50A-----	-----	-----	-----	-----	0.993	.988	0.974	0.984
1B-----	-----	-----	.989	0.986	-----	-----	.981	.982
25B-----	.990	.986	-----	-----	.990	.983	-----	-----
50B-----	.993	.982	-----	-----	-----	-----	.988	.984

Average ratio 1947/1957=0.9878
Standard deviation of the average of 24 ratios=0.0014

TABLE 4 (b).—Comparison of 1947 10^{-9} -g samples and 1957 10^{-9} -g standards using reflux condensers for de-emanation

Ratio of activities of 1947 to 1957 standards				
1957 Standards	10 ⁻⁹ -g solutions made from six 1947 gamma-ray standards			
	I	II	V	VIII
50A-----	{ 0.988	0.990	0.984	0.986
	{ .993	-----	-----	-----
25B-----	{ 0.996	0.983	0.988	0.989
	{ .989	.993	.985	-----
50B-----	{ 0.987	0.984	-----	0.980

Average ratio 1947/1957=0.9885

The direct measurements of the ratios for the standards and samples for the different years are therefore:

$$\begin{aligned} 1947/1940 &= 0.9722 & 1940/1947 &= 1.0286 \\ 1940/1957 &= 1.0267 & 1957/1940 &= 0.9740 \\ 1957/1947 &= 1.0124 & 1947/1957 &= 0.9878 \end{aligned}$$

$$\text{Product of 3 ratios} = 1.0124 \quad = 0.9896^2$$

The three estimates, if completely consistent, should multiply together to give exactly unity. The slightly less than 1-percent uncertainty in the individual ratios accounts for this discrepancy.

The three estimates may be slightly adjusted by a least-squares technique to give estimates that are consistent. For example, in addition to the direct estimate of the 1957/1947 ratio (1.0124) an indirect estimate may be obtained by multiplying the estimate for 1957/1940 by the estimate for 1940/1947. The result of multiplying 0.9740 by 1.0286 is 1.0019. Most weight must be given the directly obtained value of 1.0124. The proper average is obtained by taking the cube root of the product of the square of the directly observed ratio by the indirectly estimated ratio for 1957/1947. Thus

$$\sqrt[3]{1.0124 \times 1.0124 \times 1.0019} = 1.0089.$$

The adjusted estimates of the ratios between standards and samples for different years, obtained by this least squares technique are:

$$\begin{aligned} 1947/1940 &= 0.9688 & 1940/1947 &= 1.0322 \\ 1940/1957 &= 1.0231 & 1957/1940 &= 0.9774 \\ 1947/1947 &= 1.0089 & 1947/1957 &= 0.9912 \end{aligned}$$

$$\text{Product of 3 ratios} = 1.0000 \quad = 1.0000$$

The adjustments do not exceed twice the estimated standard deviation in the direct estimates of the ratios. The standard deviation of the average ratios listed in tables 2, 3, and 4 is slightly less than 0.2 percent. These adjusted values combine all the evidence and are the preferred ratios.

5. Comparison of the 1940 and 1957 10^{-11} -g Radium-Solution Standards by the Method of Radon Analysis

As a further check between the 1940 and 1957 series of standards three 1940 10^{-11} -g radium-solution standards of 100 ml, certified as containing 1.025×10^{-11} g and designated as 11, 12, and 13, were compared with three 1957 10^{-11} -g radium-solution standards of 100 ml, designated as 21, 22, and 23 and found to contain 1.001×10^{-11} g, by the method of radon analysis using alpha-particle-ionization chambers 7 and 12 and de-emanating by boiling in the reflux condensers.

In view of the longer collection times that were involved and the fact that the readings were only some ten times background the results took much

² The reciprocals of the ratios as determined from the original values are shown for convenience in computation, should they be desired.

longer to obtain and it was not, therefore, possible to carry out as exhaustive comparisons as with the 10^{-9} -g standards. The results, after correction for radium decay, are shown, however, in table 5. The average ratio of 0.986 (1957/1940), with a standard deviation of the average of 0.020, is in fairly close agreement with the value of 0.9774 (1957/1940) for the adjusted ratio for the 10^{-9} -g radium-solution standards. It must, however, be borne in mind that additional errors are introduced at such low concentrations as 10^{-11} -g radium per 100 ml by uncertainties in the radium content of the diluting carrier solution, as will be apparent from measurements made on such solutions which will now be described.

TABLE 5. Comparison of 1959 and 1940 10^{-11} -g standards using reflux condensers for de-emanation

Standard No. ^a	Radium content in units of 10^{-11} g	
	Chamber No. 7	Chamber No. 12
11-----	1.040	-----
21-----	-----	1.032
12-----	1.001	-----
22-----	-----	1.016
11-----	-----	1.047
21-----	1.022	-----
12-----	-----	1.007
13-----	1.032	-----
23-----	-----	0.990
13-----	-----	1.018
23-----	1.012	-----
22-----	0.987	-----

Average ratio 1957/1940=0.986
Standard deviation=0.020

^a 1940 Standards designated as 11, 12, and 13; 1957 Standards designated as 21, 22, and 23.

6. Determination of the Radium Content of the Carrier Solution

The carrier solution used in the dilutions, shown schematically in figures 1 and 3, consisted of 0.2 percent by weight of $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$. In order to determine the radium content of this carrier solution, 31 g of the barium chloride used in its preparation was dissolved in 100 ml of radium-free distilled water and this solution was then analyzed for radium by the radon method. Three measurements of this sample gave values of 0.1245, 0.1258, and 0.1261×10^{-12} g, or an average of 0.1255×10^{-12} g, radium per gram of $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$. Thus a 100-ml sample of the 0.2 percent by weight barium chloride carrier solution would contain a total of 0.025×10^{-12} g of radium.

There is no record of the method of measuring the 1940 blank solutions certified as containing 0.25×10^{-12} g of radium per 100 ml. Attempts were made, however, to measure by the radon method the radium content of both 1940 and 1957 blank solutions. These attempts resulted in a wide range of values being obtained, some of which were as much as ten times greater, in the case of the 1957 blank solutions, than the value obtained from the measurements of the nearly saturated solution of barium chloride.

These variations reflect the limitations of the

radon method for measurements which are, in this case, of the order of one-tenth background. The average value obtained, however, for the 1940 blank solutions was 0.18×10^{-12} g of radium per 100 ml as compared with the certified value of 0.25×10^{-12} g of radium per 100 ml.

It appears that the 1940 10^{-11} -g radium-solution standards which are certified as containing 1.025×10^{-11} g of radium were derived from the dilution of the 10^{-9} -g radium solution, certified as containing 10^{-9} g of radium per 100 ml, with the blank solution which was said to contain 0.025×10^{-11} g of radium per 100 ml. If this last figure were obtained, however, by the radon method it would appear to be no more reliable than that of 0.18×10^{-12} g of radium per 100 ml recently determined.

Under these circumstances the value of 0.986 obtained for the ratio of the 1957/1940 10^{-11} -g radium solution standards cannot be said to differ significantly from that of 0.977 obtained for the ratio of the 1957/1940 10^{-9} -g radium-solution standards.

7. Summary

From the measurements on the 1940 and 1957 10^{-9} -g radium-solution standards, which are confirmed by those of the 10^{-11} -g series, it would appear that there is an error of about 2.6 percent in the 1940 series of 10^{-9} -g and 10^{-11} -g radium-solution standards. As no corresponding discrepancy has been observed in the microgram series of standards it is assumed that the error is one which occurred in the dilution down to 10^{-9} g and 10^{-11} g per 100 ml in 1940.

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Branching Ratio in the Decay of Polonium-210

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Using a micro-calorimeter to determine the alpha-particle activity and a sodium-iodide scintillation counter of high efficiency to measure the gamma-ray intensity, the branching ratio of polonium-210 has been found to be equal to $(1.22 \pm 0.06) \times 10^{-5}$. This value is based on the assumption that the energy of the main alpha-particle group from polonium-210 is 5.301 Mev. In the calibration of the scintillation counter the angular anisotropy of the gamma rays from cobalt-60 was found to be 1.164 ± 0.002 .

1. Introduction

The number of gamma quanta emitted per disintegration of polonium-210 was recently determined by Grace, Allen, West, and Halban [1]¹ in the course of the investigation of a method for standardizing polonium sources by measurement of the hard 0.804-Mev [2] gamma radiation. The intensity of the alpha radiation emitted from a 200-mc source of polonium-210 deposited on one side of a platinum foil was measured directly by means of a low-geometry defined-solid-angle method, whereas the 0.804-Mev gamma-ray intensity was determined in terms of a source of cobalt-60 by means of a cylindrical aluminum Geiger-Müller counter of unspecified filling. The absolute disintegration rate of the cobalt-60 source, which it is assumed was of a similar geometry, to that of polonium-210, was determined by means of the method of beta-gamma coincidence counting whereas the relative efficiencies of the Geiger-Müller counter to the gamma radiations from cobalt-60 and polonium-210 were assumed to be the same as those determined for a counter of similar geometry by Bradt, Gugelot, Huber, Medicus, Preiswerk, and Scherrer [3].

The opportunity has arisen to repeat the measurement of the branching ratio of polonium-210 using, however, a micro-calorimetric method to determine the rate of alpha-particle emission and a sodium-iodide scintillation counter, of high detection efficiency, to determine the intensity of the 0.804-Mev gamma rays by comparison with a cobalt-60 source, the gamma-ray intensity of which was determined by the method of gamma-gamma coincidence counting.

2. Preparation of the Polonium-210 Sources

Experiments were performed on two polonium-210 sources, the first of which, however, was too weak to give satisfactory accuracy. This had been supplied sealed in a nickel tube about 1.5-cm long and 0.15-cm in external diameter. The gamma-ray spectrum indicated some energetic gamma-emitting contamination, but with an activity corresponding to 3.7 mc only a very approximate value for the branching ratio of 1.1×10^{-5} gamma quantum per alpha particle was obtained.

The second polonium-210 source was prepared from some 60 mc of solution that had been carefully purified prior to delivery. This purification consisted of depositing polonium-210 onto a silver foil from a stock solution containing approximately 130 mc of polonium-210. The silver foil was then washed free from solution and dissolved in concentrated nitric acid. After expelling most of the nitric acid by evaporation the silver was precipitated by means of hydrochloric acid. The solution was filtered from the silver chloride and its acidity adjusted for deposition on a second silver foil, which was again processed in the same way as the first, the filtrate from the silver chloride being the final solution containing some 60 mc of purified polonium-210.

Upon receipt of this solution its acidity was adjusted to a value of pH equal to 1 and 0.1 g of finely powdered silver was allowed to stand in the solution overnight. After standing for this time the solution was filtered through a plug of glass wool in a glass tube having external and internal diameters of approximately 3 and 2 mm. The silver powder that was retained by the glass-wool plug was ultimately found to have extracted some 50 mc of the polonium-210. The glass tube was then fused off below the glass-wool plug and above the silver powder to give a source 37-mm long and whose approximate form is illustrated in figure 1. On examination of its gamma-ray spectrum, this source was found to be free from the contamination that had been present in the first source.

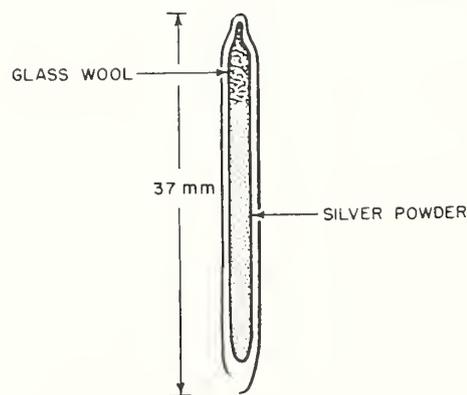


FIGURE 1. Polonium-210 source.

¹ Figures in brackets indicate the literature references at the end of this paper.

3. Measurement of the Alpha-Particle Activity

The alpha-particle intensity of the polonium-210 source was determined by means of a gold-cup radiation balance that was constructed to compare four national radium standards [4]. The energy emission was combined with an average value² of 5.301 Mev for the energy of the polonium-210 alpha particles to give their intensity.

As this radiation balance had previously been used primarily for relative measurements, it was carefully recalibrated so as to give absolute rates of energy dissipation. Such a calibration consists essentially in determining an average value of dE/dT for the two Peltier couples, and for this purpose a completely new set of compensated calibration coils, encased in copper sheaths and with permanent potential leads, was constructed. Previous calibrations had been made with coils that had been wound on glass of such dimensions as to simulate the United Kingdom and the United States Hönigschmid primary radium standards and the Canadian national radium standard [4]. The results obtained for dE/dT with the "Hönigschmid", "Canadian", and the new copper-sheathed coils are shown in table 1.

TABLE 1. Measurement of the thermoelectric power, dE/dT at 25° C

Coil	dE/dT^a
	$\mu w/deg$
"Hönigschmid".....	58.86
Do.....	58.95
"Canadian".....	58.71
Do.....	58.59
Copper-sheathed.....	58.49
Do.....	58.67
Do.....	58.89
Do.....	58.79
Do.....	58.79
Do.....	58.60
Do.....	58.70
Do.....	58.62
Do.....	58.53

^aThe average value of dE/dT is 58.71 $\mu w/deg$ at 25° C.

The source of polonium-210, that is illustrated in figure 1, is such as to enable a further important check to be made in connection with the operation of the radiation balance. It has been assumed [4] that the thermal "mixing" in the gold cups is of such a magnitude as to render any differences in temperature distribution in the cups due to difference in source geometry unimportant. A series of measurements was carried out therefore with the polonium-210 source, and a dummy source containing a glass-wool plug and inactive silver powder, in which the energy emission from the source in the erect position, as in figure 1, was compared with that as measured with the source and dummy upside down. In the erect position the polonium-210 is concentrated in the lower part of each cup in turn, whereas in the upside-down position a relatively good thermal insulator, the glass-wool plug, is in close proximity

² From values 5.304 and 5.298 Mev [5, 6].

TABLE 2. Energy dissipation from polonium-210 source as measured in erect and upside-down positions

Date, 1954	Energy emission (erect)	Energy emission (upside down)
	μw	μw
June 22.....	1323.6	1332.4
June 29.....	1276.9	1275.9
July 6.....	1235.2	1234.9
July 20.....	1151.2	1151.2

TABLE 3. Energy dissipation from polonium-210 as a function of time

Date, 1954	Time (EST)	Hours from zero	Observed energy emission	Energy emission corrected to zero date ^a (using half-life of 138.39 days)
			μw	μw
June 22.....	13.30	-167.75	1323.6	1278.1
June 29.....	13.15	0	1276.9	1276.9
July 6.....	15.05	169.75	1235.2	1279.8
July 13.....	14.40	337.25	1190.5	1277.3
July 20.....	14.20	505.25	1151.2	1279.2
August 24.....	13.25	1344.25	961.43	1272.8
August 31.....	10.05	1508.75	929.29	1273.2
September 7.....	14.30	1681.25	899.37	1277.5

^a Average value is 1276.9 μw , with a standard deviation of 0.2 percent.

with the Peltier couples at the bases of the cups.

As is seen from table 2, there is no significant difference between the measurements in the two positions; the difference between the first two measurements is not outside the possible range of experimental error.

Finally, in table 3 the results are shown for rate of energy emission from the polonium-210 source on various dates of measurement. Also shown are the values that have been obtained after correction, using a half-life of 138.39 days [7], to a zero date of 13.15 hours EST on June 29, 1954, on which date, in the morning, the gamma-ray intensity was determined.

Using an alpha-particle energy of 5.301 Mev, which corresponds to a total disintegration energy of 5.404 Mev, and the average value of 1276.9 μw for the energy emission, the alpha-particle activity at the above time is found to be $1,475 \times 10^6$ disintegrations per second. As the gamma-ray measurements were made some 3 or 4 hours earlier, it is appropriate to correct this value to $1,476 \times 10^6$ disintegrations per second at the time of the gamma-ray experiment.

4. Measurement of the Gamma-Ray Intensity

The intensity of the gamma radiation from the same polonium-210 source as used in the above experiment was determined by comparison with a cobalt-60 source, in the form of cobaltous chloride in aqueous solution, through the use of a sodium-iodide scintillation counter of high detection efficiency. The disintegration rate of the cobalt-60 source had been determined absolutely by the coincidence method.

The experimental procedure was as follows: The polonium-210 and cobalt-60 sources of identical dimensions as in figure 1 were placed alternatively on the axis of, and 3 in. from the face of, a cylindrical sodium-iodide, thallium-activated, crystal, 5 in. in diameter and 4 in. long, as shown in figure 2. This crystal was hermetically sealed in a thin aluminum container, using magnesium oxide as a diffuse reflector on all surfaces except that which was viewed by a 5-in. diameter photomultiplier (type K1198). The output of the photomultiplier tube was fed through a linear amplifier into a single-channel differential pulse-height analyzer from which the intensity versus pulse-height spectra shown in figure 3 were obtained. As the sodium-iodide crystal is not quite totally absorbing, the relative efficiency for the detection of the gamma rays from the polonium and cobalt sources must be calculated. This relative efficiency is given by

$$\epsilon = \frac{\int_0^\alpha (1 - e^{-\mu(E_1)\rho r(\beta)}) \sin \beta d\beta}{\int_0^\alpha (1 - e^{-\mu(E_2)\rho r(\beta)}) \sin \beta d\beta}, \quad (1)$$

where

$$\begin{aligned} x(\beta) &= t \sec \beta & 0 \leq \beta \leq \tan^{-1} r/(D+t) = \beta' \\ x(\beta) &= r \csc \beta - D \sec \beta & \beta' \leq \beta \leq \tan^{-1} r/D = \alpha \\ \mu(E) &= \text{mass absorption coefficient for energy } E \\ \rho &= \text{density.} \end{aligned}$$

The values of $\mu(E)$ for sodium iodide were obtained from the tables of White [8] for energies corresponding to those of 0.804 and 1.25 Mev, the latter value being the average energy of the cobalt-60 gamma rays; this artifice introduces negligible error. For the geometry and dimensions given in figure 3, one obtains a value 1.123 for the relative detection

efficiency, ϵ , by numerical integration of eq (1). The disintegration rate for the polonium gamma ray is given by

$$N_{0\gamma}(\text{Po}^{210}) = \frac{A(\text{Po}^{210})}{\epsilon A(\text{Co}^{60})} N_0(\text{Co}^{60}), \quad (2)$$

where A is the area under each of the spectra of figure 3. The area under the cobalt-60 spectrum is given proper weight to take into account the cascade nature of the gamma radiation. Four determinations of the relative areas give a value $A(\text{Po}^{210})/A(\text{Co}^{60}) = 0.648$, thus we obtain a value for the disintegration rate of the polonium gamma ray in terms of the Co^{60} disintegration rate, i. e.,

$$N_{0\gamma}(\text{Po}^{210}) = 0.5770 N_0(\text{Co}^{60}).$$

The estimated error of this relationship may be as great as 5 percent because of the uncertainty in correcting for secondary effects, such as backscattering into the detector.

The disintegration rate of the cobalt-60 source was determined by the gamma-gamma coincidence method. This disintegration rate in terms of ob-

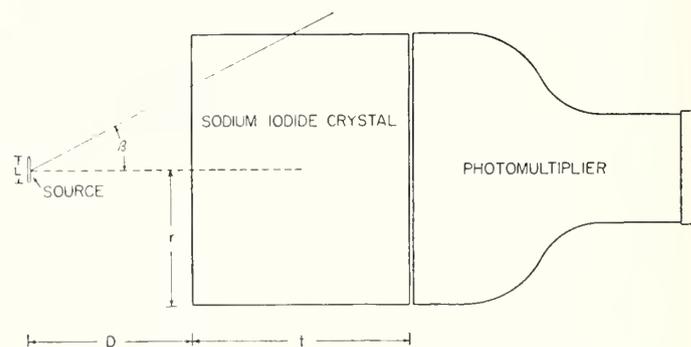


FIGURE 2. Geometrical arrangement for comparison of gamma-ray spectra of cobalt-60 and polonium-210 sources.

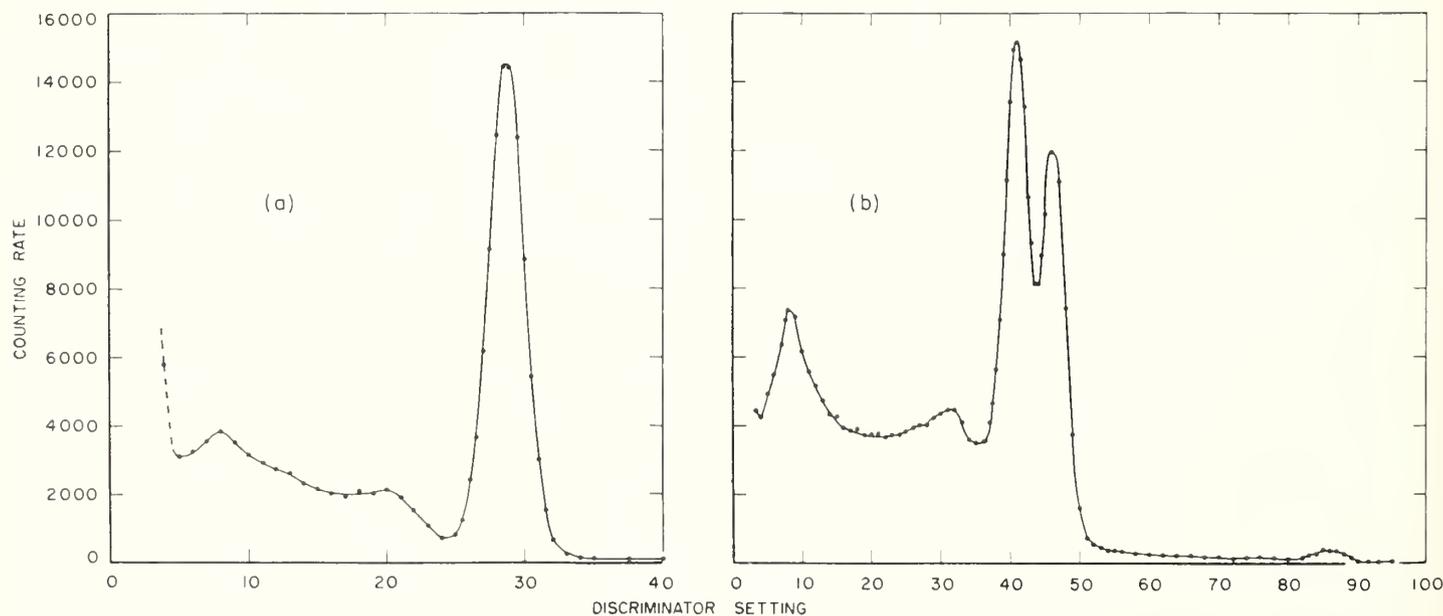


FIGURE 3. Scintillation pulse-height spectra of (a) polonium-210 source, (b) cobalt-60 source.

servable quantities is given by

$$N_0 = \frac{N_1 N_2}{N_c} \frac{\zeta_1 + \zeta_2}{(\zeta_1 + 1)(\zeta_2 + 1)} f(\vartheta), \quad (3)$$

where N_1 and N_2 are the counting rates in each of two similar counters, N_c is the number of coincidence counts between the two counters, ζ_1 and ζ_2 are the ratio of efficiencies for the two gamma rays in the first and second counters, respectively, and $f(\vartheta)$ is a function depending on the geometry of the source, counters, and any angular correlation between the coincident gamma rays. For the cobalt-60 source where there is a definite angular correlation, this function takes the form (9).

$$\begin{aligned} f(\vartheta) = & 1 + (J_2/J_0)^2 0.1020 P_2(\cos \vartheta) \\ & + (J_4/J_0)^2 0.0091 P_4(\cos \vartheta) \\ & + \frac{1}{4} (L/D)^2 [1 - P_1(\cos \vartheta)] \{ 0.1020 P_1(\cos \vartheta) \\ & + 0.0091 [P_1 \cos \vartheta + \frac{7}{3} P_3(\cos \vartheta)] \} \end{aligned} \quad (4)$$

The terms containing J_l are attenuation coefficients due to the finite angular resolution of the counters, where the J_l is given by

$$J_l(\alpha) = \int_0^\alpha P_l(\cos \beta) (1 - e^{-\mu(E)\rho x(\beta)}) \sin \beta d\beta. \quad (5)$$

The expression for $x(\beta)$ is of the same form as in eq (1). The last term in eq (4) is a correction term [10] due to the finite length of the source. The numerical coefficients of the Legendre polynomials are theoretical values for a cascade transition between states with spins $4 \rightarrow 2 \rightarrow 0$ [11].

With a geometry as shown in figure 4, the theoretically expected anisotropy is $f(\pi)/f(\pi/2) = 1.150$. The experimentally observed value was 1.147, indicating that when the corrections for the finite sizes of source and counters are accounted for, the measured differential anisotropy of 1.164 is well within the probable error (approximately 1 percent) of being the same as the theoretically expected value of 1.167 [11].

Several determinations of the disintegration rate under different geometrical conditions gave a value

$$N_0(\text{Co}^{60}) = (3.087 \pm 0.030) \times 10^4 \text{ dps.}$$

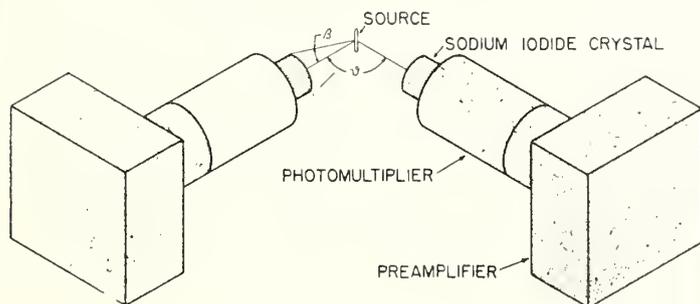


FIGURE 4. Geometrical arrangement for coincidence measurements on cobalt-60 source.

Using this value, we obtain as the intensity of the polonium-210 gamma rays

$$N_{0\gamma}(\text{Po}^{210}) = (1.782 \pm 5\%) \times 10^4 \text{ dps.}$$

5. Branching Ratio

Combining the gamma-ray and alpha-particle intensities, the following value for their ratio is obtained:

$$\frac{N_{0\gamma}}{N_{0\alpha}} = \frac{1.782 \times 10^4}{1.476 \times 10^9}$$

$= (1.207 \pm 0.060) \times 10^{-5}$ gamma quantum per alpha particle.

This value of $(1.21 \pm 0.06) \times 10^{-5}$ is to be compared with that of $(1.8 \pm 0.14) \times 10^{-5}$ obtained by Grace, Allen, West, and Halban [1] and $(1.6 \pm 0.2) \times 10^{-5}$ by M. Riou [12]. The internal conversion of K-shell electrons in polonium-210 is 0.0084 [13] and correction for L-shell conversion electrons [14] gives a total conversion coefficient of 0.0107. Using this value, the branching ratio is found to be $1.22 \times 10^{-5} \pm 5$ percent.

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