NATIONAL BUREAU OF STANDARDS · DEPARTMENT OF COMMERCE

NBS TECHNICAL NOTE 869

U.S. DEPARTMENT OF COMMERCE / National Bureau of Standards

Temperature Section Activity Summary, 1974

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Office of Standard Reference Data — Office of Information Activities — Office of Technical Publications — Library — Office of International Relations.

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[1] Headquarters and Laboratories at Gaithersburg, Maryland, unless otherwise noted; mailing address Washington, D.C. 20234.
[2] Part of the Center for Radiation Research.
[3] Located at Boulder, Colorado 80302.
[4] Part of the Center for Building Technology.

Temperature Section Activity Summary, 1974

J. F. Schooley, Editor

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Washington, D.C. 20234

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J. F. Schooley, Editor

This report summarizes the progress in calendar 1974 of the technical program of the Temperature Section of the Heat Division, Institute for Basic Standards. Separate sections of the report are devoted to the various projects, to highlights of the year's activities, and to other agency interactions.

Key words: Annual report; calibrations; progress report; standards; temperature scale; thermometry

I. Introduction

This report is written to enable the Temperature Section to report some of the accomplishments arising from its activities in calendar 1974, and to summarize the work of its projects, many of which are long-term activities for which there exist no current formal publications of results. Individual portions of the report, written by the various project leaders in the Temperature Section, follow a general editorial discussion of the year's highlights. A concluding section contains a summary of thermometry projects applied directly to the needs of other government agencies.

II. Section Highlights - J. F. Schooley, Chief

The new year brought a change in leadership to the Temperature Section. R. P. Hudson, Chief of the Heat Division, acceded to H. H. Plumb's long-standing desire to return to a more active scientific role in the Section's activities, thus bringing to an end an era that began in October, 1966.

Dr. Plumb had been appointed Chief of the Temperature Section to succeed J. F. Swindells, who, in turn, had been named Assistant Chief of the Heat Division for Thermometry. The appointment came just after the National Bureau of Standards laboratories were moved to the present Gaithersburg site from their former location at Connecticut and Van Ness Streets in Washington, D. C. Besides overseeing the "settling in" process for the Temperature Section, Dr. Plumb was heavily involved in his first year as Chief in preparing for the 1967 meeting of the International Advisory Committee on Thermometry (CCT). During the course of that meeting, which was held in part at the National Bureau of Standards and in part at the National Research Council laboratories in Ottawa, the CCT approved a new International Practical Temperature Scale. This scale was recommended for adoption in the following year by the International Committee for Weights and Measures, and is now known as the IPTS-68.[1]

In 1971, Washington, D. C. was the site of the Fifth International Symposium on Thermometry, and Dr. Plumb served both as the General Chairman of the Program Committee for the Symposium and as Editor-in-Chief of the Proceedings, which were published as a three-volume work in 1972.[2]

Under Dr. Plumb's leadership, the Temperature Section gained both stature and versatility. In 1966, over 50% of the Section's effort was devoted to calibration activities; this figure is now below 20%. Projects have been initiated in calorimetric measurement of thermodynamic temperatures, in medical thermometry, and in a continuing study of the national temperature measurement system. The Section is presently represented on two of the four working groups of the CCT which were in existence in January, 1974, as well as on the temperature committees and subcommittees of both the American Society for Testing and Materials and the American National Standards Institute.

Although Dr. Plumb is once again actively collaborating with G. Cataland in the laboratory, his advice and influence will continue to be reflected in the course of the program of the Temperature Section.

The new Chief of the Temperature Section, J. F. Schooley, was assigned the position of Deputy Chief under H. H. Plumb in March, 1973, with his principal duty being the direction of the study of the National Temperature Measurement System. Formerly a member of the Cryogenic Physics Section, Dr. Schooley had spent some ten years studying the phenomenon of superconductivity. He has been an NBS employee since 1960.

Several of the highlights of the year's activity in the Temperature Section appear in the Minutes of the May, 1974 meeting of the CCT at the International Bureau of Weights and Measures (BIPM) in Sèvres, France.[3] The National Bureau of Standards was represented at that meeting by R. P. Hudson, Chief of the Heat Division, and by H. H. Plumb. Technical contributions were submitted by staff members of the Cryogenics Physics Section, of the Equation of State Section, and of the Temperature Section. A summary of the Temperature Section contributions follows:

1) A brief discussion of the cryostat and methods used in the calibration of capsule platinum resistance thermometers from 13.81 K to 90.188 K;

2) recent gas thermometry results which provide information on the differences between the IPTS-68 and thermodynamic temperatures from the triple point of water to 415 K (142 °C). Working Group 3, of which L. A. Guildner is a member, reported it;

3) a brief description of apparatus, currently under construction, for the comparison of standard platinum resistance thermometers (SPRT's) from 90 K to 900 K;

4) the results of a study of the freezing temperatures of six experimental aluminum freezing-point cells; and

5) editorial suggestions regarding changes in the text of the IPTS-68. Consideration of possible revisions of the text was the particular duty of Working Group I of the CCT, of which H. H. Plumb is a member.

The CCT meeting was quite successful from several points of view; technical work at NBS was relevant to the Committee's deliberations in many areas, the quality of the work was praised by the Committee members, and, most satisfying, the actions taken by the CCT reflected in large measure the influence of technical progress at the NBS.

There were other notable features of the year's activities in the Temperature Section, as well. A semi-final draft report on the study of the National Temperature Measurement System was submitted to the Institute for Basic Standards. This study has been accomplished by many staff members of the Heat Division under the leadership of J. F. Schooley; it is an attempt to delineate the thermometry activities of the United States, both as to extent and as to capabilities, with special attention to the way in which NBS can continue to provide assistance and, in some areas, leadership. In its final form, the report will become part of a series of publications through which the Institute for Basic Standards will portray the effectiveness of the NBS in fulfilling its metrological functions throughout the United States' science and technology.

As usual, the Section offered a week-long seminar on precision thermometry. The attendance, however, was more than double the usual number. Some twenty-seven participants crowded the calibration laboratories during the week of 10-14 March. In addition to providing both that seminar and a three-hour thermometry session which formed a part of a local university's short course in standards laboratory operation, the calibration staff tested over one thousand precision thermometers and answered a like number of written and verbal inquiries on thermometry to round out a very satisfactory year.

The Section's work in thermocouple thermometry included the publication of the long-awaited Thermocouple Reference Tables, Monograph 125, and a highly successful study of a new high-temperature nickel-silicon-chromium thermocouple system developed in Australia.

The low-temperature acoustic thermometer program, buoyed by the return to full-time research status of H. H. Plumb to assist his colleague G. Cataland, yielded results of thermodynamic measurements of the transition temperatures of two elements of the NBS superconductive fixed-point device.

The medical thermometry program, begun only a year ago but now the largest in the Section, has made rapid progress toward its goal of improving the nation's medical care by improving the quality of thermometry in medicine. New precision clinical laboratory thermometers were developed, equipment and procedures for characterization of thermistors and small platinum resistance thermometers were completed, collaborative work on an environmental scanning radiometer and on thermometry in medical diathermy was accomplished, and exchange of equipment and ideas took place between the medical thermometry group and staff members of hospitals, clinics, and medical instrument manufacturers.

Closely allied to the field of medical thermometry, as well, is a new project begun in the past year by W. S. Hurst. Partially funded by the National Institutes of Health, this project will be an experimental study of thin-film capacitance thermometry.

Relevant Documents

1) "The International Practical Temperature Scale of 1968", Metrologia 5, 35 (1969).

2) "Temperature", Vol. 4, H. H. Plumb, Editor-in-Chief, Instrument Society of America, Pittsburgh, 1972.

3) The minutes are published (in French) by the International Bureau of Weights and Measures, Pavillon de Breteuil, F 92310, Sèvres, France.

III. Calibration of Contact Thermometers - J. F. Schooley

1. Introduction

The NBS offers calibration services in many areas. These services are summarized in NBS Publication 250 "Calibration and Test Services available at the NBS"; the Temperature Section collaborates in this activity by offering a variety of both regular and special thermometric calibrations. Liquid-in-glass thermometers, thermocouples, and platinum thermometers receive by far the bulk of these calibrations; however, tests which involve unusual temperature ranges or conditions, or unusual thermometers, are often performed. Like the regular calibrations, these tests are billed on the basis of labor and materials actually expended.

All calibrations are referred to the IPTS-68 by means of carefully preserved platinum resistance and platinum-rhodium thermocouple thermometers used in combination with a variety of fixed-point cells and comparison devices.

In addition to the calibrations themselves, the staff provides an annual Precision Thermometry Seminar which is attended by standards laboratory personnel from other government agencies, from industry, or from foreign countries.

Finally, the staff continually monitors its own capability to disseminate accurate thermometry as well as that of other laboratories through the medium of Measurement Assurance Programs (MAP's). These programs are currently underway in several areas.

The calibration of small, non-standard thermometers such as thermistors and small, commercial resistance temperature detectors (RTD's) are presently accomplished only on a research basis, and this work is done within the Medical Thermometry group. As the characteristics of these thermometers become better known, however, regular calibrations may well be offered through the calibrations program and listed in the SP 250.

2. Results

a. Thermometers calibrated*

Liquid-in-glass	800
Thermocouple	200
Platinum resistance	70

b. Attendance at 1974 Precision Thermometry Seminar

State Weights and Measures Offices	4
Industrial Laboratories	12
Government Laboratories	8
Foreign Countries	3

c. Two-week special training session for a Brazilian National dedicated to forming a national standards laboratory.

3. Relevant Documents

a. "The International Practical Temperature Scale of 1968", Metrologia 5, 35 (1969).

b. "Platinum Resistance Thermometry", J. L. Riddle, G. T.Furukawa, and H. H. Plumb, NBS Monograph 126, April 1973.

c. "Methods of Testing Thermocouples", W. F. Roeser, S. T. Lonberger, NBS Circular 590, February 1958.

d. "Thermocouple Reference Tables Based on the IPTS-68", NBS Monograph 125, Mar 1974.

e. "Calibration of Liquid-in-Glass Thermometers," NBS Monograph 90, Feb 1965

4. Personnel associated with the Calibration Programs

J. F. Schooley, Project Leader
J. A. Wise, Liquid-in-Glass Calibrations
G. T. Furukawa, Platinum Resistance Thermometry, Project Leader
J. L. Riddle and W. R. Bigge, Platinum Resistance Thermometer Calibrations
G. W. Burns, High-Temperature Thermocouples, Project Leader
M. G. Scroger, Thermocouple Calibrations

Total man-years of effort - 3.0.

IV. Medical Thermometry - B. W. Mangum

1. Introduction

There is a well-documented need for accurate and reliable measurement and control of

*Since the accounting is done on a Fiscal Year basis, the data are for FY 74.

temperature in clinical laboratories, in intensive care units, in surgery, in certain areas of medical research, in patient temperature measurements in hospital rooms or wards, and in the medical instrument manufacturing industry. At the present time, this thermometry is at a very rudimentary level and it is our objective to assist medical personnel in putting their thermometry on a sound scientific basis. By helping the medical community solve its temperature measurement and control problems and helping it develop expertise in thermometry, better medical care can be achieved.

Clinical laboratories are becoming highly automated in their testing and a large fraction of the results obtained are sufficiently sensitive to temperature to demand that small, stable, highly sensitive, accurate, and rapidly-responding temperature sensors be used. Those sensors should also be capable of producing electrical signals that can be digitized in order to be used with data recording and analysis systems.

In instruments for enzyme rate analysis in which samples of only a few microliters are used, the requirements for accuracy, response time and size of the temperature sensor are paramount, and in instruments for acidity and blood gas analysis, temperature measurements in the immediate vicinity of the measuring electrodes require small, accurate sensors. In routine clinical tests that are presently being performed, temperature errors produce such a variation in the results of enzyme tests that comparisons among laboratories, and even within a given laboratory, are useless. This is a sad state of affairs, especially when one realizes that inadequate thermometry in clinical laboratory tests may result in incorrect diagnosis and improper treatment of the patient.

At the present time, 20% of all temperature measurements of patients in hospitals and in physician's offices are taken using rapid, disposable and inexpensive temperature sensors which are rapidly displacing conventional mercury-in-glass thermometers and, yet, these sensors have not been adequately evaluated!

Just as in the case of the electronic fever thermometers, the temperature sensors in use today throughout the medical field appear to have many of the required qualities, but they have never been adequately characterized. In addition to having sensors with the desired properties, it is necessary to be able to check their calibrations periodically in the field or to enable new calibrations to be made accurately. The best way of doing this would be through the use of a set of standard temperature-reference-points in the range of biological interest and a practical simple mechanism to relate the patient temperature measuring instruments and the various devices utilized in the clinical laboratory to those reference points. None exists at the present time.

Miniature temperature sensors which are insensitive to electromagnetic fields are needed in order to be able to determine the temperature distributions produced in tissues being irradiated by the electromagnetic waves in diathermy (a medical treatment in which heat is produced in the tissues by a high frequency electric current). Temperature mapping in the tissues is required in order to set standards of safe levels of electromagnetic radiation from the diathermy machines. This technique is required since direct measurements of electromagnetic field intensities in tissue are difficult to perform, and, in any case, temperature is the critical variable inside the tissue. Heretofore, such temperature measurements have not been performed in a satisfactory way.

The overall objective of the Medical Thermometry Program is to help solve the problems listed above and, thereby, improve the health care of every patient in the United States. This requires us to provide leadership to the various medical and voluntary standards involving thermometry. Also, we must

1) evaluate the performance of modern temperature sensors as to reproducibility, sensitivity, interchangeability, time response, and other parameters of use,

2) determine temperature distributions in "phantom" materials being irradiated by electromagnetic waves,

3) develop temperature reference points in the range of biological interest and develop test procedures for their clinical laboratory use, and

4) interact actively with appropriate medical technical societies, instrument manufacturers and medical researchers and disseminate to these groups measurement techniques and standards resulting from our work.

The establishment of measurements on a sound physical basis and the establishment of laboratory standards and improved quality-control procedures are essential for valid interpretation and meaningful comparisons of measurement data.

We shall now give briefly our progress during 1974 toward achievement of the program's goals.

2. Progress during 1974

a. Development of Precision Clinical-Laboratory Thermometers.

In order to obtain reproducible results within any clinical laboratory and in order to make a meaningful comparison among laboratories, the temperatures at which tests are performed not only must be carefully controlled but must be known absolutely. In recognition of this situation, the Expert Panel on Enzymes of the International Federation of Clinical Chemistry Committee on Standards has recommended that the temperature accuracy of the reaction must be assured to ±0.05 °C by calibrations against the IPTS-68, that the temperature variation of the reaction mixture should be held within ±0.05 °C of the set point, and that the choice of reaction temperature should be either 25, 30, or 37 °C.

Because of these needs and in order to assist in getting a usable temperature scale into the clinical laboratories, we developed some precision laboratory thermometers which are being sold through the Office of Standard Reference Materials as SRM 933 and SRM 934. These thermometers were designed especially for use in clinical laboratories. See NBS Special Publications 260-48 for their description and use.

b. Evaluation of Small Temperature Sensors

i) Thermistors

Thermistors are ceramic semiconductors composed primarily of manganese and nickel oxides. The relative proportions of these as well as the addition of several other oxides, notably cobalt, copper, or iron, or of Pyrex determines the resistivity of the material. Thermistors are available in a great variety of shapes and sizes. There are beads, discs, rods, and flakes. The beads and flakes are available in sizes ranging from approximately 0.1 mm to several mm in diameter or on an edge and their resistances range from a few ohms to megohms. The discs and rods have the same resistance range but generally have larger physical dimensions than those of the beads and flakes.

Thermistors are small, rugged, sensitive sensors which apparently can be made interchangeable and they are in widespread use throughout the medical field, although their properties as regards reproducibility upon thermal cycling, stability upon ageing at a fixed temperature, interchangeability, and time response have never been adequately evaluated. (Thermistors are also used widely in applications unrelated to health care.) Since they are widely used in medicine in spite of a lack of proper evaluation, we have undertaken a study of properties which are listed above. We are evaluating beads and discs of each of the three basically different chemical compositions which are available. Of each of these compositions, we have selected those resistances which are most widely used in medical applications.

The instruments and other equipment necessary for the stability, reproducibility, interchangeability, and sensitivity tests were constructed and/or assembled, wired, modified as necessary, calibrated when appropriate and put into operation. Included were:

1) three constant temperature baths,

2) two precision resistance bridges,

3) a fume-exhaust system for the baths, especially for those operating at elevated temperatures, and

4) several stepping switches to which the thermistors were connected.

One hundred and thirty-five thermistors, beads and discs, obtained from the six manufacturers who provide almost all of the thermistors used in the United States were wired as four-lead resistors, connected to the stepping switches, and placed in each of two baths. One bath was maintained at 30 °C and the other one at 0 °C. We elected to begin the ageing study at these temperatures because of their immediate usefulness to both the medical community and to oceanographers.

The computer programming necessary for the statistical analysis of the data was performed in close cooperation with Dr. J. J. Filliben of the Statistical Engineering Section.

The stability tests upon ageing at 30 °C were begun at the end of May and those at 0 °C were begun in mid-October. Preliminary analysis of the data obtained on the one hundred and thirty-five thermistors (65 beads, 70 discs) maintained at 30 °C showed, after 119 days of elapsed time in the bath, that in general the beads have smaller drift rates than do the discs. Bead resistances were decreasing and disc resistances were increasing, with few exceptions. The lower resistance beads (2 kΩ and 10 kΩ) exhibited less change than those of the highest resistance (30 kΩ). There appeared to be little correlation between disc resistances (1 kΩ, 2 kΩ, 5 kΩ, and 10 kΩ) and drift rates. There were exceptions but, generally, thermistors of a given resistance value from the same manufacturer behaved in a similar manner. This may indicate that those thermistors were prepared from the same batch and/or during the same production run.

Representatives of three thermistor manufacturers visited us during the year and we visited two others for the purpose of discussing our evaluation program.

ii) Platinum RTD's

Platinum resistance-temperature-detectors (RTD's) are industrial platinum thermometers which usually are much smaller, far more rugged, and cost considerably less than the standard platinum resistance thermometers (SPRT). The platinum resistance element of the RTD is supposedly mounted in a strain-free manner, for example, on a grooved ceramic post, and encased in either a stainless steel, glass, or ceramic sheath with ceramic insulation. Such methods of mounting may cause contamination problems at elevated temperatures and cause changes in their calibration. The different techniques used by the various manufacturers for obtaining "strain-free" mounts are usually proprietary and thus not available to the customer. The RTD's are available in sizes ranging from about 1 mm on an edge to several centimeters in length and several millimeters (up to about 1 cm) in diameter and their R_0 values, i.e., their resistances at 0 °C, cover the range from about 10 Ω to 7000 Ω. The Pt RTD's are widely used not only in temperature measurements but also in temperature control systems. Oceanographers use Pt RTD's for "accurate" measurements of ocean temperatures and many instrument manufacturers use them in their temperature control systems.

Other than the limited tests by T. J. Quinn of England's National Physical Laboratory and by K. R. Carr of the Oak Ridge National Laboratory, there has not been any independent evaluation of the industrial Pt RTD's. To fill the need of providing an evaluation of the RTD's with regard to

1) stability upon thermal cycling (to determine the importance of cycling as a cause of calibration drift),

2) self-heating,

3) insulation resistance,

4) time-response, and

5) stability upon ageing at a fixed temperature,

we have undertaken a study of 125 Pt RTD's obtained from six manufacturers. Most of the RTD's, however, were obtained from only five of the six manufacturers.

All of the bridges, furnaces, constant temperature baths, and ancillary equipment required for this study were assembled, appropriate alterations made, tested for proper operation, calibrated, and put into operation. The manufacturers of the RTD's completed delivery of all 125 sensors at the end of the summer and leads were connected to 50 of them in order to begin thermal cycling tests. Although most of the RTD's are two-lead devices (a few have three leads), we converted them into "four-lead" sensors by connecting the four leads as closely as possible to the points at which the two leads from the sensor leave the sensor sheath. The RTD's which we chose to study are those of small physical dimensions and having R_0 values ranging between 50 Ω and 2000 Ω. These are the types most suited for medical and/or oceanographic applications. I am including oceanography here since the temperature range of interest to oceanographers is included in that of interest to the medical community.

At the end of the year, the study of the stability upon thermal cycling between 250 °C and room temperature was started for 50 Pt RTD's. Prior to heating the RTD's to 250 °C, they had been measured between 0 and 100 °C over a period of time to test for stability before the thermal cycling began. Although the study has only just begun and no results can be given, we can say that thermal cycling, in which the RTD's were held at 250 °C for 6 hours, has a fairly large effect on some of the sensors. At least one sensor had a change in R_0 by an amount equivalent to 0.3 K.

3. Scanning Radiometer

Human thermal comfort has always been a consideration in the design of buildings, whether homes or businesses. In the "good old days" when energy was plentiful and inexpensive and buildings did not need to be so carefully designed, the thermostat in the room or building could be set such that one would be comfortable at any given location in the room. When his location was changed to a different part of the building or even a different part of the same room, however, it is very likely that a change in the thermostat setting would be necessary in order for the person to be made comfortable again. Since energy required to keep one comfortable is now very expensive and becoming more and more so with the likelihood of an energy shortage, well insulated buildings designed with human thermal comfort maintained by a minimum use of energy are a prime consideration.

A number of different types of thermometers have been used by design engineers and probably the one most widely used at the present time is the globe thermometer. This is a very crude thermometer system which attempts to measure the mean radiant energy over a solid angle of 4π steradians. Such a thermometer is not a good indicator of human thermal comfort. This can easily be seen by a simple example. The human body is, to a first approximation, a 2-dimensional system and if a person is near a fire, then one side (say his front) is exceedingly hot whereas the other side (his back) may be excessively cold. As a result of this exchange of radiant energy, the person is most uncomfortable but since the globe thermometer measures the mean radiant energy over 4π steradians, it would indicate a temperature which would be pleasingly comfortable to the person. This, then, points up the problem with such a thermometer.

In order to help solve this problem of human thermal comfort, we have been collaborating with Dr. T. H. Benzinger and Dr. J. E. Hill of the Center for Building Technology on a project to develop a Scanning Radiometer which will permit valid measurements of the radiant energy as would be experienced by a person.

A prototype of a scanning radiometer was constructed and tested for the best reflecting and absorbing materials to be used on it, its sensitivity, and the best methods to be used in its calibration. The final product consisted of twenty semiconductor-type thermopiles, ten with black anodized aluminum foil and ten with thin, highly-polished gold foil, arranged in a checker-board fashion in two columns. The foils were firmly attached to one of the exposed surfaces such that the foil was in good thermal contact with but electrically insulated from the thermopiles. This arrangement eliminated effects of convection and conduction, thereby giving an output due to radiation alone. The back sides of the thermopiles were electrically insulated from but in good thermal contact with an aluminum block on which they were mounted and which provides the reference temperature. An industrial platinum resistance thermometer was mounted in the center of the aluminum block and was connected to a linear bridge having an output of 1 mV/°C. The reflecting and absorbing thermopiles were connected in series opposition and shunted to give an output of 1 mV/°C. The radiometer thus developed measures the mean radiant energy either impinging upon it from or radiating from it to a solid angle of 2 π steradians and it is so mounted that it can be rotated through an angle of 2 π radians.

A paper describing the scanning radiometer, its construction, test performance, and its calibration is in the process of being written and may be consulted for further details.

4. Measurement Assurance Programs

We were engaged in a measurement assurance program with one high quality scientific laboratory, Woods Hole Oceanographic Institution (W.H.O.I.), and with one small but technically very advanced thermistor manufacturer. This involved several different types of measurements but primarily it involved the calibration of 40 thermistors over the temperature range 0 to 30 °C with measurements every 5 °C. Of the 40 thermistors calibrated, 19 were calibrated by W.H.O.I., the manufacturer, and NBS. The remaining 21 were calibrated by the manufacturer and NBS.

As a result of these measurements, W.H.O.I. discovered an error in their measurement technique which manifested itself as an error in the temperature of from 30 to 50 mK. This is a significant error, especially so since they want to measure temperatures to the nearest mK. Their ocean temperature measurements are compared with those obtained by oceanographers of other countries so measurements must be made relative to a recognized scale, IPTS-68. Plans were started for an international comparison of thermometers, probably thermistors, with W.H.O.I., NBS and selected oceanographers of other countries participating so that, eventually, all oceanographic groups will be making accurate measurements on the same temperature scale.

The program with the manufacturer progressed as indicated above and is continuing. Several other measurements need to be made by them and by us before the program with them is completed. It appears, however, that they make accurate temperature measurements, obtaining values which are about 3 mK different from those obtained by us.

5. Temperature Measurements in the Presence of an Electromagnetic Field

The purpose of this work is to measure the temperature distributions produced in tissues or "phantom" materials while being irradiated by electromagnetic radiation at frequencies of either 13 MHz, 27 MHz or 2450 MHz. Such measurements are necessary in order to set standards of safe levels of radiation. The present ANSI standard which specifies, quite arbitrarily, a maximum exposure level 10 mW/cm^2 is generally accepted by most western countries, including the United States, but this level is 1000 greater than the U.S.S.R. standard for industrial workers. No one really knows what levels are safe, so temperature measurements are essential.

The problem in making temperature measurements in the presence of an electromagnetic field is that the usual thermometers are metallic and, consequently, are heated by induced currents. We had to either select some thermometers already in existence in which inductive heating was small or to develop new thermometers which are non-metallic.

A considerable amount of time was devoted to developing a system in which light from pulsed LED's was transmitted through glass or plastic fibers to a temperature sensor which was either a liquid crystal which then reflected part of the light through other fibers (in the same bundle of fibers) to a photodiode, or was an optically active material, lithium tantalate ($LiTaO_3$), sandwiched between a polarizer and an analyzer. In the case of the liquid crystals, the intensity of the reflected light rather than its frequency was measured, since the incident light from the LED was essentially monochromatic and not white light. A number of problems relating to stability of the electronics and to maintaining a constant intensity of light emitted by the LED were overcome. The latter problem was solved by

1) providing a constant-current source for the LED,

2) rigidly mounting the LED and the optical fibers in a BNC connector so that electrical connections to the LED are made rather than making connections along the light path, and

3) by maintaining the LED at a constant temperature.

With the stable light source and detection system developed, a number of different liquid crystal mixtures, both microencapsulated and non-encapsulated, were tested for feasibility for use as an accurate thermometer. Liquid crystals from several suppliers were tested. Our results showed that with the liquid crystals available to us, their stability was such as to severely limit their use as thermometers, even at a 0.1 to 0.2 °C level of uncertainty. We want to make measurements to a ±0.05 °C uncertainty but for some cases we would accept an uncertainty of ±0.1 °C.

With the conclusion that liquid crystals are not sufficiently stable to serve as accurate thermometers, it came to our attention that $LiTaO_3$, an optically active material whose optical rotatory power is apparently a sensitive function of temperature, might make a suitable thermometer. This material is placed between a polarizer and an analyzer and reflector. The light from the LED is conducted by the optical fibers to the polarizer where it is polarized, transmitted and rotated by the $LiTaO_3$, analyzed, reflected back through the analyzer, the $LiTaO_3$, the polarizer and through the fibers to the photodiode detector. The intensity of light arriving at the photodiode will then be a function of temperature. Preliminary tests indicate this to be a suitable thermometer. A patent disclosure is being made and a description of the instrument is being written up for presentation at the Union Radio Scientifique Internationale meeting in June, 1975.

In addition to work on the crystal optics thermometer, a special thick film thermistor-integrated circuit thermometer (manufactured commercially) was tested for feasibility. In this device, the thermistor flake is connected by high resistance (100 kΩ) leads, 5 μm thick and separated by 5 μm, to an integrated circuit Wheatstone bridge which has four high resistance (100 kΩ) graphite impregnated Teflon leads. Two of these are current leads and the other two are potential leads which are to be connected to the detection system. The thermistor, thin-film leads, etc. are mounted on sapphire and covered by a protective coating. These thermometers can detect changes of less than 0.1 °C while in an electromagnetic field and tests in the presence of a field indicate negligible heating. The heating that occurs appears to be in the graphite-impregnated Teflon leads. Several of these thermometers have been ordered but only one had been delivered by the end of the year.

In conjunction with the thermometers described above, an infrared (IR) camera will also be used in making temperature measurements in "phantoms". In order to properly calibrate it, a blackbody source whose temperature can be controlled to better than 0.01 °C has been constructed. The calibration will be performed with and without the plastic covering in which the "phantom" material is placed in order to prevent its deterioration.

The IR camera, the special thermistor bridge, and the crystal optics thermometer are ready for calibration and as soon as this is completed, measurements will be made of the temperature distributions developed in the "phantoms" while being irradiated by the electromagnetic waves.

6. Cooperative Efforts with Selected Hospitals to Check Their Thermometry and to Test a Simple and Inexpensive Temperature Measurement System

During the year we started a program with the Clinical Chemistry Section of the Clinical Center of NIH, with the Pathology Department of York Hospital of York, Pennsylvania and with the Sacred Heart General Hospital of Eugene, Oregon, in which we have provided a simple and inexpensive but accurate temperature measurement system for them to use to test the temperature measurement and control systems of the various instruments in their laboratories. By this means we shall have access to data on most of the instruments commercially available to the clinical laboratories. This should provide a good guide (and useful data) for manufacturers as to what is desired by the clinical chemists and the pathologists and what is required of new instruments.

The measurement system which we provided consists of a constant current source, a calibrated thermistor, and a standard resistor. The participating laboratory provides a digital voltmeter by which they measure the voltage drop across the thermistor and hence, its resistance. Then, by finding that resistance in the resistance-temperature table which we provide with the thermistor, the temperature can be determined. The uncertainty in the calibration of the thermistor is ±0.01 °C.

We plan to extend this program to several more hospitals.

7. Voluntary Standards Work

We have participated in voluntary standards activity through the ASTM Subcommittee on Medical Thermometry, E20.08 (a new Subcommittee which has been in existence for about one year), and through the National Committee for Clinical Laboratory Standards.

The ASTM Subcommittee has been engaged in writing standards for electronic fever thermometers, for continuously monitoring systems, and for precision thermometers used in the clinical laboratory. Several working sessions were attended.

One meeting of the NCCLS was attended and I prepared for their consideration a Proposed Standard for Temperature Calibration of Water Baths, Instruments and Temperature Sensors. That was submitted to the full committee by the chairman of the Area Committee on Instrumentation and is now in the process of being evaluated.

8. Calibrations

Several thermistors were calibrated for people both at NBS and outside NBS. This was in addition to those provided under 4 and 6 above.

9. Visits

Several hospitals, instrument manufacturers, scientific laboratories, and scientific meetings were visited or attended during the year. Several manufacturers, pathologists, clinical chemists, and medical technologists visited us at NBS this year.

10. Relevant Documents

a. B. W. Mangum, Clin. Chem. 20, 670 (1974).

b. NBS Special Publication 260-48, May 1974.

c. "Proposed Standard for Temperature Calibration of Water Baths, Instruments and Sensors".

d. "Design, Construction and Calibration of a Scanning Radiometer", to be published.

11. Personnel Associated with the Medical Thermometry Program

B. W. Mangum, Project Leader
T. C. Cetas
S. D. Wood
J. L. Sligh
S. B. Tillett

V. Calorimetric Measurement of Temperature - M. L. Reilly

1. Introduction

The primary goal of this research is to determine thermodynamic temperatures above 0 °C using an experimental method based upon the Stefan-Boltzmann law, which states that the total power radiated by a blackbody is directly proportional to the fourth power of its thermodynamic temperature. The apparatus constructed for this measurement consists of three main components:

1) a radiator which closely approximates a blackbody source;

2) a heat flow microcalorimeter in which radiant power is absorbed and measured; and

3) an aperture cavity, lying between the radiator and calorimeter, through which passes that fraction of a radiant power which is emitted by the radiator and enters the calorimeter.

In principle, from the ratio of two powers measured with the calorimeter one can directly obtain the ratio of the corresponding thermodynamic temperatures of the radiator. By selecting the triple point of water (defined to be 273.16 K) as one of the radiator temperatures, the other temperature is uniquely determined on the thermodynamic scale limited only by the accuracy of the measured power ratio.

At present a careful investigation of all known factors that might influence the accuracy of the measurements is in progress. The approach has been to study the effect that various controlled changes in the apparatus have upon the measured steam-point/triple-point (SP/TP) power ratio. The steam point was chosen because it is the only other thermometric fixed point with a temperature assignment of sufficient accuracy for these tests. Based upon recent results of gas thermometry work at NBS, the fourth power of the ratio of the SP/TP temperatures expressed on the thermodynamic scale is believed to have an absolute error of less than .01% which is the accuracy desired of this experiment.

The first phase in the search for errors was testing those factors which influence the radiator and its immediate surroundings, the guard shield. The measured SP/TP power ratio was found to be lower than the expected value calculated from the fourth power of the ratio of the corresponding thermodynamic temperatures, and non-blackness of the radiator cavity appeared to be an attractive explanation for this observation. Because the vacuum manifold surrounding the radiator and guard shield remained near room temperature at all times, considerable radiation characteristic of room temperature entered the radiator cavity. A non-black radiator would reflect some small fraction of this power causing an apparently lower steam-point power, an apparently higher triple-point power and a corresponding low SP/TP power ratio.

Several cavity geometries and coating materials were studied together with changes in the nature and temperature of the surfaces immediately enclosing the radiator. Although some of the modifications produced slight changes in the SP/TP power ratio, the total spread of all observations remained within ±.02%. The conclusion drawn from this series of tests was that any error due to non-blackness of the radiator was an order of magnitude smaller than the difference between the measured and expected power ratio.

The second phase of the error investigation was the study of the aperture cavity. The most recent work on this problem will be discussed below in greater detail.

2. Recent Progress

The purpose of the aperture cavity is to transmit to the calorimeter an invariant fraction of the power emitted by the radiator and to absorb radiation from all other sources. The original cavity was in the form of a right circular cylindrical tube 62 mm long and 14 mm in diameter. It was closed at each end by a disc containing a circular aperture which was concentric with the tube axis. The entrance aperture was about 2 mm in diameter and the exit aperture was either about 5.7 or 8 mm in diameter. There were five internal annular baffles each with a circular opening of about 10 mm in diameter and also concentric with the tube axis. The entire interior surface area was coated with electrolytically deposited platinum black which is known to have a very high absorptivity. The fraction transmitted by this cavity was only a few percent of the radiator power entering the cavity. The remaining amount had to be totally absorbed within the cavity.

The cavity had been designed by ray-tracing techniques because no formal method was available for treating the radiant exchange within a cylindrical cavity containing annular baffles. In order to examine its performance more critically, it was necessary to develop a computer based model which described the propagation of radiation through the cavity. The first major point determined with the aid of the model was that, even with optimistic estimates for the reflectivities of the surfaces involved, an excess power of several tenths of a percent would be transmitted to the calorimeter by multiple internal reflections within the aperture cavity. This power excess was greater for the triple-point radiation than for the steam-point radiation and therefore the measured SP/TP power ratio must be lower than the expected value.

Diffraction had been considered to be the principal source of potential error associated with the aperture cavity. The power lost through diffraction should be greater for the triple-point radiation than for the steam-point radiation and therefore the measured SP/TP power ratio should be higher than the expected value; a "worst case" calculation indicated that the ratio would be increased by about one percent. The fact that the measured ratio was low seemed to indicate that diffraction was not the major source of error in the measurements. In addition, measurements made using the two exit apertures of different diameter had shown agreement within .02% for the SP/TP power ratios; this gave apparently strong experimental evidence that diffraction errors were small. (Model calculations, however, showed that this agreement between the two measured ratios was inconsistent with the observed low values obtained for the ST/TP power ratios.)

The second major point identified with the aid of the model was the critical test parameter for the study of the aperture cavity performance. Immediately surrounding the aperture cavity is a radiation shield which is cooled by liquid nitrogen. A circular opening in this shield (termed the 77 K aperture) restricted the field of view at the entrance aperture of the aperture cavity to about the remote two-thirds of the interior of the radiator cavity. A black coating, applied to that region of the cool radiation shield which was visible at the entrance aperture, gave those surfaces a high absorptivity. As predicted by the model and confirmed by experiment, it was not possible to detect any changes in the level of radiant power originating at those surfaces. As a consequence, the amount of power entering the entrance aperture of the cavity depended almost entirely upon the area of the 77 K aperture; in effect, the 77 K aperture became a virtual two-dimensional source emitting power characteristic of the radiator. From power measurements at the steam point and triple point employing at least three different 77 K aperture areas it would be possible to estimate the error due to the excess power provided, of course, that the observations agreed with the model predictions.

The results of this series of experiments can be summarized as follows. When the 77 K aperture area was increased to 69 and then to 96 mm^2, the power measured with the radiator at 0 °C increased by .29 and .48% over that measured with the original 77 K aperture area of 50 mm^2. The precision of the 0 °C power measurements was ±.02%. With the radiator at 100 °C, the corresponding increases in the measured power were .22 and .32%, with a measurement precision of ±.01%. The ST/TP power ratios associated with the 77 K aperture areas of 50, 69 and 96 mm^2 were 3.470, 3.468 and 3.464, respectively. The expected power ratio is 3.4817 ±.0001. These results unambiguously identified the original aperture cavity as a major source of experimental error.

It was not possible to simultaneously obtain from the model calculations an acceptable representation of both the power ratios and the power increases observed for each radiator temperature. By selecting an appropriate value of the single adjustable model parameter, it was possible to reproduce the increased powers within about ±.05% but the corresponding ST/TP power ratios were approximately 4.0% lower than the observed quantities. Alternatively, it was possible to reproduce the measured power ratios within about .1% but the associated power increases at each radiator temperature were more than an order of magnitude less than the observed quantities. Clearly, the model, although extremely useful in identifying the critical measurement parameter, failed to adequately describe the experimental observations.

The most obvious explanation for the discrepancies between the model and the experimental observations was error due to diffraction at the entrance aperture of the aperture cavity. Allowance for this contribution was not included in the model because the mathematical formalism necessary to treat a diffracting aperture irradiated by a partially coherent, extended source close to the aperture does not yet exist. Also, as discussed earlier, at the time the model was being developed, there was experimental evidence suggesting that any error contribution due to diffraction was perhaps an order of magnitude less than the apparent error in the measured SP/TP power ratio.

Steel, De and Bell recently published an expression for the diffraction loss at a circular aperture irradiated by a coherent source of finite size. Although the conditions of their derivation are not completely consistent with this experiment, when estimated diffraction losses calculated by their approach were combined with the excess power calculated with the model, the experimental observations were represented within the uncertainty of the corrections themselves. The calculated increases in the measured SP/TP power ratios due to diffraction were +.28, +.23 and +.18% for 77 K aperture areas of 50, 69 and 96 mm^2, respectively. Applying these corrections it was then possible to reproduce the resultant power ratios with the model within about ±0.03%, the experimental uncertainty of the ratios themselves. The percentage increase in the observed powers at each radiator temperature could be similarly reproduced within ±.03%.

With the reservation that the diffraction corrections applied may not be valid, this interpretation of the experimental observations indicates that both diffraction at the entrance aperture of the cavity and multiple reflections within the cavity contribute large but partially offsetting errors to the observed measurements. Specifically, for a 77 K aperture area of 69 mm^2 and a radiator temperature of 0 °C, the diffraction loss was estimated to be about .8% of the measured power while the reflection excess was estimated to be about .9%. For the same 77 K aperture area but a radiator temperature of 100 °C, the estimated diffraction loss was about .6% and the reflection gain was about .3%. The overall uncertainty of these estimates is probably not less than .5%. Because of the apparent magnitude of these error estimates no attempt was made to take additional data with this cavity. Without a means to independently verify the estimated reflectivity of the aperture cavity, it was not possible to obtain from these data a useful estimate for the thermodynamic temperature of the steam point.

Based upon knowledge gained about the performance of the original aperture cavity, a new cavity has been designed and built. Interior annular baffles, a major source of trouble in the original design, have been eliminated and the interior surface area has been increased by several orders of magnitude. It was necessary to increase the overall separation between the radiator and calorimeter in order to accommodate the new aperture cavity which is substantially larger than the original. However, the original aperture geometry was retained due to limitations imposed by the original design of the radiator and calorimeter. The revised apparatus will allow selection of any of three 77 K apertures as well as either of two entrance apertures of the aperture cavity during the course of a single experiment. It is hoped that the error due to excess power transmitted by multiple internal reflections will be reduced below .01% so that the error due to diffraction can be studied independently.

3. Relevant Documents

a. Ginnings, D. C. and Reilly, M. L., *Temperature, Its Measurement and Control in Science and Industry* (Instrument Society of America, Pittsburgh, 1972), Vol. 4, Part 1, p. 339.

b. M. L. Reilly "Angle Factors for Radiant Exchange Within a Cylindrical Enclosure Containing Annular Baffles" to be published in International Journal of Heat and Mass Transfer.

c. Steel, W. H., De, M., and Bell, J. A., J. Opt. Sec. Am. 62, 1099, Sept. 1972.

4. Personnel Associated with the Project

M. L. Reilly.

VI. High Temperature Gas Thermometry - L. A. Guildner

1. Introduction

At the NBS an investigation is in progress to make more accurate determinations of the present differences between the International Practical Temperature Scale of 1968 and the Kelvin thermodynamic scale. Values of thermodynamic temperatures, determined by means of a constant-volume gas thermometer, are compared with values of the same temperatures on the international scale.

As originally adopted in 1927, the International Temperature Scale was intended to correspond as closely as possible to the thermodynamic temperature scale. The values of the ice and steam points were defined as fundamental fixed points. The values assigned to the oxygen, sulfur, silver, and gold points had been determined by gas thermometry measurements. Up to the gold point, gas thermometry had been then, and continues to be, the most convenient and accurate method for determining the values of temperature on the thermodynamic scale.

Early in this century, the techniques for gas thermometry reached a state of refinement which is not readily surpassed. For the scale of 1927 it was possible to choose values of the fixed points from determinations which vary from one another scarcely more than the variation between results reported more recently.

Within the NBS program, intensive effort is being made to reduce the random and systematic errors of measurement. Briefly, the gas thermometry is organized in the following way: the constant volume portion of the thermometer is separated by a differential pressure indicator from a mercury manometer. Thus the manometer can have large menisci while the dead space of the gas thermometer is kept small and constant. Thermostats have been made that have an even and constant temperature distribution around the thermometer bulb. Special equipment has been made to measure characteristics of the thermometer system, such as the temperature coefficient of volume of the bulb, and the thermomolecular pressure difference in a small bore tube passing through a large temperature gradient.

Measurements which have been reported in published documents have demonstrated the value of minimizing sorption effects in gas thermometry. Beginning at the triple point of water as a base temperature, the thermodynamic steam-point temperature (assigned the value of 100.00 °C on the IPTS-68) has been found to be 99.970 °C ±.0035 °C, and the apparent steam-point temperature has been shown to be quite dependent upon the state of cleanliness of the gas thermometer. The significance of this result can be appreciated most readily in the context of the IPTS-68 itself; in Table 7 of the text, the estimated thermodynamic uncertainty of the 100.00 °C steam-point temperature is given as ±.005 °C. The NBS result indicates an error roughly six times as large as was thought possible!

2. Present Results

During the past year, the gas thermometer has been operated at temperatures as high as 460 °C. Preliminary values of the tin and zinc freezing points were communicated to the CCT during its meeting in May. A summary of these data, along with the IPTS-68 values, is given below:

Fixed point	NBS determination		IPTS-68 value	
triple point of water	given		defined	0.01 °C
steam point	99.970	±.0035 °C	100.00	± .005 °C
tin freezing point	231.92	±.015 °C	231.9681	± .015 °C
zinc freezing point	419.51	±.02 °C	419.58	± .03 °C

It may be worth noting that the CCT, during its deliberations on a new text to accompany the IPTS-68, decided to remove the table of uncertainties.

Further details of this project can be found in publication 3a.

3. Relevant Documents

a. "Differences of Thermodynamic Temperatures and International Practical Temperatures Between 409 K and 730 K", L. A. Guildner and R. E. Edsinger, to be published.

b. "The Thermodynamic Kelvin Temperature Scale from 273.15 K to 415 K", L. A. Guildner and R. E. Edsinger, J. Res. NBS 77A, 383 (1973).

c. "Effects of Sorption on the Realization of the Thermodynamic Scale", L. A. Guildner, Richard L. Anderson, and R. E. Edsinger, *Temperature, Its Measurement and Control in Science and Industry*, Vol. 4, Part 1, p. 313, (Instrument Society of America, Pittsburgh, 1972).

4. Personnel Associated with this Project

L. A. Guildner, Project Leader
R. E. Edsinger

VII. Acoustical Thermometry - H. H. Plumb

1. Introduction

Recent publications present in some detail the rationale and history of the low-temperature acoustic thermometry project (see Refs. 3b and 3c). Basically, the experiment employs the principle of measuring the speed of sound in an ideal gas; for a monatomic, ideal gas the relation

$$W^2 = 5/3 \frac{RT}{M} \text{ holds.}$$

The NBS Provisional 2-20 K Scale is based on results obtained in this project for helium gas.

In order to help eliminate possible systematic errors in these temperature measurements, the original apparatus has been modified by replacing the solid quartz rod which was used to measure the displacement of the reflecting piston. Now this purpose is served by a laser interferometer (see Ref. 3b).

2. Present Activities

The modified acoustic thermometer has been employed in the past year to obtain thermodynamic values of the superconductive transition temperatures of the lead and indium samples supplied with the NBS superconductive thermometric fixed-point device, SRM 767. This work will be reported in the literature during the coming year, so that only a summary will be given here:

Sample	Transition temperature
Lead	7.1990 ±.0007 K
Indium	3.4142 ±.0008 K

In other experiments, isotherms which had been studied previously are being reexamined to determine the effect of the equipment modifications.

An apparatus which previously had been used in the calibration of doped germanium resistance thermometers is now serving a new function. The stability of germanium thermometers which have been in use in laboratories in various parts of the world has come into question, and sample thermometers are under study in the calibration apparatus. In addition, the apparatus is being used to study the stability and reproducibility of iron-doped rhodium resistance thermometers developed at the National Physical Laboratory in England. Both of these experiments are part of the goal of developing practical thermometers for the temperature region below 30 K.

3. Relevant Documents

a. "Fixed Points: Superconducting Transition Temperatures of Lead and Indium", G. Cataland and H. H. Plumb, submitted to Metrologia.

b. "Appendix 1 - NBS Acoustical Thermometry", H. H. Plumb and G. Cataland, NBS Technical Note 830, May 1974.

c. "Low Temperature Thermometry: Interim Report", G. Cataland and H. H. Plumb, NBS Technical Note 765, May 1973.

4. Personnel Associated with this Project

H. H. Plumb, Project Leader
G. Cataland

VIII. Platinum Resistance Thermometry - G. T. Furukawa

1. Introduction

From 13.81 K to 903.89 K the International Practical Temperature Scale of 1968 (IPTS-68) is based on the standard platinum resistance thermometer (SPRT), nine defining fixed points, and specified equations for interpolating between the fixed points. The functions of the NBS Platinum Resistance Thermometry Laboratory include the maintenance of the IPTS-68; realization of the IPTS-68 fixed points; calibration of SPRT's for the government, university, and industrial laboratories; research to improve the IPTS-68; and liaison with national and international individuals and groups to advance accurate thermometry. In conjunction with these efforts, new thermometric equipment and instrumentation are developed as needed and automation of the measurement process, data logging, and computation is introduced wherever feasible. The staff participates in seminars and conferences in thermometry and related fields.

2. Freezing Point of Aluminum as a Fixed Point

The SPRT scale above the zinc point (419.58 °C) is obtained by extrapolation. Any error in the calibration at the tin point (231.9681 °C) or at the zinc point is amplified at the higher temperatures; at the upper temperature limit of the SPRT scale (630.74 °C) the error is amplified by about 3.4 times. A fixed point at the upper temperature limit would help reduce the uncertainty of the SPRT scale at the higher temperatures. To help serve this need, the reproducibility of the freezing point of six aluminum samples was investigated. For five of the samples the range of the freezing points in terms of R(Aℓ)/R(TP), the ratio of the resistance of the SPRT at the aluminum point to that at the TP of water, corresponded to 0.51 mK. The R(Aℓ)/R(TP) of the sixth sample deviated by an amount corresponding to -1.31 mK from the average of the other five; the sixth sample may have been contaminated during the preparation of the freezing-point cell, or perhaps the original sample bar was not homogeneous. The results show that the freezing point of aluminum (near 660 °C) is at least as reproducible as the freezing point of antimony (near 631 °C). Since aluminum metal is easier to obtain at high purity than is antimony and since the freezing point of aluminum is more convenient to realize than the freezing point of antimony, the aluminum point may well be a useful fixed point for the IPTS.

To test the performance of SPRT's at the aluminum point, six SPRT's were "calibrated" at the TP of water and the freezing points of tin, zinc, and aluminum. For each SPRT the quadratic relation

$$W(t') = R(t')/R(0\ ^\circ C) = 1 + At' + Bt'^2, \qquad (1)$$

based on the measurements at the TP of water and the freezing points of tin and zinc, was extrapolated to the aluminum point to obtain t'(Aℓ). The mean t'(Aℓ) of five SPRT's was 660.4021 °C (range = 3.3 mK, standard deviation = ±1.3 mK); the t'(Aℓ) of the sixth SPRT deviated +9.1 mK from the mean of the five SPRT's. The depression of the freezing point of the aluminum sample was estimated for the known impurities; the adjusted freezing point for pure aluminum was computed to be 660.407 °C. The estimated uncertainty of the value is ±5 mK, half of the uncertainty originating from the estimate for the depression of the freezing point by the impurities. The value is 0.010 K lower than the value reported by McAllan and Ammar in 1972; however, in the same paper McAllan and Ammar reported the freezing point of antimony which is 4 mK higher than the value reported by McLaren and Murdock in 1968. Considering the errors of measurement and of the extrapolation, the values are closely consistent.

Results show that the aluminum point could be employed advantageously as a fixed point at the upper limit of the present SPRT range. The suggested formulation is:

$$W(T^*) = 1 + At^* + Bt^{*2} + Dt^{*2}(t^* - 231.9292)(t^* - 419.58). \qquad (2)$$

For calibrations employing the TP of water and the freezing points of tin, zinc, and aluminum, the coefficients A and B of eq. (2) are the same as those of eq. (1). When a suitably assigned value of t*(Aℓ), based on quadratic extrapolation of selected SPRT's, is used the coefficient D should be small (about 10^{-16} or smaller). If D is large, an error in calibration should be expected. In eq. (2) any error in the aluminum-point calibration affects the calculated values of temperatures below the zinc point by at most only 2 percent of the error.

It is expected that more precise results can be obtained by employing SPRT's of higher resistance than those (0.25 ohm at 0 °C) that were used in the present investigation. Procurement and construction of SPRT's of higher resistance are planned. Also, aluminum samples of higher purity are expected to become available in the near future.

3. Freezing Point of Zinc

As part of the work to investigate the reproducibility of the zinc point, the freezing curves of zinc-point cells prepared from three batches of samples were studied. Two of the batches were NBS SRM 43h and 740; the other batch was from a commercial source. The SRM 74 zinc is nominally purer than 99.9999 percent, while SRM 43h is of lower purity. The preliminary freezing curves obtained with SRM 740 show that the temperature changes by less than 0.1 mK during the first 60 percent of the freeze. The freezing curve of the cell containing zinc from a commercial source changes only slightly more than that of SRM 740. The freezing curve of SRM 43h changes significantly more rapidly than that of SRM 740.

Cells made from SRM 740 zinc are used in the calibration of SPRT's. Because of occasional breakage (more than with tin) replacements are necessary. Additional zinc-point cells of SRM 740 must be assembled before further experiments on the reproducibility of zinc points can be continued. Additional samples were ordered, but the specimens that were received contain cracks, a possible source of contamination. The replacement of the samples is at present being negotiated.

4. Freezing Point of Mercury

The freezing point of mercury is a useful secondary fixed point (near -39 °C). Mercury can readily be obtained at high purity (less than 1 ppm of impurity). Six freezing-point cells of borosilicate glass were assembled from three sources of mercury. (Spectrochemical measurements cannot distinguish them. One of the samples is claimed to have total impurity of 20 parts in 10^9.) Two cells of stainless steel will also be assembled in an attempt to develop a more durable device and the freezing point compared with those contained in borosilicate glass. The effect of contamination, if any, by the stainless steel will be tested by accelerating the reaction by heating the cell to about 100 °C for several weeks. The freezing process will be controlled by adjusting the thermal insulation around the mercury cell immersed in a Dry Ice - alcohol mixture. The measurements are expected to begin around May 1975.

5. Triple Point of Water

The triple point of water is the principal reference point in thermometry. The equations of the SPRT scale are expressed in terms of the resistance ratio R(t)/R(0 °C), where R(0 °C) is obtained from the measurements at the TP of water. The investigation of the reproducibility of the TP of water is planned. A large insulated container for 8 or 9 cells was assembled. The comparison work is expected to be done during March and April 1975.

6. SPRT Comparator Apparatus, 90 K to 900 K

A comparator is being built to investigate the reproducibility of the IPTS-68 between 90 K and 631 °C by intercomparing long-stem type SPRT's. The assembly of the furnace core to contain the tubes for accepting the long-stem type SPRT's has been temporarily halted because of accidental damage to the vacuum induction furnace in the NBS Shops. Meanwhile the assembly of associated components such as shields and shield heaters has been in progress. The assembly of the furnace core was resumed in February 1975.

7. Effect of Pressure on Deep Sea Thermometers

The effect of pressure up to 10,000 psi on deep sea platinum resistance thermometers is being investigated. Except for the installation of the wiring system to the thermometers, the pressure apparatus for testing the thermometers has been assembled. Preliminary test measurements are expected to start around the latter part of February 1975.

8. SPRT Calibration (See also Section III)

During the calendar year 1974 70 customers' SPRT's were calibrated. The calibration data taken during the two-year period between July 1972 to July 1974 were analyzed along with the control measurements that are taken with the calibrations. The results of the analysis will be presented in a publication to demonstrate the maintenance of the IPTS-68 from 90 K to 631 °C at the NBS. The analysis shows that over 98% of the fixed-point calibrations were reproduced within ±0.5 mK, with a maximum deviation of 0.7 mK.

9. National Measurement Assurance Program on SPRT's

As part of the Measurement Assurance Program (MAP) on the calibration of SPRT's a group of three long-stem type SPRT's is being shipped to participating laboratories for comparison of calibrations. When a laboratory completes the calibration, the SPRT's are shipped back to NBS for the check calibrations; then the SPRT's are shipped to the next laboratory. During the calendar year three laboratories (Army Metrology and Calibration Center, Air Force Aerospace Guidance and Metrology Center, and Army White Sands Missile Range) were involved in the program. All of the laboratories received immediate benefit from the intercomparison results, e.g., the Army WSMR calibration using the ac bridge technique was found to be in good agreement with the NBS dc calibration. (Earlier, NBS provided WSMR a SPRT calibration facility, including the ac bridge). The MAP is continuing with the collaboration of both industrial and governmental laboratories.

As a part of the program, certain measurements are obtained by the participating laboratories and by NBS to test the effect of shipping the SPRT's. (After the calibration of SPRT's at the NBS a large number of the SPRT's are shipped back to the customers by common carrier.) The results show that the SPRT's are not affected significantly during shipment. (The cases containing the SPRT's are cushioned in soft polyurethane foam in a large wooden box.)

10. International Comparison of Temperature Scales

As a part of the international program for the comparison of the temperature scales from 13.81 K to 373.15 K a set of three capsule-type SPRT's were calibrated three times. Two of the calibrations on three SPRT's were selected to represent the NBS temperature scale. A special carrier was designed and the SPRT's were transported to the National Physical Laboratory (United Kingdom) where the intercomparison work is expected to begin soon.

11. Relevant Documents

a. "The IPTS-68 from 90.188 K to 903.89 K as Maintained at the NBS", G. T. Furukawa J. L. Riddle, and W. R. Bigge, to be published.

b. "Investigation of the Freezing Temperatures of NBS Aluminum Standards", G. T. Furukawa, J. Res. NBS 78A 477 (74).

c. "The Freezing Point of Aluminum as a Temperature Standard", G. T. Furukawa, W. R. Bigge, J. L. Riddle and M. L. Reilly, Proceedings of the European Conference on Temperature Measurement, April, 1975.

d. "The IPTS-68 from 13.81 K to 90.188 K as Maintained at the NBS", G. T. Furukawa, J. L. Riddle, W. R. Bigge, J. Res. NBS 77A 309 (1973).

12. Personnel Associated with this Project

G. T. Furukawa, Project Leader
J. L. Riddle
W. R. Bigge

IX. High Temperature Thermocouples - G. W. Burns

1. Introduction

The investigations of refractory metal thermocouples and thermocouple materials at high temperatures (above 1500 K) are directed towards providing pertinent information on their selection, preparation, properties, and performance; improving the reliability of sheathed thermocouple sensors for both nuclear and non-nuclear applications; and advancing the general state of the art of high temperature thermocouple thermometry. When promising new thermocouples that have application below 1500 K become available, it is also important to characterize their behavior and to prepare tabular values of emf and temperature to facilitate their use.

The refractory-metal thermocouple investigations were initiated under the sponsorship of NASA (Lewis) and had application in the use of thermocouples for nuclear reactors in space propulsion. The investigations were most fruitful and are being continued. Major AEC contractors have particular need for this knowledge in their design studies and development of nuclear reactors (fast breeder and gas cooled.) The Oak Ridge National Laboratory has emphasized the importance of characterizing certain temperature dependent properties of W-Re alloys and other refractory-metals so as to provide a basis for selecting optimum materials for use in sonic, noise, and thermocouple thermometers that are essential in irradiation experiments. In addition, there is a need for a more reliable (longer life) thermocouple than the presently used Type K thermocouple for use in monitoring temperatures in critical industrial applications such as the coolant loops of nuclear reactors, heat-treating furnaces in the aircraft industry, and the processing of semiconductor devices.

The evaluation of the performance and properties of thermocouple materials and thermocouple sensors at high temperatures are performed by heating them in existing high temperature furnaces and annealing apparatus in which high purity gaseous environments or vacuum can be created. Appropriate handling, cleaning, heat treating, and measurement techniques are employed; materials are monitored by metallurgical and chemical procedures.

2. Recent Progress

The testing of several Mo sheathed, BeO insulated W-3% Re/W-25% Re and W-3% Re/W-15% Re thermocouple sensors, which was begun during the previous FY, was terminated after 2094 hours of exposure at 1800 °C. The emf drifts of the thermocouple sensors were less than the equivalent of 3 °C during the test.

Tests with 0.25 mm diam doped Mo and undoped Nb wires were continued. Specimens of the doped Mo were exposed for periods up to 1000 hours at 2000 °C in high purity argon. While the exposed Mo specimens had excellent room temperature ductility, they exhibited gross changes in their thermoelectric properties. Spectrochemical analysis of the "as received" Mo wire indicated the presence of about 0.3% silicon. We suspect that a change in the physical state or loss of this impurity leads to the observed thermoelectric instability. In the case of the undoped Nb wire, attempts to electrically heat wire specimens for periods in excess of about 20 hours have been unsuccessful due to mechanical failure of the wires. An additional lot of the doped Mo wire was ordered for evaluation. Some undoped Mo and Nb-1% Zr wires were ordered as well.

Our previous examinations of some commercially built W-3% Re/W-25% Re thermocouple sensors that were heated in excess of 10,000 hours showed the formation of sigma phase in the W-25% Re wire. Tests were undertaken with commercial W-25% Re thermocouple wire to further investigate this behavior. Specimens of W-25% Re wire were heated for 1000 hours at temperatures of 1300 and 1500 K. Examinations of them by X-ray diffraction analysis revealed sigma phase. Large changes in their thermal emfs were also found. We discovered that the formation of sigma phase and large emf changes in the W-25% Re wire could be virtually eliminated if the wire was first given a short anneal (2 minutes) at 2400 K.

A joint program with the Oak Ridge National Laboratory (ORNL) was initiated to characterize various temperature dependent properties of W-Re alloys containing up to 26 percent Re by weight. The alloys for the program were given appropriate heat treatments by NBS to stabilize their electrical properties. These alloys were acquired under the earlier NASA contract. A quantity of the heat treated alloys was supplied to ORNL.

Wet chemical and mass spectrographic analyses were completed by NBS. Metallographic examinations and tensile tests were performed by ORNL.

In collaboration with Mr. Noel Burley, a guest scientist at NBS from the Australian Materials Research Laboratories (AMRL), studies were initiated on the electromotive force characteristics of Nicrosil and Nisil. These are new, highly stable, nickel-base thermocouple alloys developed by AMRL. They are suitable for use in air at temperatures up to about 1300 °C. The materials for the program were supplied by three manufacturers in this country and two European manufacturers to rigorous chemical and thermal emf specifications. After making preliminary calibration checks of all the materials supplied, a number of the most representative wire lots were selected for further study. Samples from these lots were tested against the platinum thermoelectric reference standard, Pt-67, at temperatures ranging from -110 to 1300 °C. Sufficient data for developing tables of emf and temperature for the Nicrosil/Nisil thermocouple in the above range were obtained. Other tests were performed to establish what effect variations in the major solute levels have on the emf-temperature relationship of the alloys. Tests were also made to investigate the effects of residual internal stresses. As a result of this investigation, suitable heat treatments were developed for relieving stresses in the "as received" materials. The high temperature phase of the program was completed in April, and Mr. Burley, with personnel of the Cryogenics Division at NBS, Boulder, has gathered the necessary low temperature data. Preliminary tabular reference values were established and have been sent to the thermocouple manufacturers and various ASTM E-20 committee members for evaluation, prior to formal publication.

At the request of the Aerospace Guidance and Metrology Center (AGMC) at the Newark Air Force Station, a commercially built furnace was modified so that it could be used to heat a gold freezing point cell that we previously assembled for them. Modifications were made by the NBS Instrument Shops Division. The furnace and the previously assembled gold freezing point cell were delivered to AGMC.

To assist the NBS Center for Building Technology with an evaluation of the instrumentation and data logging systems for a solar energy mobile research laboratory which was built by Honeywell, Inc. with support from the National Science Foundation, we examined and evaluated the thermometry systems. In order for Honeywell engineers to precisely compute the efficiency of the solar heating system, accurate measurements of temperature differences in the fluid heat transfer loops are essential. Seven sheathed thermocouples located at critical points in the heat transfer loop were removed and calibrated by us against a platinum resistance thermometer in a stirred liquid bath. The digital voltmeter used to measure the thermocouple outputs was also checked by us.

Finally, NBS Monograph 125 "Thermocouple Reference Tables Based on the IPTS-68" became available for distribution. This work, a collaboration between the Heat and Cryogenics Divisions of the NBS, contains reference emf values that cover temperature from -270 °C to 1800 °C for seven commonly-used types of thermocouples. It will be the standard reference guide for thermocouple and temperature control equipment manufacturers throughout the United States, as well as an indispensable tool for major users of thermocouples in process control and instrumentation applications. To prepare for the expected volume of demand, 20,000 copies were run off in the first printing.

3. Relevant Documents

a. "Thermocouple Thermometry", G. W. Burns, Proceedings of the European Conference on Temperature Measurement, April 1975, to be published.

b. "Nicrosil and Nisil; Their Development and Standardization", N. A. Burley, G. W. Burns, and R. L. Powell, Proceedings of the European Conference on Temperature Measurement, April 1975, to be published.

c. "Highly Stable, Sheathed, Beryllia Insulated, Tungsten-Rhenium Alloy Thermocouples", G. W. Burns and W. S. Hurst, Proceedings of the International Colloquium on High Temperature In-Pile Thermometry,

Petten, Netherlands, December 1974, to be published.

d. "Thermocouple Reference Tables Based on IPTS-68", R. L. Powell, W. J. Hall, C. H. Hyink, Jr., L. L. Sparks, G. W. Burns, M. G. Scroger, and H. H. Plumb, NBS Monograph 125, March 1974.

e. "High Reliability Sheathed, Beryllia Insulated, Tungsten-Rhenium Alloy Thermocouple Assemblies - Their Fabrication and Emf Stability", G. W. Burns and W. S. Hurst, NASA CR-134549, NBSIR 74-447, June 1974.

4. Personnel Associated with the Project

G. W. Burns, Project Leader
W. S. Hurst
M. G. Scroger

X. Thin Film Temperature Sensors - W. S. Hurst

1. Introduction

Temperature sensors that can be miniaturized, are of low thermal mass and exhibit fast response, are stable, and are sensitive to small temperature changes would find application in areas as diverse as the study of biochemical reactions and oceanography. These requirements may be met in a sensor whose active element consists of an evaporated thin-film capacitor. Such capacitors can exhibit a relatively strong temperature dependence that derives from electron trapping effects in the thin amorphous dielectric film. The first studies of such devices have been reported by Maserjian (1). He constructed 0.5 mm diameter, 400 nm thick capacitors that were supported by a thin anodic oxide film and obtained sensors with sensitivities of 80 μ °C and response times of less than 1 ms.

Initial work at NBS will be to further develop and characterize thin film temperature sensors. A vacuum system for fabrication of thin film multilayer devices employing several deposition techniques and accurately registered masking will be constructed. Fabricated sensors will be characterized as to their temperature stability, frequency dependence, response time, dielectric strength and noise level. In addition, information will be sought on the shape of the space-charge barrier within the dielectric, and the average trap density and cross-section. The materials of construction, the method of fabrication, the sensor design and the insulating coating are all variables that will need to be investigated if optimum performance is to be achieved.

2. Relevant Document

1. Maserjian, J., Temperature IV, Part 3, pp. 2159-2167 (Instrument Society of America, Pittsburgh, 1972).

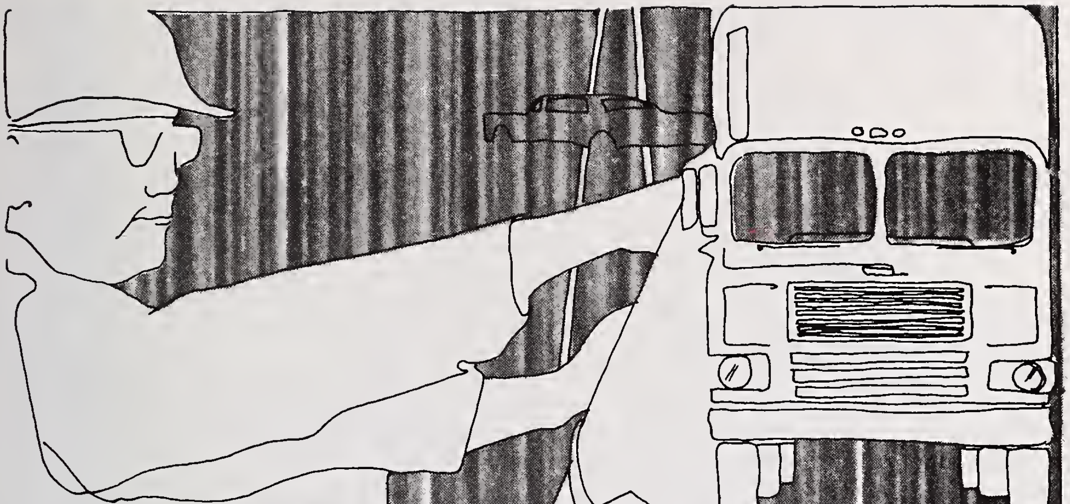
3. Personnel Associated with this Project

W. S. Hurst, Project Leader.

XI. Interaction With Other Government Agencies

Although for the most part those portions of the various Temperature Section programs which involve thermometry applied directly to the needs of other government agencies were discussed in the corresponding preceding sections of this report, it may be useful to summarize them in a separate section for easy reference. Accordingly, the following is a list of such projects, some involving a transfer of funds and others not, along with the agency and the Temperature Section program.

Project	Agency	Temperature Section Program
Scanning Radiometer	Department of Housing and Urban Development (via the NBS Center for Building Technology)	Medical Thermometry (Section IV)
Thermistor M.A.P.	Woods Hole Oceanographic Institute	Medical (Section IV)
Thermometry in medical diathermy	Bureau of Radiological Health, FDA	Medical (Section IV)
Clinical Laboratory Temperature Calibration	National Institutes of Health and various Hospital Clinical Labs	Medical (Section IV)
Deep Ocean Thermometry	National Oceanographic Instrumentation Center	Platinum Resistance Thermometry (Section VIII)
Standard PRT M.A.P.	U.S. Army, U.S. Air Force	Platinum Resistance Thermometry (Section VIII)
Metallurgy of W-Re Alloys	Oak Ridge National Laboratory	Platinum Resistance Thermometry (Section VIII)
Gold-point Calibration Facility	U. S. Air Force	High-Temperature Thermocouple (Section IX)
Mobile Solar Research Laboratory Calibration	National Science Foundation (via the Center for Building Technology, NBS)	High-Temperature Thermocouple (Section IX)
Thin-Film Capacitance Thermometers	National Institutes of Health	Thin-Film Temperature Sensors (Section X)



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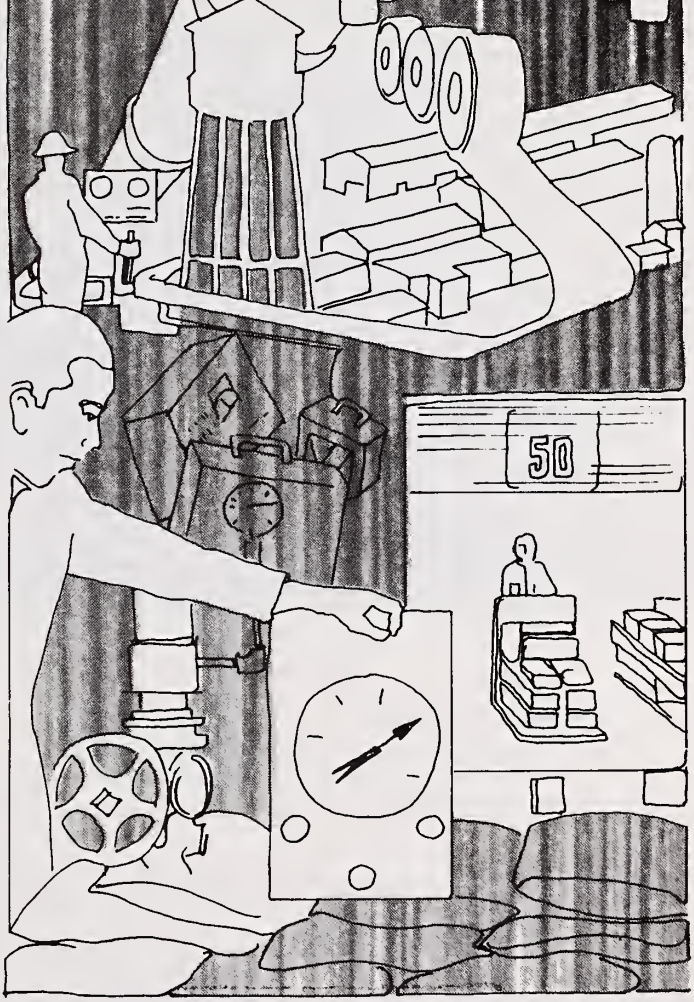

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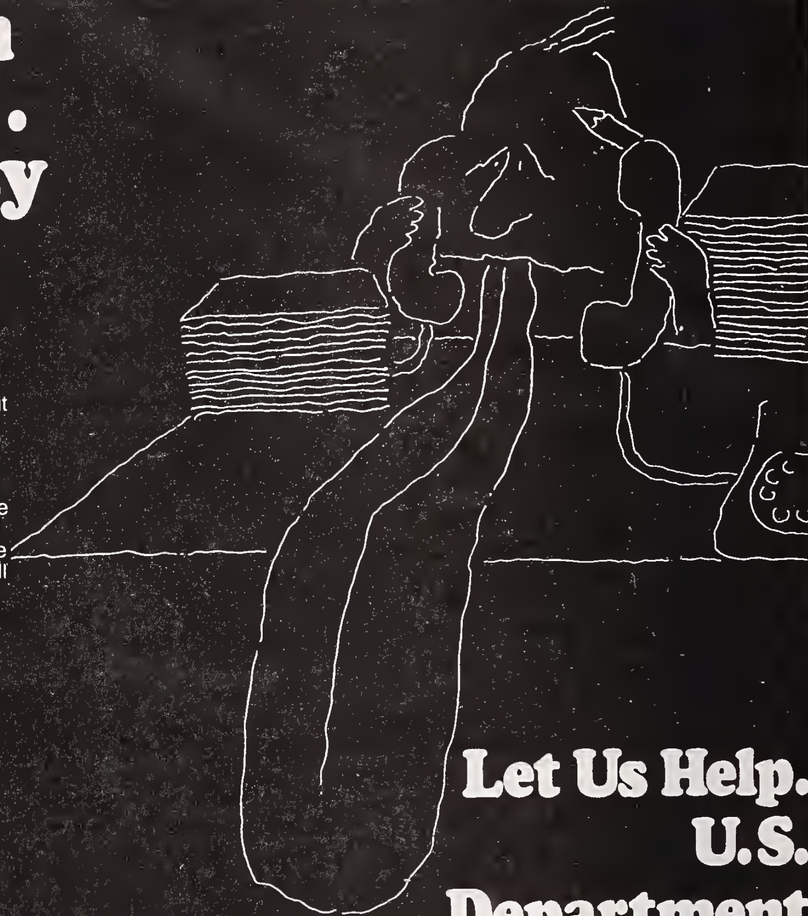

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16. ABSTRACT *(A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here.)*

This report summarizes the progress in calendar 1974 of the technical program of the Temperature Section of the Heat Division, Institute for Basic Standards. Separate sections of the report are devoted to the various projects, to highlights of the year's activities, and to other agency interactions.

17. KEY WORDS *(six to twelve entries; alphabetical order; capitalize only the first letter of the first key word unless a proper name; separated by semicolons)*

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