DEPARTMENT OF COMMERCE

CIRCULAR

OF THE

BUREAU OF STANDARDS

S. W. STRATTON, DIRECTOR

No. 107

THE TESTING OF PAPER

FEBRUARY 12, 1921

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ABSTRACT

This circular contains information relating to the methods of testing and the apparatus employed in the paper laboratories of the Bureau of Standards for the routine testing of paper. In the introduction a brief description of the raw materials used, the size and importance of the paper industry, and the general groups or classes of paper are given. The classification of paper is only of a general nature. The purpose of such tests and the development of methods of testing is touched upon, and suggestions are given as to the methods of developing specifications.

The testing of paper is divided into three groups, and the methods are classed as physical, chemical, and microscopical. Under each group, the various standard methods are given in detail with photographs of apparatus employed. No attempt is made in this circular to interpret results of tests. It is brought out that changes of temperature and humidity affect the physical qualities of paper, and for this reason a constant-temperature and humidity room has been installed. It has not been possible to give the relation between humidity and temperature changes and the physical characteristics of paper, but it is hoped to have this information available later. The chemical testing of paper is concerned with the determination of the amount and kind of filler or loading materials used and the amount and kind of sizing in the paper, and the methods are given in some detail, both qualitative and quantitative. It is desirable to know the kinds of fibrous materials out of which a sheet of paper is made and for this purpose certain stains are used to color the fibers on a microscopical slide. The procedure is given, and suggestions are made as to the value of microphotographs. A short working bibliography is included, as well as regulations for tests and methods of sampling and submission of samples for test.

CONTENTS

I. INTRODUCTION [1]

The testing of paper by means of scientific methods has received an increasing amount of attention in the United States during the past 10 years. This has been brought about largely by the increase in the number of technically trained men in the industry and the desire of placing empirical and practical tests upon a more accurate basis.

The purpose of this circular is to describe the methods of testing paper as used by the paper section of the Bureau of Standards and adopted as a result of testing a large number of samples of various kinds of paper during a period of years. These methods are those that are in common use in paper-testing laboratories and are given to fill a need expressed by laboratories and the general public. Proposed new methods and special tests are not included in this circular, but these will be published separately as they are developed.

The structure of paper is such that there is variation in some of its characteristics, even in a single sheet. It is therefore obvious that precaution must be taken to sample a lot of paper in such a way as to obtain a test sample representative of the whole lot. It is also necessary to make a sufficient number of tests in each case, in order that an average result may be obtained. It is important to obtain this average by testing 10 sheets of paper, rather than by making 10 tests on a single sheet.

[1] This circular was prepared by F. A. Curtis, chief, paper section, Bureau of Standards, and contains information relating to the methods of testing and the apparatus employed in the paper laboratories of this Bureau for the routine testing of paper.

1. SIZE OF INDUSTRY

The production of paper in the United States has increased approximately 55 per cent in the last 10 years, and there is now being manufactured annually, in the United States, approximately 7,000,000 tons of paper of all kinds. The pulp and paper industry in 1914 ranked sixth among the industries of the United States in regard to the amount of capital invested and in regard to the value of the product, and ranked fourth in regard to the value added by manufacture. It can thus be readily seen that the pulp and paper industry is an important one, and there is little doubt but that technical development is essential for the future of the industry.

2. RAW MATERIALS

Paper can be made from most fibrous vegetable matter, but technical difficulties, cost of manufacture, and grade of paper produced preclude the use of many fibrous materials. Wood pulp, old rags, old papers, straw, and old rope are the substances generally in use in this country. Wood pulp is produced by four processes, one of which, "ground wood" pulp, is mechanical, while the other three, "sulphite," "soda," and "sulphate," are largely chemical. There are a large number of commercial grades of rags and waste paper. All these materials are used in different classes of paper for different purposes, or in many cases mixtures are made to produce certain qualities in paper. In addition to the fibrous material entering into the manufacture of paper, certain noncellulose materials are necessary or desirable. The addition of rosin sizing to paper pulp, and its precipitation by alum, gives the finished paper certain difficultly definable qualities. Clays and similar materials, when added, produce a more even or smooth surface to the paper which is necessary for some kinds of printing.

Experiments have been made, some of them a hundred years ago, to produce paper from various kinds of grasses and fibrous material. An incomplete list is here given: Asparagus, bagging, bamboo, banana, beet root, blue grass, bran, broom corn, cabbage stumps, coconut husks, cottonseed hulls, cotton stalks, corn husks, palm, esparto, ferns, flax, grapevines, hay, hemp, leaves, moss, mulberry, nettles, peat, plantain, reeds, rice straw, rushes, sawdust, seaweed, thistles, tow, and many others. Some of these experiments have been successful on a laboratory scale, some have even produced paper in large enough quantity for printing, but only a few have ever reached a commercial standing.

A study of Table 1 brings out an interesting change in the proportions of various raw materials used at various periods.

TABLE 1.—Percentage Proportions of Constituent Materials Used in Paper Making, by Decades, Since 1880

Materials	1880	1900	1910	1918
	Per cent	Per cent	Per cent	Per cent
Rags	30	14	8	6
Straw	40	21.5	6.5	5.5
Wood pulp	.....	38	61	57
Old papers	14	21	21	30
Miscellaneous	16	5.5	3.5	1.5

It is to be noted, however, that wood pulp was used to some extent in 1880, but was not recorded and tabulated as a separate item.

3. DEFINITION AND TYPES OF PAPER

Paper is a matted or felted structure of fibrous material, formed into a relatively thin sheet. It is composed essentially of cellulose fibers obtained from vegetable growths in a more or less pure state. These fibers may be grouped as follows: (*a*) Seed fibers, or seed hairs, (*b*) stem or bast fibers, (*c*) leaf fibers, (*d*) fruit fibers, and (*e*) wood fibers. In general, it is customary to consider and classify paper according to its use rather than according to its constituents. In the testing of paper the use to which it is to be put determines the kinds of tests necessary to evaluate its quality.

The classification of the various types of paper has not reached a satisfactory state, and the nomenclature and trade names are often very confusing to the layman. This is largely due to the lack of standardization, the difficulty of defining numerically certain qualities, and the fact that the distinctive line between various kinds of paper is, in many cases, so slight that it is almost impossible to tell by what name to call it. The following general classes of paper given in Table 2 refer to broad types, and in each class there are variations of constituents as well as quality:

TABLE 2.—Percentage Output of the Several Classes of Paper Products

Paper products	Percentage of total, 1918	Fibrous materials used in varying proportions
Boards	30.4	Old paper, wood pulp, straw, old rope
Building	5.3	Old paper, wood pulp, asbestos, waste, rages, etc.
Blotting	.2	Rags, chemical wood pulp, cotton linters
Book and cover	15.3	Rags, chemical wood pulp
News and hanging	23.3	Mechanical and chemical wood pulp
Tissue	2.0	Chemical wood pulp, rags, old rope
Wrapping	16.8	Old rope, rags, sulphite, and sulphate wood pulp
Fine (writing, bonds, etc.)	5.8	Rags and chemical wood pulp
Specialties	.9	

II. QUALITY OF PAPER

The production of paper was for many years an art, especially during that period before the paper machine came into general use. It is obvious, therefore, that in the past the quality of paper was judged and determined by empirical methods and esthetic standards which were in many cases crude from a scientific point of view. It is true that an experienced man may determine much in regard to a sheet of paper by tearing it, by examining the finish or surface, and by looking through the sheet toward the light. Yet in all such tests the individual must be very experienced, and there is always the personal factor to be considered.

1. PURPOSE OF TESTS

The use of scientific or technical tests on paper has little value unless the purpose for which the paper is to be used is taken into consideration. The tests described in this circular are those which have been found effective in determining the relative value of various grades of paper and are given in their present state of development. These tests do not always give sufficient data, but their accuracy is generally within the variation in uniformity of the paper. There are, however, certain qualities of paper that it is difficult to define or record numerically, such as "color," "finish," and "formation." These three qualities are often the deciding factors in the purchase of paper, especially when it is not bought on specifications. It is therefore important to develop in the near future methods of testing these qualities.

2. DEVELOPMENT OF TEST METHODS

In the beginning of the development of paper-testing methods at this Bureau the methods used by foreign laboratories were more advanced and standardized than those in this country, and many of the former were adopted. Since then various new methods and improvements have been developed in the United States, and one of the greatest contributory agencies was the growth of a technical association in the paper industry It is true, however, that the requirements of the Government for various grades of paper and the need of placing these purchases upon a scientific basis have been a great stimulus at this Bureau in the development of the technique of paper testing. The large number of samples tested yearly to determine whether Government purchases have conformed to the specifications has given an opportunity for ascertaining the accuracy and suitability of the methods,

and has led to numerous changes and to the adoption of new methods. In general, it is necessary to develop tests for paper for a specific use. This is illustrated in the case of a tearing test, several of which have recently appeared in print; for in this case it was essential to find a test for wrapping paper which would reproduce service conditions more satisfactorily than with a bursting or "pop" test. In regard to certain other tests, such as the determination of the amount of rosin sizing in paper, it has been possible to simplify the procedure and to lessen the time required. The slight decrease in accuracy in the case mentioned, resulting from the use of the shorter method, limits its use to routine testing but conserves material and saves much time. It is becoming more and more apparent that no one testing device is applicable to all grades of paper, and it will be necessary to develop instruments for determining the physical qualities of each of the general classes of paper. This is illustrated by the fact that container board and tissue paper have different uses, are constructed differently, and should be tested differently. There is therefore a large field yet to be developed, not only in testing instruments but also in methods of analysis. Quality and uniformity depend in large measure upon methods of determining quality and uniformity, and such methods and apparatus should be made more specific and refined.

3. SPECIFICATIONS

It is becoming increasingly common to purchase material according to an agreement between the purchaser and the contractor which is based on the value of the material for the purpose for which it is bought. In such cases it is customary for the purchaser or the contractor to submit specifications of the material in question, which specifications are agreed upon by both parties interested before the purchase is ratified. These specifications may be a vague understanding between the purchaser and the contractor, they may be a very indefinite statement of the merits of the material in question, but they are becoming more and more to be based on scientific and technical data.

In the past, in the development of specifications for material, little attention has been paid to any particular standard for specifications, it being probably felt that each man or concern knew best how to write a specification. In many cases this led to a form detrimental to the purchaser, the contractor, or both. It is therefore thought that consideration should be given to specifications in general, which can then be specialized to meet

the particular case. With this in view, a brief outline is herewith given on the methods of preparing paper specifications. This topic is not here treated as a comprehensive standardization of the method of preparation of specifications, but is given merely as a guide to the preparation of specifications in a field in which the uses of specifications are still in the process of development.

(*a*) FORMULATION.—During the process of formulation of the specifications various interested contributory agencies should assist in revising them as first initiated. These specifications, as revised, should be finally agreed upon by the interested parties and be officially authorized. When so authorized they are given a certain official status, whether it be between individuals, companies, associations, or industries. Care is to be taken that the scope of application or jurisdiction of the specifications be made clear, as well as their designated or implied lifetime. It is also very desirable to include standard practice for revision of the specifications and also a standard typography and format.

(*b*) NOMENCLATURE.—A standard terminology should be adopted and adhered to throughout the specifications. When possible references and authority should be given. Special conditions or limitations as to terminology should be included, and a means for a decision as to disputes on terminology may be of assistance.

(*c*) QUALITY STANDARDS.—As a preliminary to the specifications of the quality of the material to be purchased, it is desirable to name exactly and define the material in question. The use to which the material specified is to be applied should be considered in great detail. It is then necessary to have a qualitative description of the useful properties desired and a statement of the undesirable properties. The quantitative statement of the useful properties, composition, dimensions, form, and structure, and of harmful or nonuseful properties, giving maxima and minima permissible, is that part of specifications which is often considered most important. It is necessary, however, to include a qualitative and quantitative statement of tolerances permitted in each case.

(*d*) TECHNICAL PRACTICE.—A material purchased on specifications must be tested in order to determine that it, on delivery, conforms to the specifications. Inspection and sampling are therefore necessary. Standard test methods must be a part of the specifications and naturally include a description of the test instruments and facilities to be used. Test certificates should be available, the results properly interpreted, and the material accepted, rejected, or subject to further test. All reports, records, and original data should be kept in available form.

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III. PHYSICAL TESTING

The physical qualities of paper, such as dimensions, weight, strength, and moisture content, are affected by atmospheric changes in temperature and relative humidity; but it is not possible, with the limited information available, to determine the factors necessary to reduce the data obtained at one temperature and relative humidity to that which they would be at another temperature and relative humidity, or at a standard atmospheric condition. The variation caused by changes of temperature and relative humidity is not constant for paper, in general, and is seldom constant within a restricted class of paper. In order to obtain standard conditions in which to test the physical properties of paper at this bureau, a large room has been equipped with an apparatus (Fig. 1) which maintains a temperature of 70° F and a relative humidity of 65 per cent. These conditions have been decided upon rather arbitrarily, yet seem advisable for the following reasons: It is relatively easy to produce the conditions; it is an atmosphere in which it is not uncomfortable to work; these conditions are apparently approximately midway in the increasing and decreasing curves of strength, due to variation of temperature and relative humidity, as indicated by work already done; and, lastly, most of the laboratories which control testing conditions have adopted 70° F temperature and 65 per cent relative humidity as their standard and have collected their test data under those conditions. Therefore, all samples of paper tested for physical qualities that are affected by atmospheric changes are conditioned for at least 2 hours in this room before being tested. They are placed on a rack properly fanned out in order that as much of the paper may be exposed as possible. (Fig. 2.)

1. WEIGHT

For convenience in laboratory work, a ream 25 by 40 inches in size containing 500 sheets (25×40—500) has been adopted as standard. The data are obtained in pounds per standard ream and are then converted to the ream size required for the particular sample of paper in question. Ten sheets of paper, each 10 by 10 inches in size, are weighed on a quadrant scale, illustrated in Fig. 3, having an average accuracy of approximately 0.2 per cent between 20 and 110 and a maximum error of 0.5 per cent between the same points on the scale. There are several types of scales or weighing devices on the market which may be

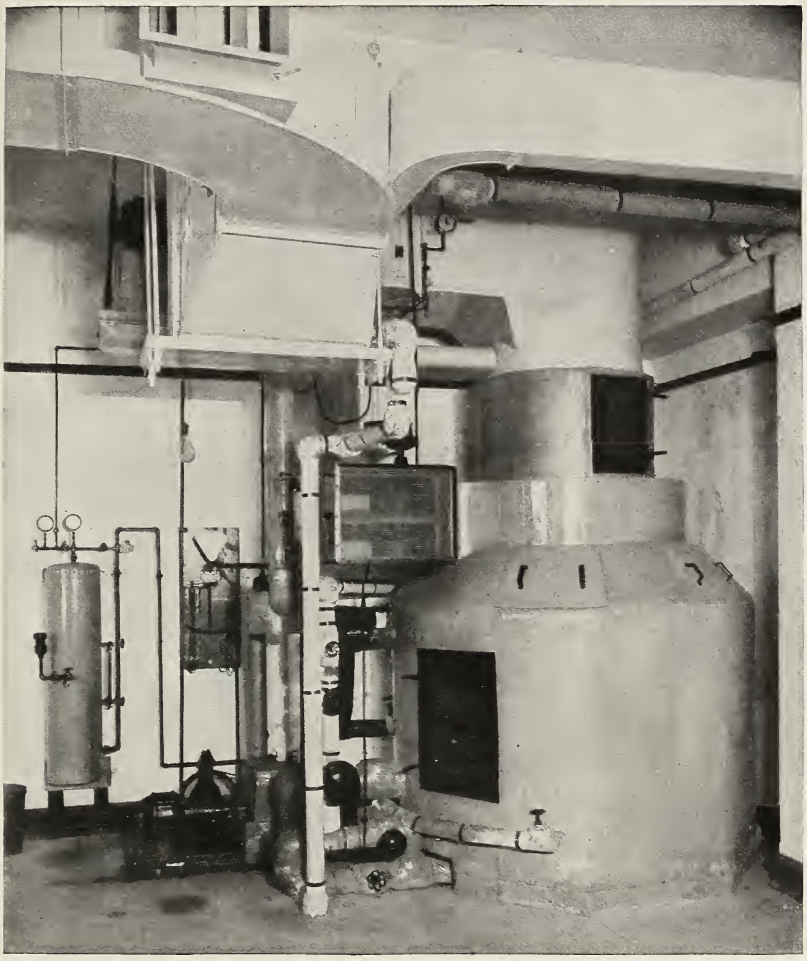

FIG. 1.—*Apparatus for maintaining standard atmospheric conditions: Temperature, 70° F, and relative humidity, 65 per cent*

This apparatus automatically washes the return air, saturates it at a given temperature, and then raises the temperature of air to a proper point so that standard conditions are maintained in the room

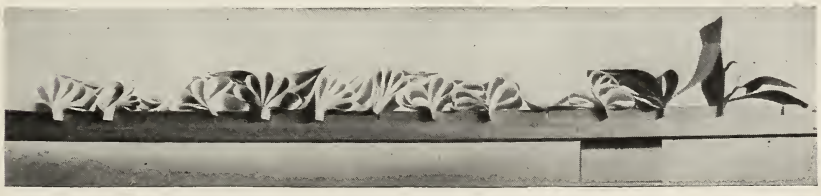

FIG. 2.—*Rack for conditioning paper*

Samples of paper, before being tested, are subjected to the standard conditions, so that all papers may be tested under the same conditions

FIG. 3.—*Scale for weighing paper*

This type of balance is used for laboratory work in determining the ream weight of paper

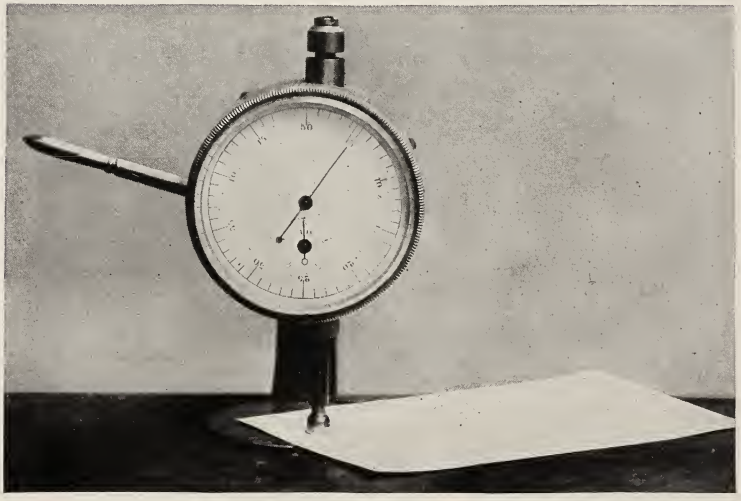

FIG. 4.—*Thickness tester*

The thickness of the paper is read off on a dial in thousandths of an inch

used for this purpose. It is obvious that the samples being weighed must be accurately measured to determine their size, and this is done by means of an accurate rule, graduated in tenths of inches. The following formula is of assistance, where a is scale reading, b is one dimension of the sample, c is the dimension at right angles to b, and d is the number of sheets of paper in the sample:

$$\frac{a \times 1000}{b \times c \times d} = \text{weight in pounds per ream } 25 \times 40\text{—}500.$$

For samples of paper weighing less than 20 on the quadrant scale a chemical balance is used. For convenience, the following formula is used:

$$\frac{(\text{Weight in grams}) \times (1.102) \times (1000)}{(\text{Area of sample in square inches}) \times (\text{number of sheets})} = \text{Weight in}$$

pounds per ream 25×40—500. To convert the weight of the standard ream to the weight of a ream of the desired trade size, it is only necessary to multiply the weight of the former by the area of the latter and divide by 1000, provided, of course, that the latter ream contains 500 sheets. This is illustrated in Table 3.

TABLE 3.—Typical Equivalent Weights in Standard and Trade Sizes

Weight of ream, 25 × 40—500	Trade size ream, 500 sheets	Area of sheet	Weight of ream, trade size
Pounds	Inches	Inches²	Pounds
52.6	25 × 38	950.0	50
64.2	17 × 22	374.0	24
100.0	20 × 25	500.0	50
156.0	22.5 × 28.5	641.3	100

This scale may be calibrated by placing small accurate weights in the pan and taking readings on the scale. An average of several readings at uniform distances apart on the scale should be obtained.

2. THICKNESS

There are a number of instruments or devices available for determining the thickness of paper. They are, usually, a spring micrometer with the dial graduated into thousandths of an inch, as shown in Fig. 4. It is not advisable to take readings much closer than half of one-thousandth (0.0005) of an inch. Some difficulty is experienced at times with the spring of the micrometer, and the needle or pointer should be adjusted to zero whenever it does not

properly return there. The thickness tester is calibrated by means of standard sheet metal leaf gages. Since the spring is not compressed to any great extent when testing most grades of paper, the error introduced by varying pressure exerted by the spring against the paper is not great. Thickness test is made on each of the 10 sheets of paper of which the test sample is composed, and an average is obtained which is reported as thousandths of an inch.

3. BURSTING STRENGTH

The bursting strength is the apparent pressure necessary to burst a hole in a sheet of paper, when the pressure is exerted against a definite area and the sheet is held taut by a clamp. There are several types of such instruments available, but there seems to be no definite relation between the data obtained with them. In any case, such an instrument is empirical and the data obtained with any one machine are relative. Various factors in the paper influence the test, such as kind and length of fiber, type or formation, weight, thickness, etc. One of the most important factors is the stretch or elongation of the paper under pressure or strain.

The type of instrument used at this time at this Bureau for this test is one in which a handwheel, actuating a piston, forces glycerin (glycerol) against a flexible diaphragm which transmits the pressure to the sheet of paper. The paper is held in place by an annular ring and the hydrostatic pressure within the chamber is indicated on a suitable gage, as illustrated in Fig. 5. A test is made on each of the 10 sheets of the test sample and an average of the gage readings is obtained. It is of considerable value in comparing the bursting strength of various samples of the same class, but of different weight, to reduce the bursting strength to a unit weight basis. This is done by dividing the bursting strength by the weight of the standard size ream (25×40—500), and the result or ratio when multiplied by 100 is reported for convenience as a percentage.

It is obvious from a study of such a device that there are several factors in its design and construction that may influence the value of the test and the data obtained. These are as follows: The age and condition of diaphragm, the pressure of the clamp (except in the case of the lever clamp type), the amount of glycerin in the cylinder, the speed and uniformity of rotation of the handwheel, and the accuracy of the gage. The flexible diaphragm deteriorates, whether used or not, when exposed to air and in contact with the glycerin, and is renewed every month. In this connection it is to be noted that the area of the diaphragm in contact with the paper at various pressures is not constant.

For this reason all results are recorded and reported as "points" (gage-scale divisions), rather than as pounds per square inch. Precaution should be taken to be sure that the cylinder is full of glycerin—that is, that there is no air present within the chamber, since otherwise the results are not reliable. It is important that the handwheel be turned at a uniform rate, and 120 rpm has been adopted as standard. The pressure gage is calibrated every month by means of a dead-weight tester, and corrections applied when necessary.

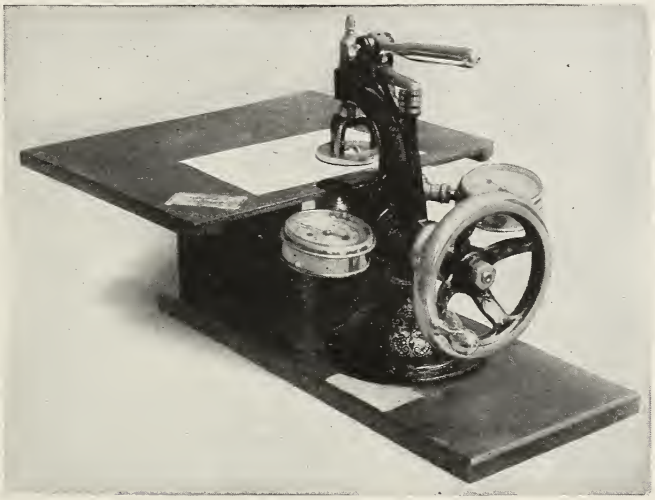

FIG. 5.—*Bursting strength tester*

The sample of paper is burst by means of pressure exerted through a rubber diaphragm which conforms to the shape of the paper

4. TENSILE STRENGTH

There do not seem to be any instruments of American manufacture built for the purpose of determining the tensile strength of paper. The apparatus illustrated in Fig. 6 is of foreign manufacture and is used, with some modifications, for testing both paper and textiles. A strip of paper 15 mm wide is clamped in the jaws, so that the distance between them is 90 mm. By means of a piston, hydraulically operated, the lower jaw is pulled away from the upper one, and the lever arm with the weight on the end is brought out at an angle until rupture of the paper takes place. A ratchet with pawls holds the arm at the point on the scale where the break occurred. This scale is graduated in fractions of kilograms and has a capacity of 50 kg. A secondary scale, midway up on the lever arm, gives the elongation of the strip of paper at the point of rupture.

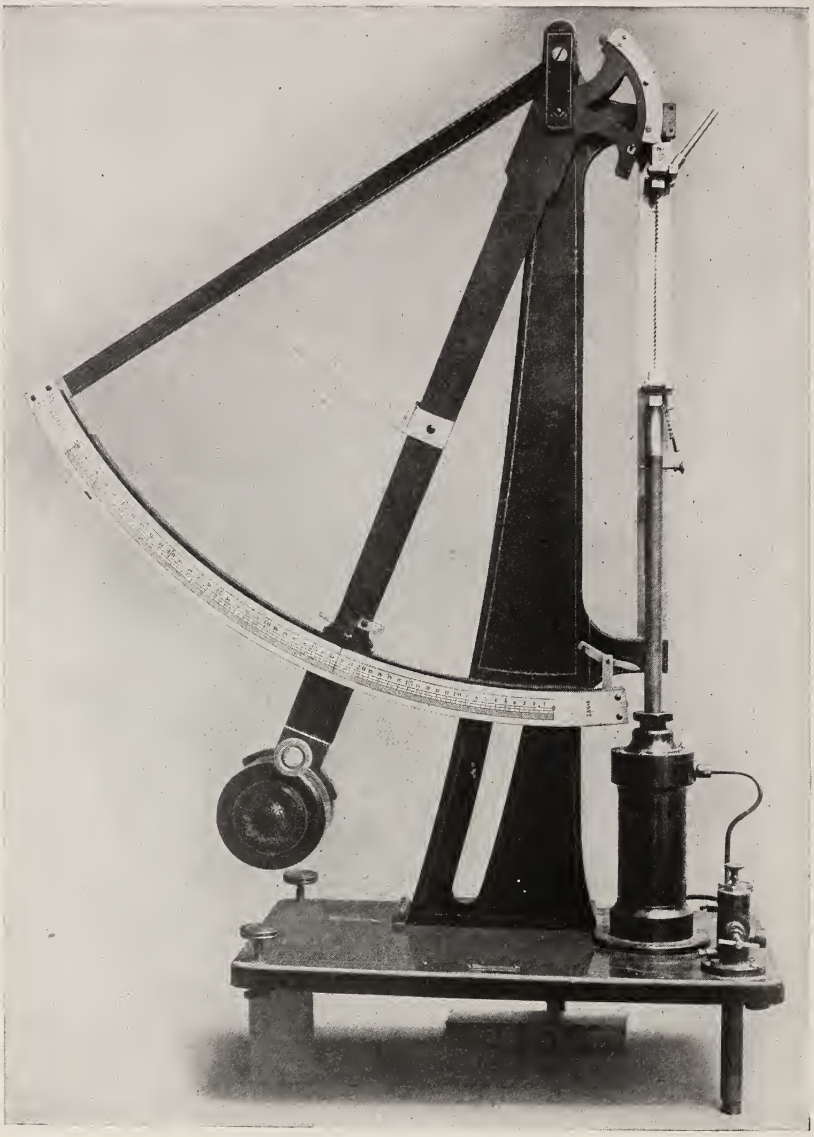

FIG. 6.—*Tensile testing machine*

By means of this instrument the tensile strength of paper and the elongation at rupture may be determined

In making the tensile test on paper, 10 test strips, each 15 mm wide and 150 mm long, are carefully cut from the test sample in both the "machine" and "cross" direction. An average of the results is obtained and is generally converted into pounds per inch of width of sample by multiplying by the factor 3.732. A useful strength factor is obtained by reducing the tensile strength to the "breaking length," or the length of paper which, if suspended by one end, would break of its own weight. This factor is obtained by the following formula:

$$\frac{\text{Tensile strength in pounds per inch width} \times 13\,889}{\text{Weight in pounds of ream } (25 \times 40 - 500)} = \text{Breaking length in yards.}$$

There are several factors of importance to be considered when operating this apparatus. The sample should be properly adjusted in the jaws, so that there is no uneven diagonal strain on the strip or incorrect alignment. This is sometimes difficult, as the upper jaw is not rigid. The test strip should be accurately cut along the "machine" direction of the test sample or at right angles to that, never diagonally across. The regulating valve of the water supply should be adjusted to permit a downward speed of 3 inches per minute of the lower jaw. It is obvious, however, that if two papers are tested at the same rate and one sample has twice the percentage stretch of the other, that the time to break will differ for the two papers. The apparatus is calibrated once a year by hanging accurate weights by a thread or light string from the upper jaw and determining the points on the scale which should correspond. The difference is the necessary correction.

5. FOLDING ENDURANCE

The folding endurance of a sample of paper is obtained by a machine which registers the number of alternate folds the sample will endure before breaking, while under a constant tension of 1 kg. The test strip, 15 mm wide and 90 mm long, is placed in the jaws and the apparatus is started. The strip is then folded flat upon itself, then opened and folded at the same line upon itself in the reverse direction, this being called a double fold. The number of double folds the sample will withstand before breaking is indicated on a dial and is reported as the folding endurance. Five tests are made in both the "machine" and "cross" direction of the test sample, and an average is obtained. A speed of 120 double folds per minute is maintained by means of a small electric

motor. This tester is illustrated in Fig. 7. It is to be noted, however, that the folding endurance test under tension seems to be more affected by atmospheric changes of temperature and relative humidity than most of the physical tests. The stretch of the paper is also a factor influencing the test. The calibration and standardization of the folding tester require much attention, since there are several moving parts that become worn. To obtain check results on different machines, it is necessary to check

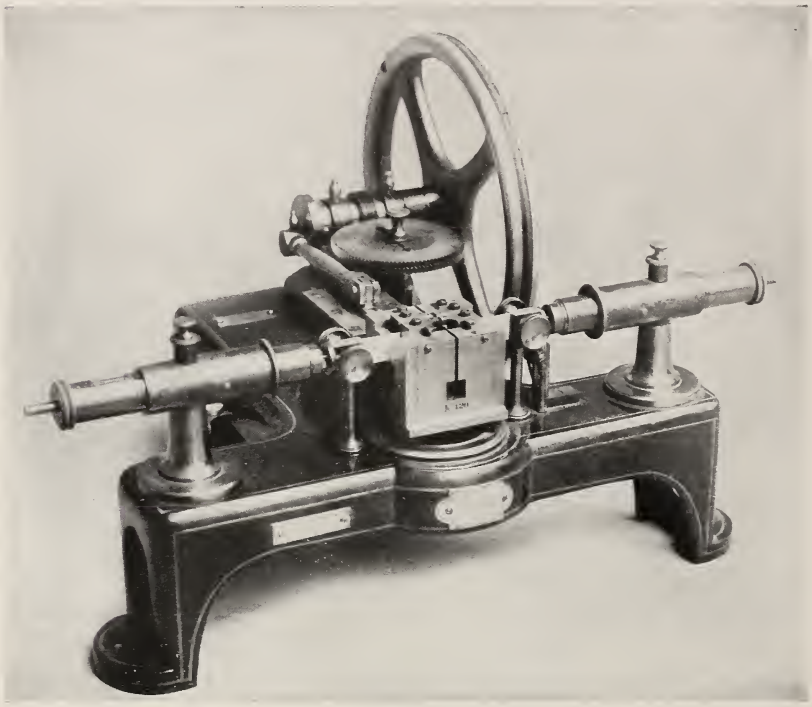

FIG. 7.—*Folding tester*

The folding endurance of paper under tension is determined by the folding of a strip of paper, and the number of double folds which the samples will resist is indicated on the dial

carefully the bearings, rollers, and the tension of the springs, and to have their dimensions all accurately the same.

6. TEARING STRENGTH

A method of determining the tearing strength of paper has become of considerable importance, and a number of devices have been proposed for this purpose. The following method has been adopted tentatively until the conclusion of further work. For this test the tensile testing machine as shown in Fig. 8 has been adapted, with the weight taken from the lever arm. Ten strips

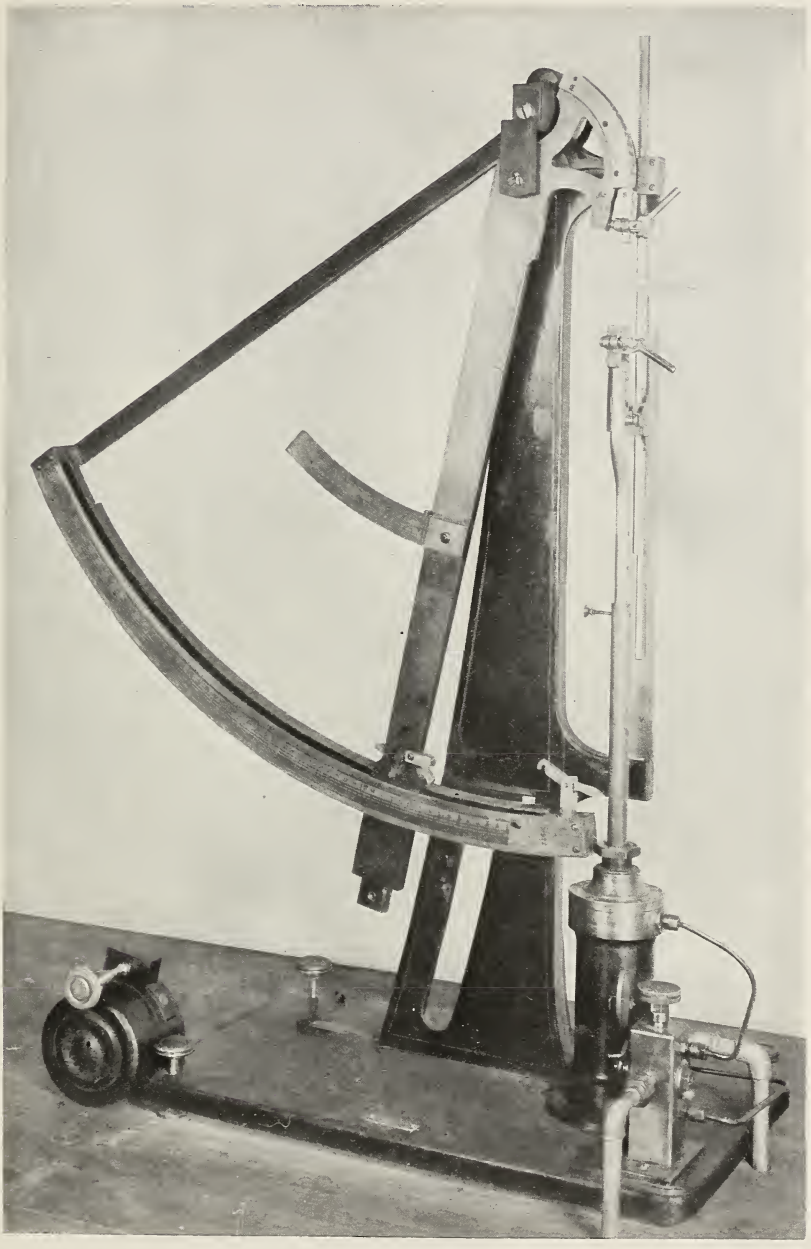

FIG. 8.—*Tearing tester*

This is an adapted tensile testing machine with the weights removed to give it more sensitiveness; it is used for tearing a strip of paper in determining its resistance to tear. There are other devices being developed for this purpose

1 inch wide are slit halfway down the middle and each half-inch end is placed in one of the jaws, the lower jaw being offset one-half inch as illustrated. A lineal downward speed of 3 inches per minute of the lower jaw is used, and the tearing strength is indicated as points on the scale. The apparatus must be calibrated with the weight off the lever arm, and a conversion curve may be plotted in order that the readings from the scale may be readily obtained in grams.

Ten tests are made in both the "machine" and "cross" directions, 10 sheets being used in each test to obtain an average. A maximum tearing strength in grams is obtained by this method, and the average of the 10 tests is reported as such. It is to be noted that with heavy or thick samples it is necessary to use a smaller number of sheets in each test sample, as there is a tendency for the tearing edges to rub or bind. This friction has been eliminated to a considerable extent by having the lower jaw offset, so that the tearing strain is parallel to the test strip. This test will probably be modified to some extent in the near future, as a maximum value is not always in agreement with service tests.

7. ABSORPTION

There are several methods for determining the absorbency of bibulous papers, and a study of these methods is now in progress to determine their relations. In the case of blotting papers the test should reproduce service conditions as much as possible, since not only the total absorption and the rate are important, but also the effect of repeated applications. In addition, the effect of ink in place of water during the test is to be considered. The method as given is used for testing the absorption of blotting paper, but is open to the criticism that the area of the cross section or thickness is not taken into consideration.

In making this test, using the "strip" method, a strip of blotting paper 15 mm (about three-fifths inch) wide and 150 mm (about 6 inches) long is suspended, as illustrated in Fig. 9, so that the lower end dips 3 mm (about one-eighth inch) into a pan of distilled water. Beside the strip is a scale reading in millimeters (fractions of inches), and at the end of each minute for 10 minutes readings are taken of the height to which the liquid rises in the strip. Five tests are made in both the "machine" and "cross" direction and an average obtained. The result is reported as the height to which the liquid will rise in 10 minutes. When necessary, or advisable, the same strips may be subjected repeatedly to the test, which will indicate the decreasing ability to ab-

sorb water or ink. In addition, a standard ink of the following formula may be used:

	Grams
Tannic acid	23.4
Gallic acid	7.7
Ferrous sulphate	30.0
Dilute hydrochloric acid (U. S. P.)	25.0
Phenol	1.0
Bavarian blue, S. & J. No. 478 or similar suitable dye	2.2

Water to make a volume of 1000 cc at 15.6° C.

FIG. 9.—*Absorption tester*

This is one method of testing blotting paper, and the height of rise of liquid is indicated on the two samples of paper, one on either side of the metric rule

The method as given above is adopted tentatively and will be modified or changed if future work makes it advisable. Other methods have been suggested and are being investigated, such as the method when water or ink is allowed to drop from a 1 cc pipette, or a sample is totally immersed in water or ink for a given time and the percentage amount absorbed determined.

8. TRANSPARENCY

It sometimes becomes advantageous, in order to more specifically evaluate the efficiency of two or more samples of envelope windows, tracing paper or cloth, glassine paper, etc., to assign a numerical value to the opacity or transparency of the various samples. This is particularly advantageous if the quality of two

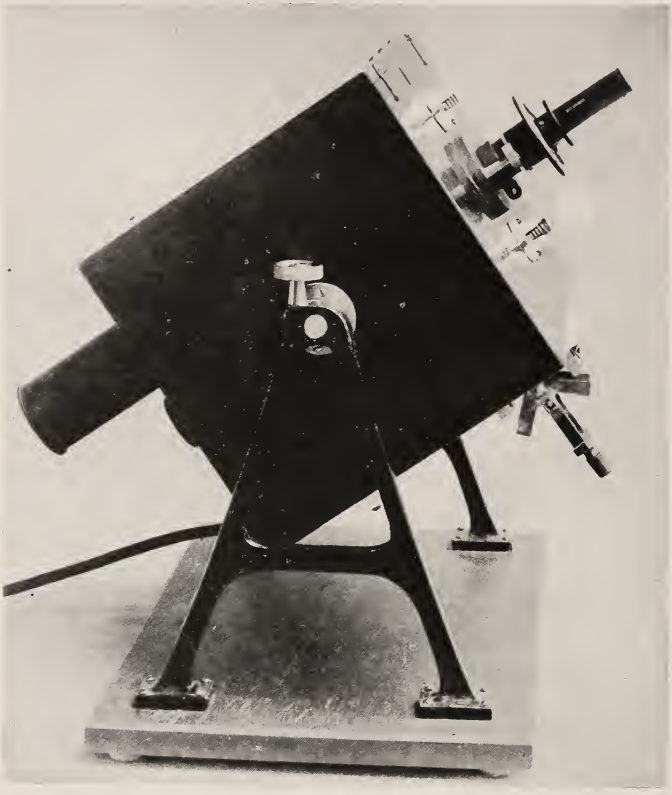

FIG. 10.—*Transparency apparatus*
This is used for determining the relative transparency and opaqueness of papers

similar papers is so nearly alike in this respect as to be indistinguishable by ordinary observation.

The Bureau has developed and adopted a standard method for determining the transparency of paper and tracing cloth, which is described in detail in B. S. Circular No. 63 and illustrated in Fig. 10. Briefly this method consists in placing a sample of the paper or cloth to be tested over two adjacent surfaces, one white and the other black, and measuring the reduction in contrast of the appearance of the two surfaces. If the material in question

is quite transparent, the contrast between the black and white surfaces as seen through the material will be quite noticeable; but if the material is opaque, none of the light incident upon its surface will be transmitted and absorbed by the black surface beneath and consequently there will be no contrast between the parts of the material covering the black and white surfaces.

In making the measurements one must use a photometer having a divided photometric field, one-half of which is illuminated by the light coming from the material over the white surface, while the other half is illuminated by the light coming from the material over the black surface. The two halves of the photometric field are then "matched" by visual observation and properly setting the photometer, and the indicated results are recorded. A simple computation based on these observations gives the numerical measurement sought, which is called the "contrast ratio." This varies between zero and unity, larger values indicating less transparency.

IV. CHEMICAL TESTING

The testing of paper to determine those constituents, other than fibrous materials, which may be harmful in excessive amounts is desirable, and for routine testing this is relatively simple. These constituents usually consist of sizing material, such as rosin, glue, or starch, and loading materials or fillers, such as clay, talc, and various other inert substances. There are other materials present in certain special papers which are not considered here.

1. LOADING MATERIAL

The determination of the amount of ash of the paper indicates the absence or presence of loading material, which is usually added for the purpose of giving the paper a smooth printing surface. Paper having an ash of less than 1.5 per cent usually has not had a filler added. In the case of all-rag papers the ash may be as low as 0.5 per cent, while with papers made from wood pulp it may be as low as 0.75 per cent. An ash greater than 1.5 per cent usually indicates that some loading or coating material has been added, except where old papers were used, or a pigment dye for coloring. The percentage of ash obtained includes nonvolatile and noncombustible materials from several sources, as follows: (*a*) Ash due to residual minerals in fibrous pulps, (*b*) ash due to loading material, (*c*) ash due to surface coating, and (*d*) ash due to mineral coloring materials or pigments and a slight residue from sizing.

A 1-gram sample of the paper, obtained by taking equal portions from each of the 10 sheets of the test sample, is weighed and placed in a nickel crucible. This is placed in an inclined position upon a triangle over a Bunsen burner, as illustrated in Fig. 11. The paper first scorches, then carbonizes and finally ignites. When the paper begins to burn, the crucible is removed from the flame and the paper is allowed to burn quietly. During the burning care must be taken that none of the ash is lost by air currents. To prevent this a cover may be placed partially over the crucible. After the burning is complete the crucible is then replaced on the

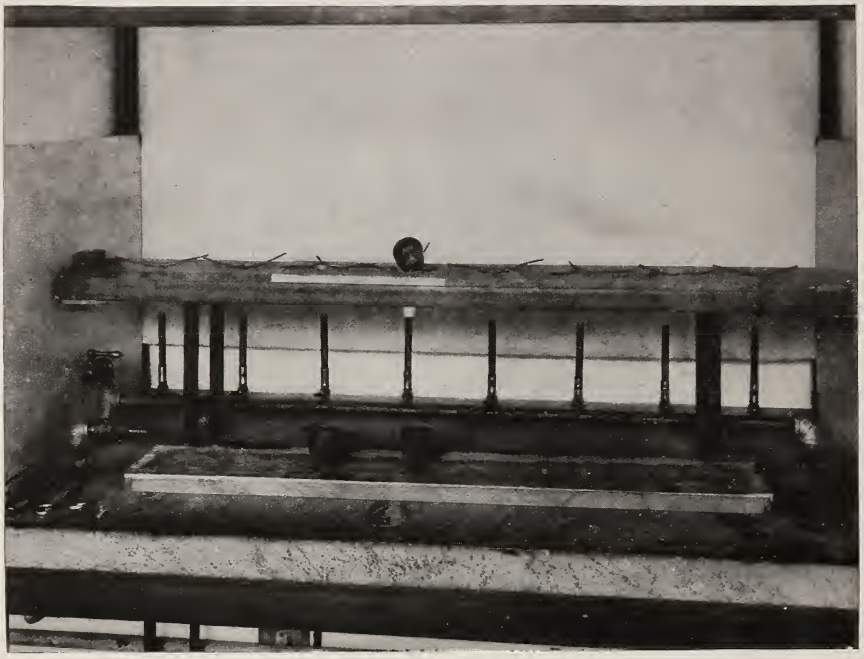

FIG. 11.—*Means of determining the loading in paper*

This illustrates a convenient way of determining the ash in paper when there are many samples daily, and shows the various positions of crucibles during the operation

triangle and the heat applied until the last traces of carbon have disappeared. In some cases it is necessary to use a blast lamp to consume the last traces of carbon. After the crucible has cooled the ash is transferred to a counterpoised aluminum pan and weighed. The result is obtained directly as a percentage. For extremely accurate work the moisture in the paper should be eliminated by drying at 100 to 105° C before weighing out the sample for test, and the ash in the crucible should be cooled in a desiccator, as a variation of 2 per cent in moisture content will give an error of 0.02 per cent for each per cent of ash.

To determine the kind of loading or coating material used, it is necessary to test the ash qualitatively, for which purpose at least 0.2g. of ash is desirable. Briefly, tests should be made for the substances indicated in Table 4, in which are also given the fillers that the presence of these substances would indicate.

TABLE 4.—Paper Fillers and Their Indicators

Substance sought	Filler indicated
Calcium sulphate	Crown filler
Calcium carbonate	Chalk
Barium sulphate	Blanc fixe
Magnesium silicates	Talc
Aluminum silicates	China clay

These fillers have various trade names and do not in all cases have definite chemical formulas, but the presence of any great amount of any of the materials in the first column would indicate the kind of filler used, and further confirmatory tests may be made.

2. SIZING

Nearly all grades of paper, except blotting, filter, and similar papers, have a sizing material added to give certain properties to the paper, including ink resistance. The sizing material is added during the preparation of the stock for the paper machine and, in some cases, is added after the paper is made by passing the sheet through a bath or tub containing glue, treated starches, or other similar material. In the first case rosin is used, and the paper is said to be "engine-sized"; in the latter case the paper is "tub-sized." It is desirable to determine the presence and amount of these sizing materials, and this is done by chemical analysis. It is not necessarily true that the percentage of sizing material will indicate the resistance of the paper to ink; for this purpose other methods are being developed.

(*a*) Total Resins.—The following methods are used for determining total resins: Five grams of paper are cut in strips about one-half inch wide from the 10 sheets of the test sample and are folded into numerous small crosswise folds. These are placed in the siphon cups and acidulated alcohol (900 cc of 95 per cent alcohol, 95 cc distilled water, and 5 cc glacial acetic acid) is poured into the cup until it starts to siphon over. When this has entirely siphoned over into the shell-shaped extraction flasks, approximately 20 cc more of the acidulated alcohol is added to the siphon

cup, which is then hung on the condenser, as illustrated in Fig. 12. The flask is set into the steam bath and the extraction proceeds for about 2 hours, or until the cup has filled and siphoned over about ten times. The extract is poured into a weighed glass dish and the flask washed out with a small portion of the acidulated alcohol, which is added to the original extract. The alcohol is then evaporated, the dish dried and weighed, and the increase in weight of the dish divided by 5 will give the percentage of total resins.

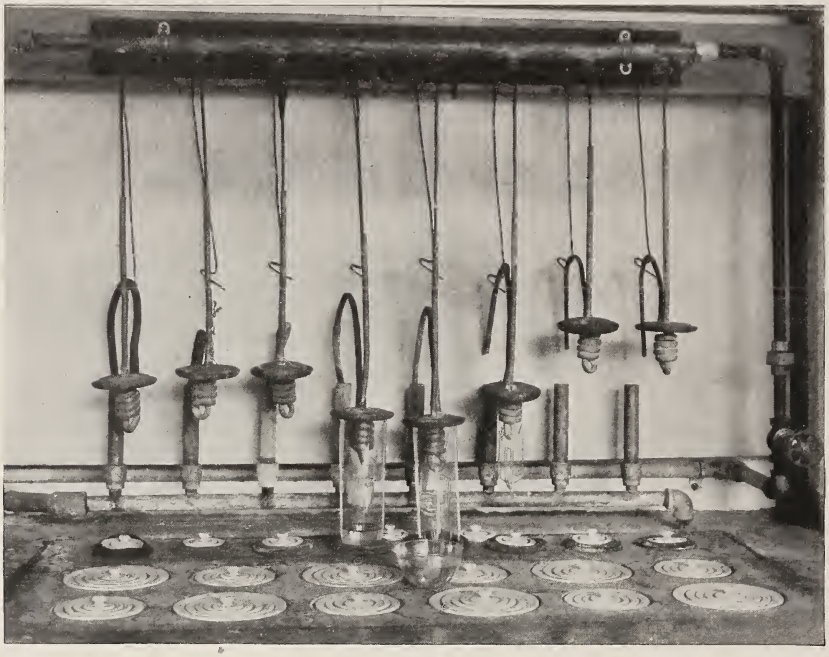

FIG. 12.—*Rosin sizing extractor*

This illustrates a convenient form of apparatus for determining the amount of rosin sizing in paper and shows the type of extraction thimbles and condensers. The chief criticism of this is that it is difficult to save the solvent, and where expensive solvents are used this form is not practical

For additional accuracy other than for routine testing or control work, the following additional method is advisable:

Just before the extracted total resins in the glass evaporating dish, in the method given above, go to dryness, they are cooled and taken up with 25 cc of ether, and transferred to a 250 cc separatory funnel containing 100 cc of distilled water and 25 cc of a saturated solution of sodium chloride, the latter being present to prevent the formation of an emulsion. The funnel is shaken thoroughly and the contents allowed to separate. The water is drawn off into a second separatory funnel, and this is washed

again with 25 cc of ether. The two ether extracts are combined and washed two or three times with 100 cc of distilled water to remove all the salt and foreign matter other than ether-soluble resins. Should glue which is extracted from the paper by the alcohol interfere by emulsifying with the ether, it may readily be removed by adding a strong solution of sodium chloride to the combined ether extracts, shaking thoroughly and draining it off, repeating if necessary before washing with distilled water. The washed extract is transferred to a weighed glass dish, evaporated to dryness slowly, dried in an oven at 95 to 100° C for one-half hour, cooled, and weighed. The increase in weight of the dish divided by 5 will give the percentage of total resins.

A qualitative test for rosin may be made by the following method: A small portion of the paper boiled with 5 cc of acetic anhydride in a test tube, cooled, and treated with a few drops of concentrated sulphuric acid added so as to slip smoothly down the side of the test tube, will develop a pink ring if rosin is present. The cover of a porcelain crucible may also be used, and in this case a drop or two of each of the liquids are brought together by glass rods. A pink color may develop where the two come together.

(*b*) STARCH.—Starch is present to some extent in some papers, and this may be indicated by placing a few drops of a pale yellow dilute solution of iodine in potassium iodide on the sample of paper. The development of a blue color indicates the presence of starch. If it is possible to extract a sufficient amount of the starch from the paper by warming with water, a few drops of the solvent containing the starch is placed on a slide, dried, and examined under the microscope. In this way the kind of starch used may be determined by the shape and markings of the granules, provided the starch was not sufficiently cooked to destroy the grains. For the quantitative test for starch, the starch may be converted into dextrose by treating a few grams of paper in water with dilute sulphuric acid and estimating the amount of dextrose present in the extract by Fehling's solution.

(*c*) GLUE.—The presence of glue in paper may be indicated by either of the following methods: (1) The extract obtained by boiling 1 g of paper in water is decanted into a test tube, cooled, and treated with a few cc of ammonium molybdate solution, followed by a few drops of nitric acid. The presence of glue is indicated by a white amorphous precipitate. (2) The extract obtained by boiling 1 g of paper in water is decanted into a test tube, cooled, and treated with a few cc of tannic acid solution of

5 per cent concentration. The presence of glue is indicated by a heavy flocculent precipitate, which, when heated, becomes coagulated and hard.

Glue may be determined quantitatively by treating 3 to 5 grams of paper by the Kjeldahl method and thus determining the amount of nitrogen present. The percentage of nitrogen obtained multiplied by 5.6 will give the percentage of glue in the sample of paper. This test, however, is of no value when both glue and casein are present, since both contain nitrogen.

V. MICROSCOPICAL TESTING

In the examination of paper to determine its quality, it is necessary to have a means of identifying the fibrous materials from which the paper is made, since some fibers do not deteriorate as rapidly as others. These materials are chiefly as follows: Rags; chemical wood pulp, consisting of coniferous and deciduous or broad-leaf woods; mechanical or ground-wood pulp; manila and jute; and straw. These occur in some cases separately but more often in combination. In addition to identifying the presence of these various fibrous materials, it is necessary to determine the amount of each present in the paper. Since, however, the determination of rag in a paper, for instance, does not indicate the grade of rag used in the manufacture of the paper, specifications built up primarily about the fiber content of the paper are not always the best.

1. PROCEDURE

It is important to obtain a representative sample, and for that reason a small corner, about as large as a penny, of each of the 10 sheets of the test sample is cut off and torn into small pieces. These are placed in a 50 cc beaker and approximately 20 cc of a 0.5 per cent solution of caustic soda is added. The beaker is placed on a hot plate or stove and brought to a boil, but the boiling is not continued for more than a minute or two. The liquor is drained off and the pieces of paper are washed several times with tap water, care being taken that none are lost. They are then washed with approximately 20 cc of 0.5 per cent solution of hydrochloric acid, and then washed twice more with tap water. A portion of the small pieces is rolled between the fingers into a pill about the size of a small pea and transferred to a test tube, which is half filled with water. The test tube is placed in a shaking machine, as illustrated in Fig. 13, and shaken until the paper is disintegrated into fibers, which normally takes about one-half minute. By means of a glass tube of about 5 mm internal diameter

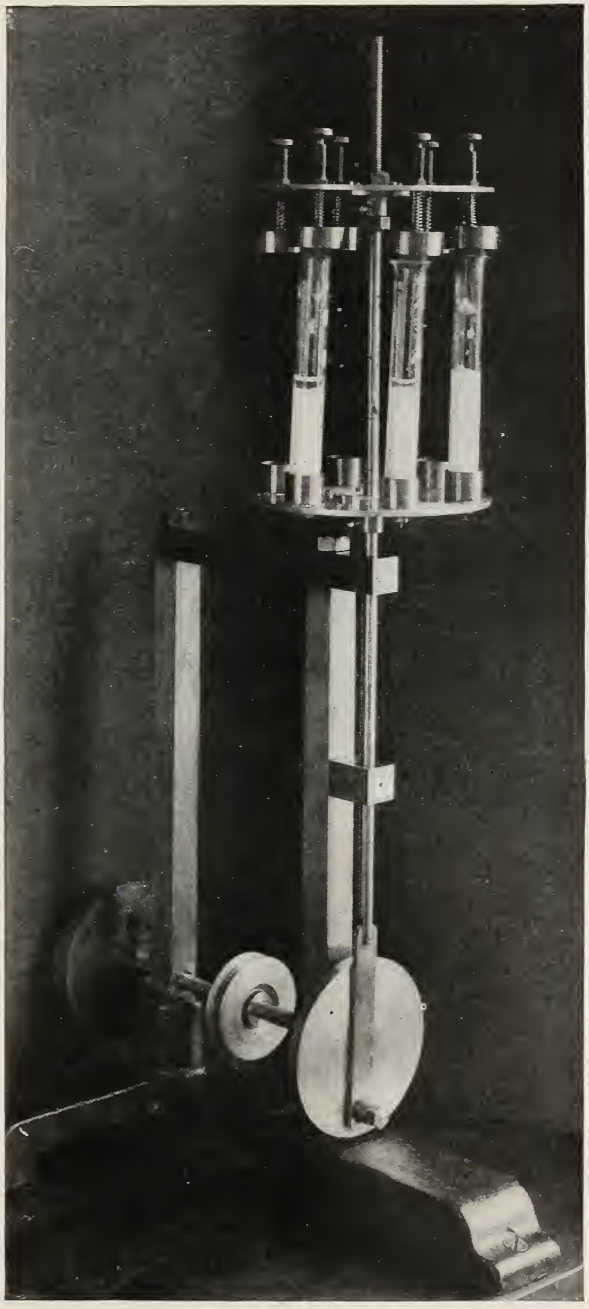

FIG. 13.—*Test tube shaker*

This device quickly disintegrates the samples of paper into fibers and eliminates all the necessity of doing it laboriously by hand

a representative amount of the fibers is transferred to a glass slide and the water removed by placing several thicknesses of hard filter paper on either side of the slide. After the excess water has been thus removed, a strip of filter paper is placed on the fibers to absorb any traces, leaving the fibers dry but not so dry that there will be difficulty in separating them. Two or three drops of the zinc-chloride-iodine stain described below are placed on the fibers, which are teased out with steel needles until the fibers are reasonably uniformly distributed and free from knots of fibers.

COMPOSITION AND PREPARATION OF ZINC-CHLORIDE-IODINE STAIN[2]

25 cc saturated zinc chloride solution at 70° F
5.25 g potassium iodine
0.25 g iodine
12.5 g distilled water

The three last ingredients are mixed together and the zinc chloride added. The insoluble matter is allowed to settle at least overnight and the supernatant liquid is decanted off. The stain must be kept in the dark or in a bottle opaque to light. It may be made up in larger quantities, provided it is kept from the light and air.

After the fibers are properly teased out with the needles, another but thinner glass slide is placed on top of the first slide, and the excess stain is squeezed out and absorbed by blotting paper. The slide is now ready for the microscope, which is of the binocular type, using artificial illumination as indicated in Fig. 14, and having a magnification of about 50 diameters. Care must be observed to keep the slide away from the light before using it for the estimation, and in any case the estimation should be made at least within one hour from the time the slide was made up. Under the microscope, using the zinc-chloride-iodine stain, the fibers appear colored, as follows:

Cotton and linen rags	Wine red
Cooked and bleached manila	
Cooked and bleached wood pulp	Blue to blue violet
Cooked and bleached straw and esparto	
Mechanical or ground wood pulp	Yellow to lemon yellow
Jute, uncooked, and manila	
Highly lignified fibers	

In making the estimation both the distinctive shape and markings of the fibers, as well as the colors as developed by the zinc-chloride-iodine stain, are of importance. Extreme care must be observed to have all beakers, test tubes, needles, and even fingers free from any fibrous material before beginning the preparation of a sample or slide.

[2] This formula is tentative. Its value is subject to a number of factors that are now being studied. Additional information may be obtained from the paper section, Bureau of Standards.

2. ESTIMATION

For general work and routine testing, the estimation method has been adopted. This consists primarily of properly judging or estimating the relative proportions of fibers present, after studying various fields of the slide. For this purpose standard samples of known content are essential, and there are available here most of the more common commercial mixtures of fibrous materials, made up under rigid conditions. Since the accuracy of this method depends largely on the experience of the operator, and since there

FIG. 14.—*Binocular microscope and daylight lamp*

For estimating and examining fibers it is desirable to have constant light conditions, and the use of a binocular microscope is of assistance when there is much continuous estimating work

is a tendency to judge or estimate by appearance or area, it is most necessary, even for the experienced operator, to refer in many cases to the standard samples. This is especially true in the case of ground wood, since the specific gravity of this material is not the same as that of rag pulp or chemical wood pulp, and if standard samples are not used low estimations are obtained. It is to be noted that the percentage of various fibrous materials present is given on the basis of the total fiber content alone and nonfibrous

materials are not considered. There are other methods besides the one given for determining the amounts of various fibrous materials in paper, but most of these are not sufficiently rapid for routine testing, and it is a question whether their accuracy is much greater.

3. MICROPHOTOGRAPHY

It is often necessary to have a permanent record of the fiber content of paper and also to have an opportunity of studying the characteristics of the various fibers. For this purpose a complete photomicrographical equipment is available, and Figs. 15, 16, 17, and 18 illustrate the distinctive markings of various fibers. This field of research has not yet been sufficiently developed, but it opens considerable opportunity. The methods employed are those commonly used with such equipment, except that a 20-foot bellows permits of a flatter field on the photographic plate and greater definition. The use of monochromatic light is also of great assistance.

VI. BIBLIOGRAPHY

Most of the data and information on paper testing have been published as articles in magazines and periodicals. The books referred to are those most available at the present time. The periodicals given are those of greatest circulation with the technical men of the industry, but there are others containing at intervals articles on paper testing. The results of investigations of Government laboratories have generally been published in the trade periodicals. Reference is also made to the secretary of the Technical Association of the Pulp and Paper Industry at 542 Fifth Avenue, New York City, N. Y.

1. BOOKS

Sindall, R. W., Paper technology, 253 pp., 13 plates, 158 illustrations; 1906. London, Charles Griffin & Co. An elementary manual on the manufacture, physical qualities, and chemical constituents of paper and of paper-making fibers.

Cross, C. F., and E. J. Bevan, A text-book of papermaking, 507 pp., 16 plates, 99 illustrations, 9 pp. bibliography; 1916. London, E. and F. N. Spon (Ltd.). Contains a chapter on paper testing.

Herzberg, Wilhelm, Papierprüfung (Paper testing), 146 pp., 65 cuts, 16 plates; 1902. Berlin, Verlag von Julius Springer. An introduction to the study of paper.

Klemm, Paul, Handbuch der Papierkunde (Handbook of paper technology) 00, 4 pp., 130 cuts, 3 colored plates; 1910. Leipzig, Th. Grieben's Verlag. With references and instructions concerning the use, manufacture, testing, and selling of paper.

2. PERIODICALS

Paper, Paper (Inc.), 251 West Nineteenth St., New York City, N. Y.

Paper Trade Journal, Lockwood Trade Journal Co., 10 East Thirty-ninth St., New York City, N. Y.

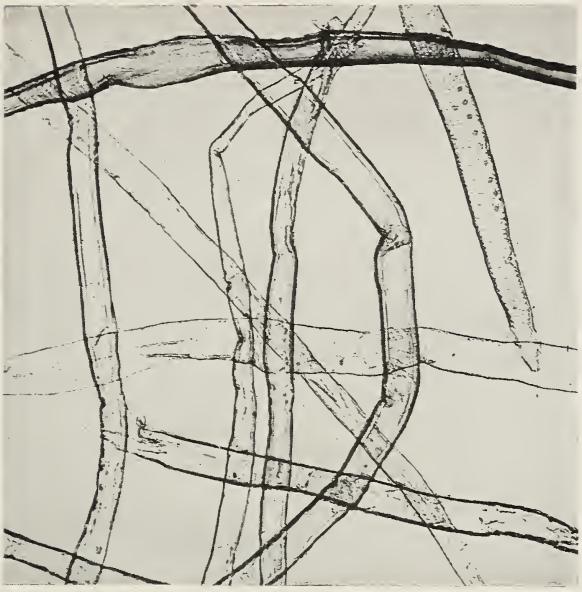

FIG. 15.—*Microphotograph of Douglas spruce fiber (Pseudotsuga taxifolia).* × 100

FIG. 16.—*Microphotograph of sugar or hard maple fiber (Acersaccharum).* × 100

FIG. 17.—*Microphotograph of fibers from rag pulp.* × 100

FIG. 18.—*Microphotograph of spruce ground wood fiber (Picea canadensis).* × 100

The Paper Industry, E. B. Fritz, publisher, 356 Monadnock Block, Chicago, Ill.

Pulp and Paper Magazine of Canada, Industrial and Educational Publishing Co. (Ltd.), Garden City Press, St. Anne de Bellevue, P. Que., Canada.

N. B.—For reference to special articles on paper testing appearing in these periodicals, the Committee on Paper Testing and the Committee on Bibliography, both of the Technical Association of the Pulp and Paper Industry, 542 Fifth Avenue, New York City, N. Y., and the Bureau of Standards, Washington, D. C., should be consulted.

3. GOVERNMENT PUBLICATIONS

The testing of materials, B. S. Circular No. 45, 89 pp.; 1913. Washington, Government Printing Office. Contains description of tests made on paper, see pp. 59–62.

Specifications and tests for transparency of paper and tracing cloth, B. S. Circular No. 63, 8 pp.; 1917. Washington, Government Printing Office.

Sammit, C. Frank, A measurement of the translucency of paper, Department of Agriculture, Bureau of Chemistry Circular No. 96, 3 pp.; 1912. Washington, Government Printing Office.

Sammit, C. Frank, The detection of faulty sizing in high-grade papers, Department of Agriculture, Bureau of Chemistry Circular No. 107, 3 pp.; 1913. Washington, Government Printing Office.

Surface, Henry E., Bibliography of pulp and paper industries, Department of Agriculture, Forest Service Bulletin No. 123, Forest Products Laboratory Series, 48 pp.; 1913. Washington, Government Printing Office.

VII. REGULATIONS REGARDING TESTS

The Bureau is equipped to make complete examination of papers for Government offices or bureaus, and will, in its discretion, perform similar service for the general public, when asked to act as referee or where the nature of the case calls for an authoritative test. The Bureau thus reserves the right to accept or decline requests for tests, depending upon the conditions in each instance.

In order that the user may more correctly judge a paper and determine its usefulness for the purpose intended, he should be informed upon the following points:

(*a*) Weight per standard ream
(*b*) Thickness
(*c*) Bursting strength
(*d*) Tensile strength
(*e*) Folding endurance
(*f*) Tearing strength
(*g*) Absorption
(*h*) Amount and kind of loading material
(*i*) Amount and kind of sizing material
(*j*) Fiber composition

1. FEES

All tests for the National and State Governments are made free of charge. For municipal governments and private parties charges are made according to nature and extent of the work

required. Where such tests are accepted the fees will, in general, be based upon the following schedule, which states the fee for each test on a single sample and the charge for the same test on each additional sample, where several are submitted at the same time.

FEE SCHEDULE 212.—Paper Materials

Test	First sample	Each additional
212*a*. Weight determination	$1.00	$0.75
212*b*. Thickness determination	.50	.50
212*c*. Bursting strength test	1.00	.75
212*d*. Tensile strength test	2.00	1.50
212*e*. Folding endurance test	2.00	1.50
212*f*. Tearing strength test	2.00	1.50
212*g*. Absorption test	2.00	1.50
212*h*. Ash content determination	1.00	.50
212*i*. Total resin determination	2.50	2.00
212*j*. Fiber composition determination	3.00	2.00

All samples of paper should be put up for shipment carefully protected by cardboard or other material to insure arrival in good condition. All paper samples themselves should be plainly marked "For Paper Section, Bureau of Standards, Washington, D. C."

A careful record should be kept by the sender, so that the identification marks may be used in the certificate to avoid repeating the detailed description of the paper. Samples submitted by Government departments should be marked in upper left-hand corner of sheet as follows:

Date— Identification marks.
Submitted by:
Kind of paper,
Item number,
Name of manufacturer,
Tests for compliance with schedule,
Remarks: (Any other information concerning the paper).

Samples submitted by private parties, mark in upper left-hand corner of sheet as follows:

Date— Identification marks.
Submitted by:
Kind of paper,
Use of paper,
Ream size and weight,
Test required,
Remarks: (Any other information concerning the paper).

As soon as the fee is assigned to the test, a bill is sent at once, and payment should be in advance, made by money order or check drawn to the order of the "Bureau of Standards." Results of tests are not certified until fees are paid.

2. SAMPLING

The following should be noted in regard to submitting samples for test:

Whenever possible, 10 sheets of each sample, accurately cut 12 by 10 inches, should be submitted. These sheets should be picked out in such a manner as will most nearly represent an average of the total amount in question. The 10 sheets of each sample should be carefully fastened together, and one outside sheet should be marked as indicated above.

In order to secure representative samples for test, it is advisable to proceed as follows:

Case lots.—Select one case from each one-third of the paper in question, and then select one sheet from each one-third of each case. The tenth sheet should be taken from a fourth case. After cutting to the proper size for forwarding for test, the remainder of the sample should be saved for record purposes. Paper in "frames" and bundles should be sampled in the same way. In the case of small boxes or packages, when possible, a sheet should be obtained from each of 10 boxes.

Rolls.—Sample by taking 1 sample from each of 10 rolls not less than 4 sheets in from the outside of the roll.

WASHINGTON, September 18, 1920.