

ISOTECH
JOURNAL OF
THERMOMETRY

Volume 2 Number 1, 2nd Quarter 1991

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The Isotech Journal of Thermometry is published and distributed by subscription twice a year, by Isothermal Technology Inc., Southport PR9 9AG England, and 2307 Whitley Drive, Durham, North Carolina 27707, U.S.A. The subscription fee is \$15 or £10 for orders including a cheque and \$25 or £16 for orders which require an invoice to be sent.

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10 YEARS OF ISOTHERMAL TECHNOLOGY LTD

Isothermal Technology Ltd. was incorporated in the Autumn of 1980, and began operating on January 4, 1981. We record our subsequent milestones as follows:

1981: The first products were metal block apparatus for thermocouple reference. During the year, new products were designed and introduced at the rate of approximately one per month.

1982 and 1983: We widened our range of thermocouple referencing equipment and introduced new products for thermometer calibration.

1984: We were invited to exhibit at Open Days of our National Physical Laboratory, where we first showed our spherical furnace for thermocouple calibration, and our self-calibrating thermocouple.

1985: We had developed our range of liquid bath products, and applied for approval as a member of the British Calibration Service (BCS).

1986: We achieved approval, and became part of the British Calibration Service.

1988: Our product range was reviewed, for appearance and ease of function, by a design consultant, and in 1988/1989 began introduction of repackaged products which still represent the basis of our wide range.

1989: We acquired the Basic Metrology Products for thermometer calibration against the definitions of the Temperature Scale from YSI, and with it began our association with Henry Sostmann. With these, we have an offering of primary temperature equipment (fixed point cells and standard platinum resistance thermometers) and apparatus for secondary laboratories and industry (a wide range of fluid baths, block furnaces, thermocouple references, transmitters, and resistance and thermocouple sensors)

These, together with our accredited calibration laboratory, close to national standards and empowered to issue internationally accepted and traceable certifications, gives us a unique opportunity to serve temperature metrology.

1990: We began publication of the Isotech Journal of Thermometry, a semiannual Journal of interest to thermometrists and laboratory managers everywhere.

1991: Looking to the near future, we will continue to develop new products and look for new opportunities. In particular:

We will offer open metal fixed point cells as well as sealed for primary laboratories, and a line of smaller and less costly cells for high-end industrial use. We will develop a service center and manufacture selected products in the United States.

Please keep these names and addresses in mind, and record them in appropriate records:

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We look forward to hearing from you!

FUNDAMENTALS OF THERMOMETRY PART III

THE STANDARD PLATINUM RESISTANCE THERMOMETER

by Henry E. Sostmann

ABSTRACT

The Standard Platinum Resistance Thermometer, acronym SPRT, is the stipulated interpolation instrument for realizing the International Temperature Scale of 1990 between the defining fixed points, over the range from 13.8 K (the triple point of eka-hydrogen) to 961.78 °C, the freezing point of silver. However it must be said immediately that no single thermometer or design of thermometer can be expected to cover this entire 1221 K span, but that it can be covered by three designs of thermometer, with substantial overlap of range. The three designs are the long-stem SPRT, the cryogenic or capsule SPRT and the high-temperature SPRT or HTSPRT.

1: THE LONG-STEM SPRT AND ITS USEFUL RANGE

The long-stem SPRT is the most commonly encountered of the types, and is the accepted interpolating standard over that portion of the temperature range from -189.352 °C (the triple point of argon) to the freezing point of zinc (+419.527°C).

The principle reason for limiting the range at the cold end is that the thermometer tube, which extends from the thermal environment into ambient, can conduct unwanted heat into a cryogenic situation. There is usually no difficulty about using this thermometer down to -200 °C.

The upper limit of +420 °C is a convention only. Certain such thermometers (those with quartz sheaths and high-temperature mica insulation) can be used, with proper precautions, to 630 °C; others (with Pyrex sheaths) can be used safely below 500 °C. 630.74 °C, the freezing point of antimony, was a secondary fixed point of the International Practical Temperature Scale of 1968, which the ITS-90 replaces, and was considered to be the upper end of the platinum thermometer range on that Scale. The antimony point, which was not very satisfactory to realize, has been replaced by the freezing point of aluminum, 660.323 °C, and this must be considered beyond the safe upper limit for any mica-insulated SPRT.

On IPTS-68, it was customary to issue a calibration table for SPRTs with an upper limit of either 500 °C or 630 °C, as the construction of the thermometer permitted and the customer requested. The calibration points were the water triple point and the freezing points of tin and zinc, and the propagation of a 1 mK error at tin and zinc could produce an uncertainty of about 8 mK at 630 °C.

On ITS-90, there are a number of specified ranges above 0 °C. These include the range 0.01° to 419.527 °C (the freezing point of zinc) and 0.01 °C to 660.323 °C (the freezing point of aluminum). Current NIST policy on calibration ranges is as follows:

"If a customer requests that his SPRT be calibrated through the zinc point and that we provide tables with extrapolation to 500 °C, we will honor that request, with a warning that the uncertainty is unknown above 420 °C. We will not extrapolate to 630 °C.

If we get a request to calibrate an 8163 or 8167 type SPRT to the aluminum point, we call the customer and advise him that the SPRT should not be calibrated at that temperature, and that if it is, it may be destroyed in calibration. If he, nevertheless, insists upon such a calibration, we will provide it, reluctantly.⁽¹⁾

The platinum resistance thermometer has a long and honorable history. It was proposed first by Siemens in a Bakerian Lecture in 1871. Siemens' thermometer consisted of a fine platinum wire wound on a fire-clay cylinder, which was then enclosed in a protective iron tube. The thermometer gained rapid acceptance, due principally to the reputation of its creator, and was abandoned almost as quickly when it was discovered to be unstable (due most likely to contamination).⁽²⁾ Also, at the cost (then) of refined platinum and good porcelain, it was too expensive for industrial use.

Callender, less than twenty years later, revived the platinum thermometer for laboratory work. Instead of a mandrel of fire clay or porcelain, he wound the platinum element on mica. He realized also that any necessary joints must be autogenously welded, since the migration of components of solders would contaminate the pure platinum. The first documented statement of an interpolation algorithm for platinum, the Callender equation (later modified by van Dusen to include temperatures below 0 °C) is given in the epochal Callender paper, "On the practical measurement of temperature" read to the Royal Society in 1886.⁽³⁾ The elements of today's thermometer are all here, waiting only refinement and reduction to practice. Among the numerous refiners, Griffiths, Holborn and Wien, Barber, one worker whose name stands out is C. H. Meyers, and a seminal paper of his is reproduced elsewhere in this issue of the Isotech Journal.⁽⁴⁾

2: THE 25 OHM LONG-STEM THERMOMETER FOR -200 ° TO ABOUT 600 °C CONSTRUCTION OF THE THERMOMETER ELEMENT

I have elected to begin discussion of construction with this type, because it includes the basic principles, and the other two are variants on it.

The thermometer element is considered to be that length of platinum wire which is active in the measuring circuit and the structure which supports it. In standards use, the element is always connected as a 4-wire resistor, so that the long lead wires which extend up the thermometer tube, and the cable from the thermometer head to the resistance-measuring instrument, do not enter into the resistance determination.

In the traditional constructions, the former or mandrel upon which the platinum wire is wound is a cross made of thin sheet mica. The four edges of the mica are cut with fine teeth or grooves to contain the wire, which is on the order of 0.1 mm (0.004 inch) in diameter. The grooves form a double helix, so that one half of the platinum wire is wound toward the outboard end of the element, leaving each intermediate groove empty, and the remaining wire is wound back into these grooves. The result is a bifilar winding which is approximately non-inductive (but which may have capacitive reactance). Fig. 1 shows, sketchily, such an element. The resistance is commonly about 25.5 Ω at 0°C. (This seemingly odd value was chosen to allow crude estimation; for a 25.5 Ω thermometer, at moderate temperatures, 0.1 Ω represents approximately 1.0°C.)

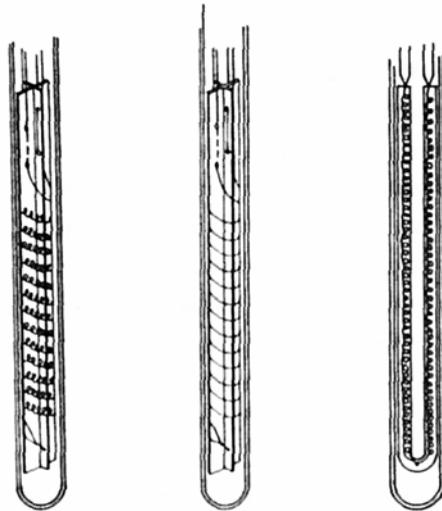


FIG 1

FIG 2

FIG 3

The mica former limits the upper temperature at which the thermometer may be used. Mica is a natural material; any of several silicates, including muscovite (ruby mica) and phlogopite (amber mica). Both forms are bound with water, and will deteriorate and flake at temperatures which drive off the water. Phlogopitic mica is used in the later quartz-tubed designs, and is limited to below 600°C for frequent use. The older designs using Pyrex tubes and ruby mica are limited to 500°C.

The wire is the purest platinum obtainable. It is referred to as "reference grade", taking the name from its acceptability as the reference leg of a standard platinum-platinum/rhodium thermocouple. Refiners of platinum, of whom only one or two produce thermometer-grade metal, occasionally make a bar of platinum which is recognized as superior, and thermometer makers reserve long-term supplies from that specific bar.

3: OTHER DETAILS OF CONSTRUCTION

The four lead wires which proceed up the thermometer stem (tube, sheath) and into the head, or handle, are generally 0.4 mm (.015 in) diameter. They are ordered, spaced and kept from touching each other by some sort of insulating device; a common plan is to use a number of four-hole mica washers, often separated by spacers of quartz tubing. Early thermometers were made with gold leads, perhaps as a matter of cost of material (or see the Meyers paper in this issue of the Journal). This had the disadvantage of placing the platinum-to-gold junctions at the hot end of the thermometer, where thermoelectric effects could occur. Most thermometers are now made with lead wires of commercial grade (or better) platinum extending into the cool thermometer head, where they are attached to the copper leads of the external cable, with great attention paid to isothermality by appropriate heat-sinking.

At the cool end of the thermometer tube (which in today's thermometers is always quartz) the lead wires are hermetically sealed into the tube. In early thermometers this was done by sealing the platinum directly through the glass, or by Housekeeper seals. Both such seals effectively prevent repairs of broken tubes except by the most heroic means. For a number of years, it has been customary to close the cool end of the tube with a glass-to-metal header including four Kovar tubes, into which the lead wires are sealed by hydrogen brazing. The glass seal includes a fifth tube which is used to evacuate the tube assembly, perform occasionally arcane and sometimes mystical cleaning processes, to finally fill the tube with a gas, and to seal off. The gas in a long-stem thermometer of this range is commonly 90% dry

argon, 10% oxygen, at a pressure which will be equivalent to 1 atmosphere at the hot end of the thermometer's range.

The quartz tubes of most thermometers are clear at the hot end for the length of the element, and for the rest of their length are roughened by sand-blasting, or coated with a black material such as Aquadag. Quartz has marvellous optical properties, and it is necessary to avoid piping heat out of (or into) the sensitive element by breaking up internal reflections of radiant energy within the walls of the quartz tube. (For fun, try pointing one end of a quartz tube at a wall while heating the other end luminously hot. The circle of light projected onto the wall drives the lesson home).

The four wires in the external cable are required to (a) be flexible and (b) represent constant resistances under such conditions as flexing. While the lead wires and the cable are not measured during a four-wire measurement, any wire not constant in resistance might change and cause errors during a measurement, say with a Mueller bridge, which requires several steps; or with a modern automatic bridge could be a source of noise. Requirement (a) could be met with stranded wire, but the strands of a stranded cable touch each other in random and inconstant parallel connections, which contradicts requirement (b). The wire commonly used is Litz wire (Litzendraht), in which the individual strands are enamel-insulated from each other except at the ends where they are soldered to the platinum lead wires or to terminal lugs. The solders used should be thermal-free. The four Litz wires are made into a cable by some sort of woven serving. Terminal lugs are advisedly gold-plated copper, appropriately labeled as current or potential leads.

4: VARIATIONS ON THE ABOVE

Virtually all thermometer designs agree with the description given above, but there are a number of different ways of arranging the platinum wire relative to its supporting structure.

It is essential that the wire, whose resistance versus temperature characteristic is the essence of the measurement, be and be maintained in a fully annealed and strain-free condition. Strained or work-hardened platinum can will be higher in resistance and lower in sensitivity than it is in the fully annealed condition. Meyer's mica cross attempted to speak to this by providing essentially point contacts between platinum and the supporting structure, and leaving it freely suspended elsewhere. Fig. 1 shows a design in which the platinum is first coiled into a helix (with a pitch diameter of about 0.4 mm (.0125 in) and then wound on the mica. There are about 30 turns; thus there are about 120 places at which platinum touches mica. Fig. 2 shows a later Meyers design in which the platinum is not coiled, but is wound straight on the cross, for about 120 turns, or about 480 points of contact.

The first Meyers design coils the wire to allow more ohms in the linear length. The second design is an attempt to place more of the wire nearer the inside diameter of the quartz, to reduce thermal losses and thermal lag, and to improve dissipation of I^2R heat. Whether one prefers Fig. 1 or Fig. 2 is a highly personal matter. My preference is for the coiled-coil design of Fig. 1. I have never found the self-heat or lag to be a nuisance, and I feel that Fig. 1, in which the platinum enjoys an additional degree of freedom, is marginally more stable.

Fig. 3 is a design by Barber, of NPL, in which the coil of platinum wire is allowed to hang freely in a U-shaped quartz capillary inside the main thermometer tube. Other designs, such as that of Curtis at Rosemount Engineering, dispose a coil of platinum inside a number of axial holes in ceramic, or on two sides of a helically-twisted ribbon of quartz which serves to separate the two coils. My experience suggests that all of these designs are marginally inferior to Meyers', in the amount of space which is left between the quartz tube and the sensing wire, and the consequent thermal loss and response lag; and the larger distance from the platinum to the quartz makes self-heating effects worse to much worse.

5: THE CRYOGENIC, OR CAPSULE, THERMOMETER

The cryogenic, or capsule thermometer, is short (usually less than 50 mm (2 in) long. The point is that does not have a stem which must emerge from the thermal zone into ambient, and does not serve as a conductor of unwanted heat into a zone one is trying to keep very cold. It is often built into equipment in a semi-permanent fashion. Its lead wires are also short, and usually are welded to thin copper or Constantan extensions, which are carefully thermally tied to cold parts of the apparatus. The element designs are similar to those for long-stem thermometers. The tube or sheath is usually not quartz but commercial grade platinum. The gas used to fill is usually helium with a fraction of oxygen, to facilitate thermal transfer at temperatures where the conductance of helium is superior to that of any other gas.

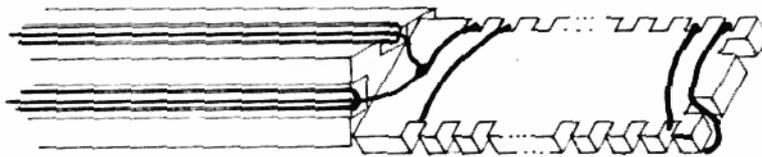
While intended for use at low temperatures, the capsule thermometer range extends from 13.8 K to as high as 250 °C, and it is used not only in cryogenics but also in equipment, such as calorimeters, where it is desirable to build in a short sensor semi-permanently.

6: THE HIGH-TEMPERATURE PLATINUM RESISTANCE THERMOMETER

The extension, in ITS-90, of the platinum range to the freezing point of silver (961.93 °C) has required the development of a new platinum resistance thermometer; acronym HTSPRT.

The first problem of extending the range is insulation. Mica cannot tolerate the temperatures. It has been necessary to make the former on which the platinum is wound of high-purity quartz. Even quartz does not provide absolute isolation at the high temperature end of the range (I have tried monocrystalline sapphire, at much greater cost than quartz, and it is about twice as good, which is not good enough). The former, or mandrel, is thus a shunt resistance across the platinum winding, and because of the uncertainty of the contacts between platinum and quartz, it is uncertain and unstable in magnitude. The practicable solution is to reduce the element resistance so that the shunt resistance produces a smaller network effect. For example, for a 25.5 Ω thermometer, suppose that the shunt resistance were 20 M Ω . Then the network resistance is 25.499967 Ω . But we require measurement assurance of better than 1 part per million, so this won't do, even if the shunt were a constant (calibratable) value, which it is not. For a 0.25 Ω thermometer, a 20 M Ω shunt gives a network resistance of 0.24999997 Ω , which is tolerable. The cost, and there is a cost, is increased difficulty on the electrical measurement side, particularly in the face of noise, which is present at high temperatures.

Another benefit accrues from a lower resistance. Platinum wire tends to show crystal growth above approximately °C. The inevitable result is a crystallization so profound that, in fine wire, fractures at the grain boundaries develop. The 0.25 Ω choice allows the use of much heavier platinum wire for the element, which prolongs life and minimizes drift. Indeed, Evans developed a 0.25 Ω high temperature thermometer, called the "birdcage", which employed platinum rods instead of wire and which was produced commercially, but years ahead of a scale to use it. In more conventional designs, the heavy wire allows the support to be a blade rather than a cross, reducing the number of quartz-to-platinum contacts by 1/2. Tavener has described such a thermometer in an earlier issue of this Journal⁽⁵⁾ and the detail of its element and lead arrangement is shown in Fig. 4. It is in current production, and has been shown by calibrations in several National Laboratories to be extremely stable.



CONTINUOUS CAPILLARIES
CONTAINING LEAD WIRES

SPADE AND 0.25Ω WINDING

(Isotech and I are studying the design of a thermometer, the top of whose range will be the aluminum point, the former of quartz, and the 0 °C value 10 Ω. For the user who does not require temperatures higher than 660°C, the higher resistance will be attractive).

Another problem which can occur at the higher temperatures, and which is subtle, is the migration of metal ions from the immediate environment, through the quartz (which is transparent to such ions as copper, iron, nickel and chromium at these temperatures) into the platinum, resulting in degradation of its purity and large consequent calibration shifts. The Inconel liner of a furnace will cause such an effect, if the thermometer is not isolated from it by, say, a thin sleeve of graphite. (The graphite crucible and sleeve of the Isotech fixed-point cells are sufficient protection). Isotech also furnishes a patented, battery-powered field generator which, when used with its Model 962 thermometers, will shield the thermometer against contaminating metal ions.

7: USING SPRTs: MECHANICAL

It has been said that, if one puts an SPRT down onto a workbench hard enough to hear it touch, it will probably be strained. Mechanical strain of the wires, due to shock or acceleration between their points of support, will shift a thermometer throughout its range, increasing W values below 0 °C and decreasing them above 0 °C, because the 0 °C value which is the W divisor has increased. The same effect can be caused by using the thermometer in an environment which imparts continuing vibration to it. Attention must be given to such factors during storage, as well.

When a thermometer is sent elsewhere for calibration, it has been my preference to carry it by hand, and to retrieve it the same way. If a thermometer must be shipped, it should be packed adequately in a rigid box, in turn contained in a box full of resilient material, and the box closed by some means which is not in itself shocking; e.g., a wooden crate closed by woodscrews, not by nails.

8: USING SPRTs: QUALITY ASSURANCE

Any thermometry laboratory should be prepared to realize the triple point of water, for at least the following reasons:

a) The ratio W of the resistance at temperature to the resistance at the triple point of water:

$$W = \frac{R(t)}{R(tp)}$$

is more stable and repeatable than the resistance. The calibration received from a calibration laboratory is given in terms of W vs. t, not R vs. t. To realize a temperature measurement accurately, the measurement of resistance at temperature must be followed by a measurement of resistance at the water triple point, and the W value calculated, so that the tables may be used.

Furthermore, Berry has shown that as platinum is heated, several surface oxidation states are produced sequentially, which alter the resistance. However a measurement at a temperature and a measurement immediately following, at the water triple point, report the platinum in the same state. (The original state may be restored by an anneal, as described in c) below).

(NOTE: In previous scales, the denominator for W was the resistance at 0 °C. In the ITS-90, it is the resistance at 0.01 °C)

The resistance at the water triple point is a good indication of the state of the thermometer with respect to change. Quality control requires that, each time the thermometer is used, the resistance at the water triple point be placed on a control chart. The history thus developed aids confidence immeasurably. I am not a believer in fixed calibration intervals for SPRTs. I am a believer in following the control chart, which will tell me whether and when the thermometer has shifted too much. Against risks in transportation, and compared to the present cost of a National Laboratory calibration, a water triple point system may pay for itself in one interval.

When the thermometer is returned after calibration, a check at the water triple point will indicate whether or not there has been a subsequent shift due to transportation, and whether one may use the calibration with confidence.

For those laboratories which are equipped to do their fixed-point calibrations in house (a rapidly increasing number) it is wise, before calibrating, to perform a measurement at the water triple point, then anneal for 4 hours at 450 °C, and measure at the water triple point again. Any measurable change will indicate that the thermometer is either unstable or not fully annealed. In the latter case, repeating the process until a constant value is obtained can save much calibration time and expense. (Recommendations for handling HTSPRTs during and after exposure to high temperatures, and annealing such thermometers, should be sought from the manufacturer).

9: USING SPRTs: SELF-HEATING EFFECTS

A resistance is passive; to measure it requires that a current be passed through it, and the inevitable effect is I^2R heating. It is typical practice in calibration laboratories to calibrate a 25.5 Ω thermometer with 1 mA applied, and a 0.25 Ω thermometer with 10 mA. (It is never wise to apply more than 5 times these current levels, even momentarily). These calibration currents will result in a slightly elevated temperature. It is the First Law of Thermometry that a thermometer measures no temperature other than its own. In the designs of Fig. 1 and Fig. 2, the elevation, at the water triple point temperature, may be as much as 0.5 mK; with designs of less favorable heat transfer it may be substantially higher.

More accuracy may be achieved, at more effort, by reducing the reading to the resistance which would be obtained if there were no source of power. This can be done by measuring the resistance at two currents, for example x 1 and $x \sqrt{2}$ the accustomed level, in the identical environment. Converted to terms of power, the zero power resistance can be extrapolated.

The heat generated by the thermometer is, of course, transferred to the medium in which it is immersed. The self-heat so dissipated in a large volume of flowing liquid is then less than that in a suffocating environment. In most circumstances, the heat generated will not be such as to consequentially raise the temperature of the environment.

10: USING SPRTs: INTERPOLATION CALCULATIONS

The mathematics of the ITS-90 has been treated elsewhere, and need not be repeated.

References 6, 7, 8, 9, 10 and 11 contain complete details. Reference 9 provides valuable advice for those who specify and order calibration services. For laboratories equipped to calibrate at fixed points, Isotech has available Daedalus 1.1, an MS-DOS interpolation program covering the full SPRT ranges. For information, consult Isothermal Technology Ltd., Pine Grove, Southport, England, or Isothermal (USA) Ltd, 250 West 57 Street, New York N.Y. 10107.

11: USING HIGH TEMPERATURE SPRTs

Quartz, as used in high-temperature thermometer sheaths, is a non-crystalline form of a material which may lapse into the crystalline form at temperatures within the upper range of HTSPRTs. This will occur inevitably if the thermometer sheath is exposed without being scrupulously cleaned of any contaminants; dust, oil, fingerprints. Manufacturers will advise methods of cleaning. The effect of devitrification is to make the quartz leaky to gasses, including its own internal gas, and ultimately to destroy its mechanical strength and integrity. For the calibration points below aluminum, HTSPRTs may be handled in the same way as the 25.5 Ω conventional thermometers. That is, the thermometers are preheated to avoid sudden cooling of the fixed-point cell when they are inserted, and to prevent excess shock to the thermometer.

At the higher calibration points (aluminum and silver), and in measurement situations at higher temperatures, special precautions are necessary, in inserting and also in removing the thermometer, to prevent excessive shifts. When used above 500 °C, it is advisable to raise the temperature of the environment containing the thermometer at a rate which achieves the desired temperature in 2 hours or more. This can be accomplished in either of two ways; at the beginning of the cycle, by placing the thermometer in a furnace which is below 500 °C and ramping the temperature gradually, or by preheating the thermometer in a second furnace on a similar time program. After the measurements are made, the thermometer should be allowed to cool in the furnace at a rate not exceeding a drop to 480 °C in 2 hours. At 480 °C the thermometer may be withdrawn safely.

It is usually necessary, during calibration, to withdraw the thermometer from the cell, so that the inner mantle can be formed in the cell using quartz rods. This is best done by raising the thermometer so that its tip is located a few centimeters above the graphite lid of the cell, waiting 5 minutes or so, raising the thermometer another 5 cm, waiting another 5 minutes, etc., until the thermometer is free from the cell guide tube. The thermometer may be transferred to another furnace at the appropriate temperature. When the thermometer is re-inserted into the cell, the procedure is followed in reverse, until the thermometer is bottomed in the cell.

The shifts which may be observed when a thermometer is cooled too rapidly are due to quenched-in crystal lattice vacancies. These can usually be removed, and the thermometer restored, by heating the thermometer, with the rise time precautions given above, to 960 °C, and holding it there for 30 to 60 minutes; then gradually cooling to below 500 °C before withdrawing the thermometer. A check at the water triple point will indicate whether the treatment has been adequate, or whether it needs to be repeated.

In Part IV of this ongoing series, we will consider resistance-measuring equipment for standard platinum resistance thermometry, and industrial platinum sensors.

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THE PLATINUM RESISTANCE THERMOMETERS OF C. H. MEYERS

Introduction by Henry E. Sostmann

That great pioneer of modern electrical thermometry, H. L. Callender, believed that, in the resistance variation with temperature of platinum wire, he had identified a universal and transportable standard of temperature. He wrote: "One has only to place a length of platinum wire in an ordinary envelope, and send it by post!"

Later studies confirmed that platinum wire makes an almost ideal temperature transfer standard, but that the envelope, which configures and protects the "length of platinum wire", must be extraordinary.

Cecil Hope Meyers, of the National Bureau of Standards, was as responsible as anyone for developing the platinum resistance thermometer into the superb instrument we know today. Harold Hoge recalls him as a "tall, soft-spoken, pleasant man competent in several fields. He was highly gifted in his ability to construct delicate apparatus." John Evans, who met him only several times when he visited after retirement, comments: "My most lasting impression of Meyers came from those jigs and tools that I inherited" (when Evans took over the development of high temperature thermometers at the NBS in 1955.) "They were what Stimmie (H. F. Stimson of the NBS) called CANBE's - cheap and nasty but effective. Many of my own devices were patterned after his, and they remained part of my lab collection throughout my entire life at the NBS."

Meyers' career at the NBS began in 1914. I have been unable to discover when he left. He was frequently seen at the NBS as late as 1948, possibly as a visiting scientist. In retirement he conducted a home business, making some of the best platinum resistance thermometers of his age, and he knew how special they were. Each one was individually signed. Don Toenshoff tells me that, when he was a youngster at Engel-hard Industries, he sought a quotation on a Meyers thermometer. There was a long silence, and then one day Meyers appeared in his laboratory to "see if he deserved one." When Meyers finally stopped working, the Meyers thermometer was transferred to Leeds and Northrup by Don Robertson, and later from Leeds and Northrup to YSI by me.

We reproduce here C. H. Meyers' seminal paper, Co.Leed F-1-lament P.ea.ttnwn Re-e .6-tance Thenmome ten.a, which appeared originally in the NBS Journal of Research, 9, 807 (1932).

COILED FILAMENT RESISTANCE THERMOMETERS

By C. H. Meyers

ABSTRACT

Platinum resistance thermometers with strain free windings which are smaller than those previously used are described, and the method of construction is given. By double helical winding the coil of a 25 ohm thermometer is reduced to about 5 mm diameter and 2 cm length, and the outside diameter of the thermometer is about the same as that of the ordinary mercury-in-glass thermometer. These thermometers are hermetically sealed, and thermometric lag is reduced by filling them with helium.

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I. INTRODUCTION

In a previous paper, T. S. Sligh has discussed the properties of platinum resistance thermometers in general, explained the method used for calculating temperatures, and described the construction of resistance thermometers ordinarily used at this bureau.

Strain-free thermometers with smaller dimensions have sometimes been found desirable for use in special apparatus, and to fill this need several such thermometers have been constructed. When these thermometers are mounted in a glass tube the over-all diameter is about 7 mm, or about the size of an ordinary mercury-in-glass thermometer. Since the length of the coil is only about 2 cm this design of resistance thermometer is suitable for any location where a mercury thermometer may be used.

A detailed description of this special design of thermometer coil and its construction will be given as well as a description of modifications in the design of leads and thermometer head.

II. THERMOMETER COIL

The design of the thermometer coil is illustrated in Figure 1, which is a photograph of a model built for illustrative purposes. In Figure 1, (A) shows this model mounted on the quartered mandrel upon which it was wound, and (B) shows the coil removed from the mandrel. The size of the actual thermometer coil is illustrated by the scale drawn in (A).

In the construction of the thermometer coil, the platinum winding was mounted upon a mica cross of the type mentioned in Sligh's paper. The various steps in the construction of this cross and the coil are given in Figure 2. The dimensions given are those actually used and are given in millimeters unless otherwise stated in the figure.

¹B. S. Sei. Papers, vol. 17 (S407), p. 49, 1922.

Mica was cut into strips by clamping the part to be used under a bar of the proper width and trimming away the portions extending on each side by a repeated back and forth motion of a sharp-edged steel tool. As mentioned in the paper already referred to, a thin safety-razor blade forms a convenient source of material for such a tool.

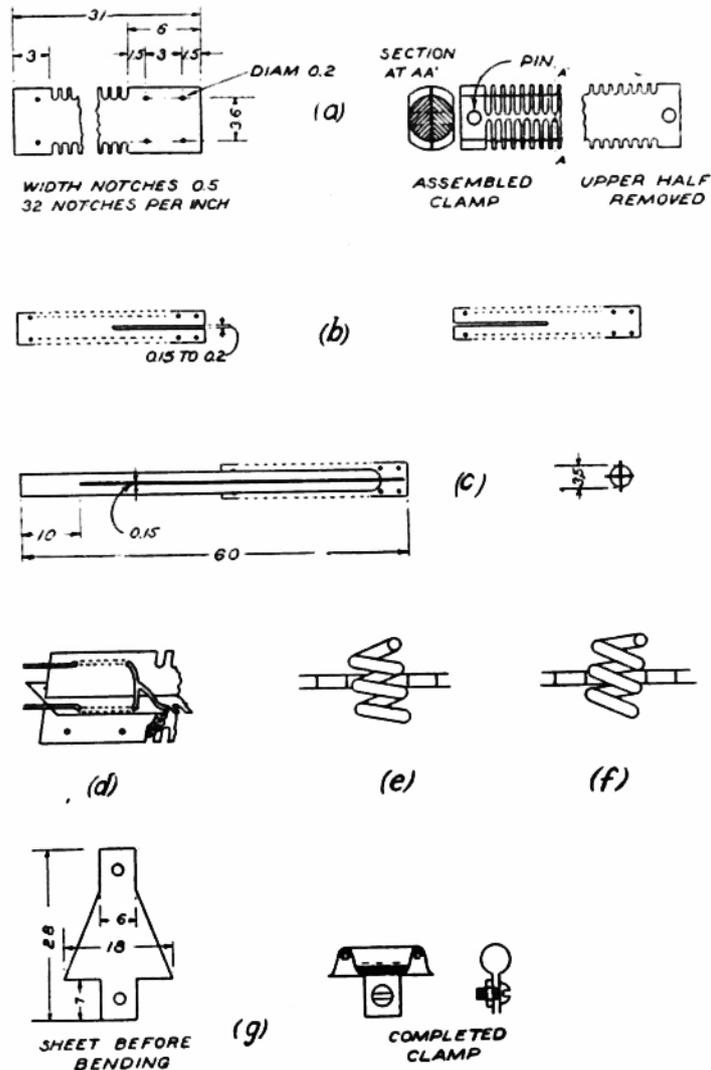


FIGURE 2.—Construction of thermometer coil

The mica strip may be split to the desired thickness (about 0.1 mm), or teeth may cut in the strip before splitting. For cutting the teeth the mica was placed in a toothed clamp, such as shown to the right in Figure 2 (a), the halves of which were kept in alignment by pins. This clamp was gripped between vise jaws. Teeth were cut in the mica with a special saw consisting of 0.5 mm steel wire mounted upon a light jeweler's hack-saw frame. The saw cut equally well when the wire was charged with carborundum by rolling between two plates of glass or when the wire was merely roughened by rubbing transversely with a carborundum stone. This saw

produced a smooth-edged notch with parallel sides and a rounded bottom. Both steel piano wire and nickel wire have been used successfully in cutting notches from 0.1 to 0.5 mm wide. Copper wire was not found as satisfactory on account of excessive stretching of the wire. After the mica had been notched it was removed from the clamp, and the six holes shown in Figure 2 (b) were drilled. Piano wire was ground to form a flat drill for this purpose. The slots shown were cut and the slotted pieces of mica were pushed together to form a cross in which the teeth were arranged to accommodate a bifilar winding of uniform pitch. This cross was inserted in the quartered steel mandrel illustrated in Figure 2 (c).

Platinum wire nominally 0.1 mm in diameter was used in these thermometers, and the length of wire required was 1.8 to 2.4 m, depending upon the actual diameter of the lot of wire used. The requisite amount of wire was cut into two pieces, each of which was wound into a helix with an outer diameter of about 0.45 mm. For this purpose a steel wire (0.23 mm diameter) was used as a mandrel, one end being mounted in a lathe and the other end attached to a ball bearing, an arrangement which permitted fairly uniform rotation of the taut steel wire. When hard-drawn platinum wire was available the following procedure was successful: One end of the wire was attached to the mandrel and the other end to a thread which led over a pulley to a weight of about 30 g. The pulley was placed in such position relative to the lathe that the consecutive turns of the helix were wound in contact; it was far enough from the lathe (about 3 m) so that the angle of winding remained substantially constant. When the winding was completed both ends of the helix were released and one end was kept from rotating while the lathe was rotated in such direction as would tend to unwind the helix until the other end began to slip on the mandrel. The mandrel was then removed from the lathe, its end freed from any roughness which might scratch the platinum wire and the helix pushed off. The helix was then stretched until its length under no tension was approximately doubled, the result being an almost uniform spacing of the wire.

The method just described was not successful for annealed platinum wire. When the use of such wire was necessary, it was either wound along with a spacing wire so that the turns were in contact and the spacing wire later unwound, or the platinum wire was fed from the moving tool carriage of a thread-cutting lathe. For the latter method a lathe with two heads geared together is desirable since the irregularities in the motion of the ball bearing cause irregular spacing of the wire. After winding, the mandrel was dissolved in hot hydrochloric acid.

Short leads of platinum were welded to form a T on one end of each of the two helices. A small oxy-gas flame was used in the present work, although the arc method described by Sligh might have been used with due precaution to arrange the work so that the electric current would not anneal the helix. It was found that the helices stretched less during winding and that the coil suffered less from accidental distortions if the wire was not annealed until the winding upon the mica cross was completed. Both pairs of leads were then threaded into the mica cross as shown for one pair in Figure 2 (d).

The choice of the direction of winding upon the cross was determined by the direction of winding of the helices which had been wound with a left-hand pitch. Figure 2 (e) and (f) show a magnified view of the edge of the mica with such a helix crossing it. The direction of winding upon the cross corresponding to that at (e); that is, a winding with a right-hand pitch was chosen as the one in which two consecutive turns of the helix would be less likely to straddle the mica. The helices were wound upon the cross under the tension caused by a weight of 2 or 3 g attached to each of their free ends. The clamp of thin sheet copper designed as shown in Figure 2 (g) for both right and left hand windings, was slipped over the coil to hold the winding in

place while the ends of the helices were joined to the platinum tie wire visible in Figure 1, and tied to the mica. After removing the clamp and the quartered mandrel from the coil, the latter was annealed in an electric furnace for two or three hours at 600° to 650° C.

During this annealing the resistance of the 25 ohm thermometers decreased about 0.5 ohm, an amount which had to be allowed for in choosing the initial length of wire.

After the preliminary annealing the resistance of the coil was adjusted by cutting out the proper amount of wire and rewelding. the mandrel and clamp being replaced during the operation. The coil was then again annealed at the same temperature for about five hours.

III. LEADS AND THERMOMETER HEAD

As in the thermometers described by Sligh, mica disks served the double purpose of separating the internal leads and breaking up convection currents in the stem of the thermometer. The mica disks were cut with a punch and die of the type ordinarily used for blanking and perforating sheet metal. An attempt to use Sligh's method of keeping the mica disks normal to the axis of the thermometer by staggering the holes through which the leads were threaded was unsuccessful for the small size of washers used.

Better results were obtained by tapering the wire so that disks with holes which fitted the wire snugly could be used. Although tapering the full length of the leads gave the best appearing result, it was found that with care the mica disks could be slid along the uniform wires without serious enlargement of the holes, and that part or all of the tapered portion could be discarded. Gold wire 0.2 mm diameter was tapered in hot aqua regia by moving the wire up and down thus varying the time of immersion along the length of the wire. The holes in the disks used with these leads were made with drills formed from 0.16-mm steel wire. A pile of the disks was placed in the jig illustrated in Figure 3 (a) and four equally spaced holes were drilled. The insertion of a piece of the wire used for drills in one hole before removal of the disks from the jig made it possible to thread the whole pile of disks onto the ends of the leads as a unit. After the requisite number of disks had been threaded on, the leads were put under tension and each disk slid singly into the desired position, the spacing used being about 5 to 10 mm. A short copper wire was silver soldered or welded to one end of each of the gold leads for convenience in later sealing the thermometer head with soft solder, and one of the short platinum leads on the thermometer coils was welded to the other end.

Since some of these thermometers were to be used in an inverted position and would probably come in contact with oil, it was desirable to avoid the use of cement or wax for sealing. The method of bringing the internal leads out through the Pyrex tube is shown in Figure 3 (b). Four platinum disks 0.02 or 0.03 mm thick were beveled at the edge by running through rolls and curled to fit the glass tube. These were temporarily held in place by a clamp of nichrome wire and fused to the glass as described by Housekeeper.² It was found that the formation of bubbles under the platinum could be minimized by keeping the work far out in the flame. The addition of some oxygen to the air supplied to the torch was also found advantageous for Pyrex. Holes through the platinum and glass were made at the centers of the disks with a hot, tungsten wire. Benton³ used a hot platinum wire for perforating glass, but tungsten has the advantage of being stiffer and forming an oxide which does not readily stick to the glass.

²J. Am. Inst. Elect. Eng., vol. 42, p. 964, 1923.

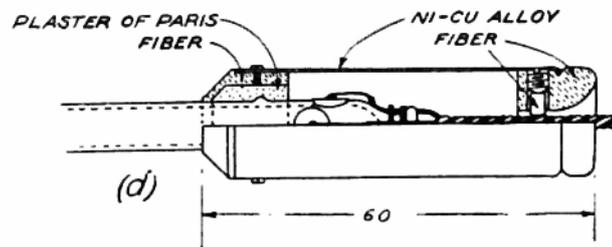
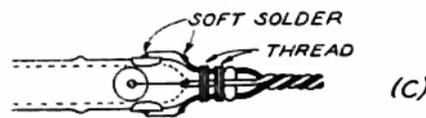
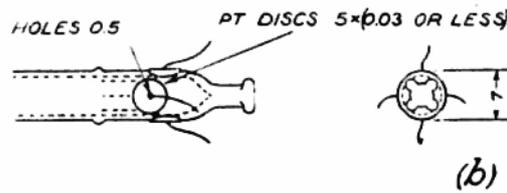
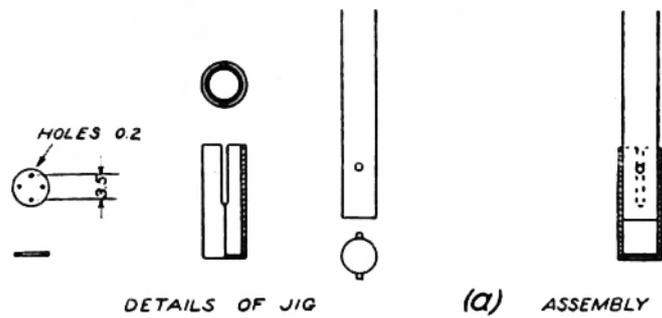


FIGURE 3.—Construction of leads and head

The short copper wires at the end of the gold leads were threaded through the holes, which were then closed with soft solder. Caution must be used in soldering; a hot iron must not be applied to the cold glass lest the platinum be loosened. The external leads were then attached as in Figure 3 (c). The assembled head is shown in Figure. 3 (d).

The thermometers were filled through the bulb end with dry helium at various pressures less than 1 atmosphere and sealed.

IV. CHARACTERISTIC PROPERTIES OF THE THERMOMETERS

As is to be expected, the values for the resistance in ice, steam, and sulphur have the same proportions as for previous thermometers in which platinum of the same purity was used.

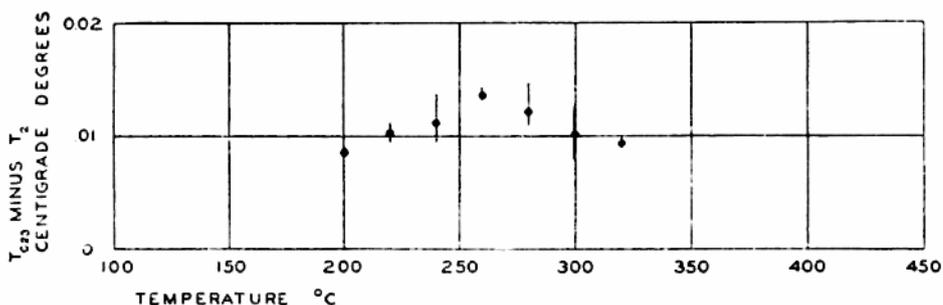


FIGURE 4.—Comparison of coiled-filament thermometer with ordinary platinum resistance thermometer

To determine whether the readings of a coiled-filament thermometer deviated appreciably from those of thermometers previously used, such a thermometer (No. 2) was compared in the range 200 to 320°C. with C₂₃, a thermometer of the customary design as described by Sligh. During this comparison the thermometers were immersed in a stirred oil bath of uniform temperature.

In Figure 4 the difference in centigrade degrees obtained by subtracting the readings of the coiled-filament thermometer from those of C₂₃ are plotted as ordinate with the temperature as abscissa. The differences are no greater than might be expected in this region between two thermometers of the customary design. Each circle represents the mean of the observations at a given temperature while the vertical line through the circle represents the spread of the observations. A curve of differences should by definition pass through zero at 100 and at 444°C, but experimental errors in calibration especially at the latter point, probably introduce considerable differences on the scale chosen in Figure 4, hence no attempt has been made to draw such a curve. It may be concluded that the coiled-filament winding introduces no appreciable peculiarity in the readings of the thermometer.

Since winding the wire in a helix reduces the effective area for heat transfer between the thermometer and its surroundings, it is to be expected that the thermometric lag will be thereby increased as well as the temperature difference between the thermometer coil and its surroundings, necessary to dissipate the heat generated by the measuring electric current.

Measurements of the apparent resistance in an ice bath for several values of measuring current show that this temperature difference is proportional to the square of the current, and for 1.5 ma through a 25 ohm coil 5 mm in diameter and 2 cm long is about 0.004°C when air filled and 0.001°C when helium filled. In the paper by Sligh already mentioned the temperature difference for the larger type of thermometer filled with air is given as 0.001°C for a current of 1.5 ma.

With the use of currents up to 5 ma the ratio between this temperature difference and the power input in the thermometer may be determined with sufficient accuracy so that variations with time in this ratio may be used as a measure of air leakage into the thermometer tube. One thermometer of the type described filled with helium at about 40 mm absolute pressure has shown no appreciable leakage over a period of two years.

³ J. Ind. Eng. Chem., vol. 11, p. 623, 1919

VI. ACKNOWLEDGMENTS

The author wishes to thank H. F. Stimson, F. R. Caldwell, and others for valuable suggestions and assistance in the construction of these thermometers, and E.F. Fiock for the data given in Figure 4.

Washington, September 1, 1932

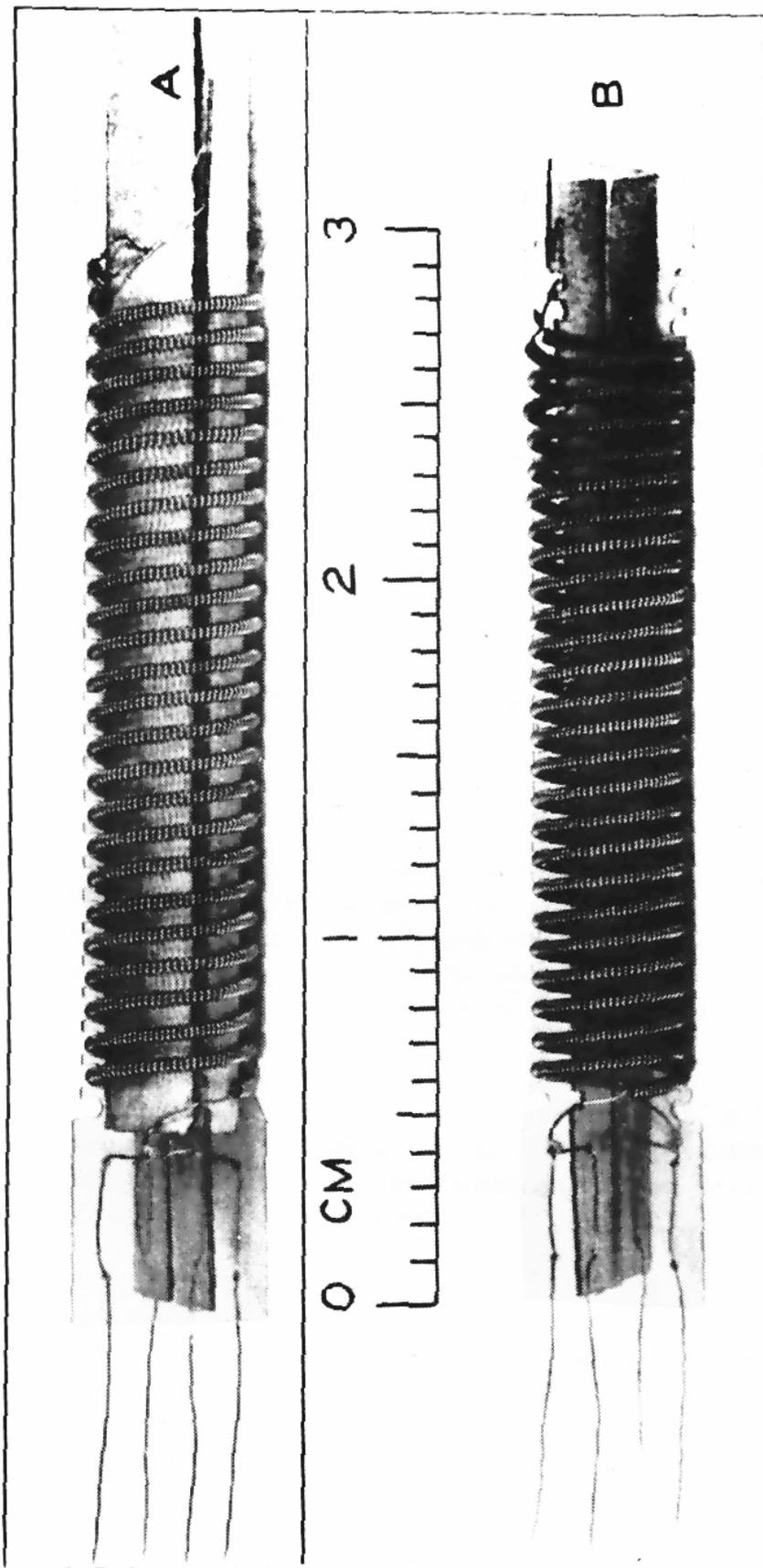


FIGURE 1.—Large model of thermometer coil

A, Mounted upon mandrel; B, removed from mandrel.

OPEN CELLS, SEALED CELLS

SLIM CELLS

by John P. Tavener and Henry E. Sostmann

ABSTRACT

In the early days, when realizations of defining thermometric fixed points were done only in National and University Laboratories, the cells in which the metals were contained were open to the atmosphere, and were protected from oxidation and contamination by informed care. As the requirements for more fundamental calibration capability in-house developed in science and industry, sealed fixed-point cells, containing a proper atmosphere and invulnerable to chance contamination, were developed(1). While sealed cells are unquestionably recommended for the majority of situations, and have distinct advantages, a few users prefer open cells and are willing to go to the expense and effort required to use them properly. Isotech offers both sealed and open cells.

THE CONSTRUCTION OF SEALED CELLS

A sealed cell is shown in Fig. 1. It contains high-purity metal (generally better than 99.9999% pure) within a dense and purified graphite crucible. The entire crucible is surrounded by a fused silica envelope which includes a thermometer well, protected by a graphite sleeve. The well projects into the metal and accommodates the thermometer to be calibrated.

In manufacture, the cell is repeatedly evacuated and washed with a pure inert gas, and finally filled with dry oxygen-free argon at a pressure which will be 1 standard atmosphere at the freezing temperature. The interior of the cell and the metal is thus permanently isolated from the effects of ambient conditions, and from contamination.

THE CONSTRUCTION OF OPEN CELLS

An open cell is shown in Fig. 2. The crucible is not completely encapsulated in quartz, but is slipped into a closed-end quartz tube (like a big test-tube). The top of the quartz tube carries a gasketed cap which (a) locates a guide tube for the thermometer and (b) provides a port for a gas-handling system. Open cells are sometimes closed with a resilient stopper. These are to be avoided; experience indicates that they are eventually degraded by heat (unless watercooled, which adds one more mechanical and thermal complexity), may stick to the quartz, may become an inadequate seal; may shed particles into the cell.

THE ADVANTAGES AND DISADVANTAGES OF SEALED AND OPEN CELLS

There is some risk of breakage when a sealed cell is shipped by common carrier, although it has been done successfully, and a new design suggests that, after additional testing and experience, shipment may become commonplace. Now, it is better to hand-carry cells from the factory in England or from stock in New York. (Isotech makes an allowance for travel to the factory, and a visit provides an opportunity for some training).

Open cells may be shipped by common carrier, disassembled, as a set of parts packed in contamination barriers. The user is expected to make the final assembly

while preserving the purity of the cell. In use, sealed cells are no more fragile than open cells.

Sealed cells are protected against contamination from all external influences, unless the quartz is broken; a condition which is easily recognizable. Metallic contaminants cannot enter the sealed system. The inert gas which is sealed into the system effectively prevents oxidation or the effects of oxygen, which alters the equilibrium temperature for some metals.

The equilibrium temperature of thermometric metals is also slightly dependant upon pressure. (For example, the freezing point of tin is shifted 3.3 mK per atmosphere; aluminum 7.0 mK). Although the correct pressure is built into a sealed cell, it cannot be directly verified, nor can one know by a pressure measurement if it has changed. But we have used sets of sealed cells for as much as 20 years and have not detected a change in equilibrium temperature; an indirect verification of correct pressure, in the terms which really matter.

Open cells must be used with a gas-handling system which provides a purgative and protective gas blanket in the cell whenever the cell is above ambient temperature, and at 1 atmosphere of pressure at the freeze equilibrium. Such a gas system (after Furukawa) is shown in Fig. 3⁽²⁾. Not all elements of this system may be required for all met-als. It is evident from the figure that to use such a system each time a calibration is performed requires careful adherence to an operating protocol. NBS 260-77 endorses sealed cells especially for aluminum.

While the internal pressure of open cells may be directly measured, they are unprotected against contamination from materials which enter the cell through its upper closure or through its gas handling system. Thus the purity of the cell must be verified, either by methods described in the literature (e.g.(3)), by comparison with another cell, or by a periodic check at a National Laboratory.

ACCEPTANCE OF SEALED CELLS BY AUTHORITIES

Isotech sealed cells are to be found among the National Standards of temperature of Germany, Holland, Egypt, Hong Kong, the Republic of China, the People's Republic of China, Korea, Portugal, Spain, Saudi Arabia, Finland, Sweden, Norway, Poland, Brazil, Switzerland, and other countries. They are used in defense, energy and industrial laboratories such as the U. S. Navy Primary Standards Laboratories East and West, Newark Air Force Base, Sandia National Laboratory, Lockheed, Boeing, and many others. In all but exceptional circumstances sealed cells are the accepted and appropriate basis for a primary thermometer calibration competence.

AND WHAT ARE SLIM CELLS?

The special requirements of immersion depth, plateau duration, etc. required for the calibration of SPRTs may not be necessary in laboratories charged with calibrating industrial resistance thermometers, thermocouples and thermistors, but mobility and cost may be more important. "Slim cells" is a name given to another category of cell, which are somewhat slimmer, slightly shorter, and lower in price. Slim cells are built using the same materials, techniques and purity of metal as the larger cells, but the uncertainties associated with them are somewhat larger, not because of the cells but precisely because their properties cannot be measured with SPRTs, and transfer thermometers must be employed in qualifying them.

In consequence of their smaller size, smaller, lighter-weight apparatus (bench-top or cart-mounted furnaces) may be used to melt and freeze the metal in these cells. Both sealed and open cells, and associated apparatus, are available from Isotech.

1. H. E. Sostmann, *A sealed metal freezing point cell for thermometer calibration*, Rev. Sci. Instr. 43, 5, pp. 833-834 (1972). This paper is believed to be the first publication on sealed cells, and is reproduced overleaf.
2. G. T. Furukawa, Application of some metal SRMs as thermometric fixed points, NBS Special Publication No. 260-77 (1982)
3. J. P. Tavener, *Confidence in the freezing point a of high-purity metals*, Isotech Journal of Thermometry, Vol 1 No 2 (1990) pp 53 et seq.

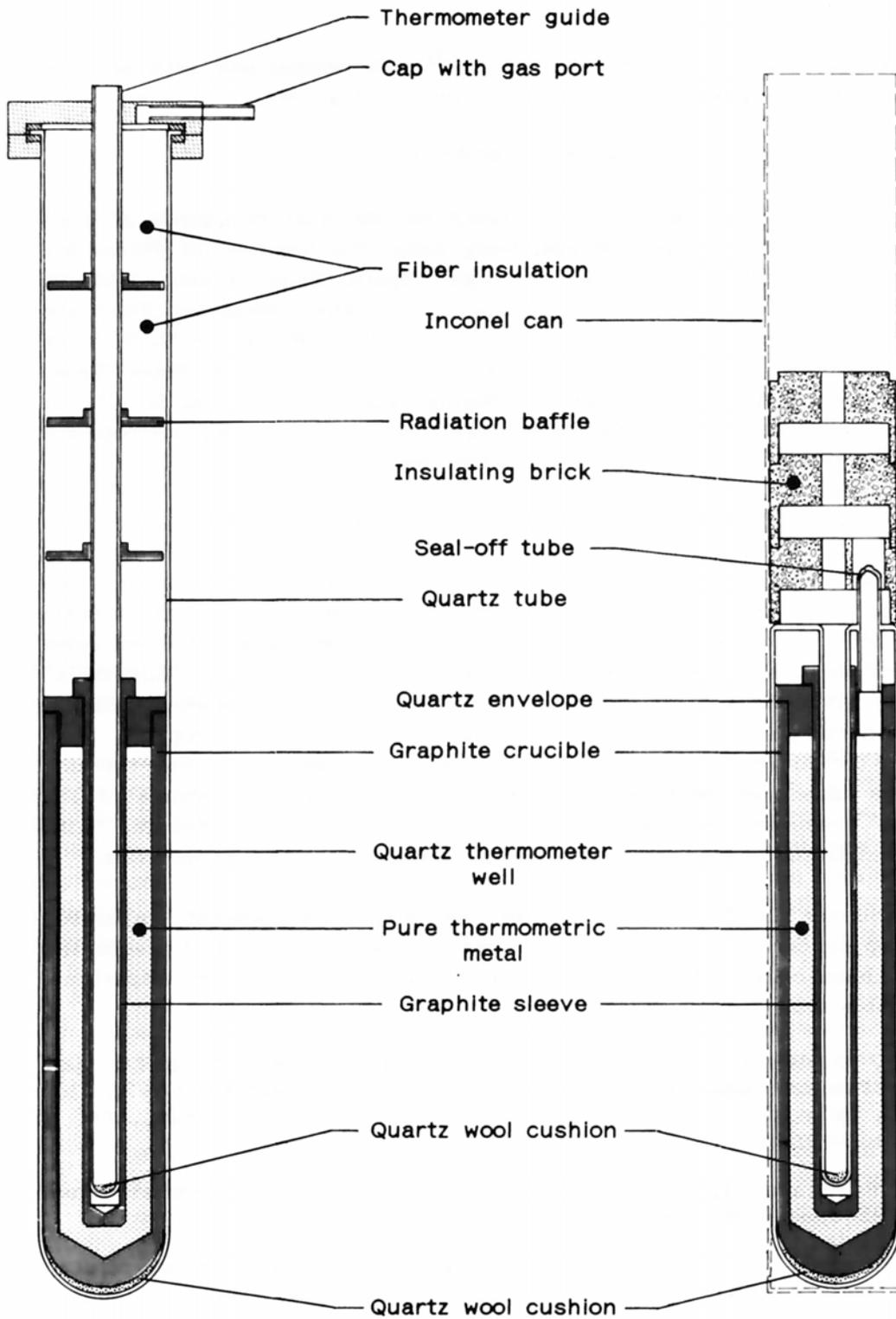


FIG. 2

FIG. 1

OPEN AND SEALED (PURE METAL) FIXED POINT CELLS

A Sealed Metal Freezing Point Cell for Thermometer Calibration

HENRY E. SOSTMAN

H. E. Sostman & Co., Union, New Jersey 07083

(Received 7 January 1972; and in final form, 18 February 1972)

Metal freezing point cells for realizing defining points of the International Practical Temperature Scale in thermometer calibration must be kept free from contamination. Design and construction are described for a sealed cell in which the danger of contamination is virtually eliminated.

Continually increasing requirements for accuracy and stability of the large number of small, rugged, and stable platinum resistance thermometers employed in industrial process measurement and control argue that the manufacturer of these thermometers be prepared to calibrate working standards and prototypes to the fixed points of the International Practical Temperature Scale.¹ These fixed points are temperatures defined by materials of high purity in two- or three-phase equilibrium.

In our laboratory solid-liquid equilibria are preferred to

liquid-vapor equilibria because of the pressure dependence of the latter. Calibrations are made at the triple point of water (0.01°C) and the liquid-solid equilibria (the "freezing points") of tin (231.9681°C), lead (327.502°C), zinc (419.58°C) and aluminum (660.37°C).¹ Impurities in each metal are less than 10 ppm (for aluminum less than 50 ppm) and are spectroanalytically known.

Cells for freezing high purity metals have been described extensively by McLaren and others.² Typically the metal is melted in a crucible of high purity graphite in a furnace

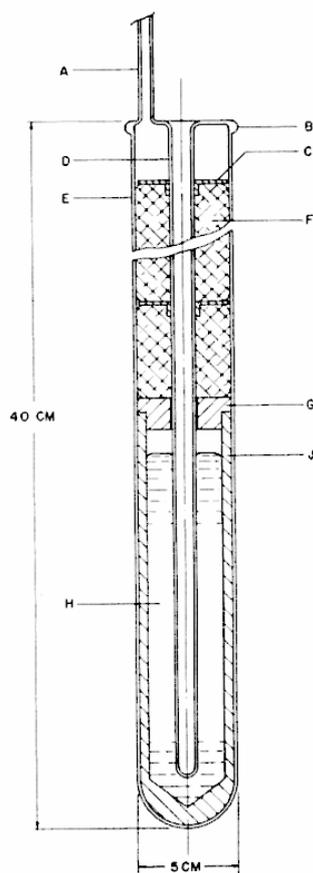


FIG. 1. A—Seal tubulature; B—lip permitting removal of the cell from the furnace with a lift ring (not shown); C—graphite radiation baffle; D—inner well; E—outer tube; F—convection baffling (washed Fiberglass); G—graphite crucible cover; H—high purity metal; J—graphite crucible.

Contamination of the metal in the cell by reaction or by the introduction of foreign matter must be avoided. In the industrial laboratory calibrations are sometimes made by workers who are not scientists, or by various people at various times, and the need for caution to prevent contamination becomes fundamental.

We have designed cells in which contamination in use is virtually impossible unless the cell is physically broken.

Figure 1 shows the cross section of such a cell. At assembly the inner well and outer tube are separate pieces and the walls are straight. The metal is melted into the crucible under argon and cooled to room temperature. The baffling is added and the glasswork is then completed by sealing over the outer tube to the inner well and adding a fill tube, which may be a length of tubing, a graded seal, or a glass stopcock. The cell is successively evacuated and washed with argon several times, and finally sealed off containing argon at 1 atm pressure at the freezing temperature of the metal.

Pyrex glass is used for the tin and lead cells, and Vycor for the zinc and aluminum cells. A graphite protective well around the inner glass well has been found advisable for the zinc and aluminum cells.

A comment has been made that particles of the material of the convection baffling might conceivably fall into the metal and provide points for early nucleation.⁴ No such difficulty has been recognized, although it would be more likely if the material were Fiberglass. However the next generation of cells will probably be designed so that the seal is immediately above the crucible cover, and the baffling external to the seal.

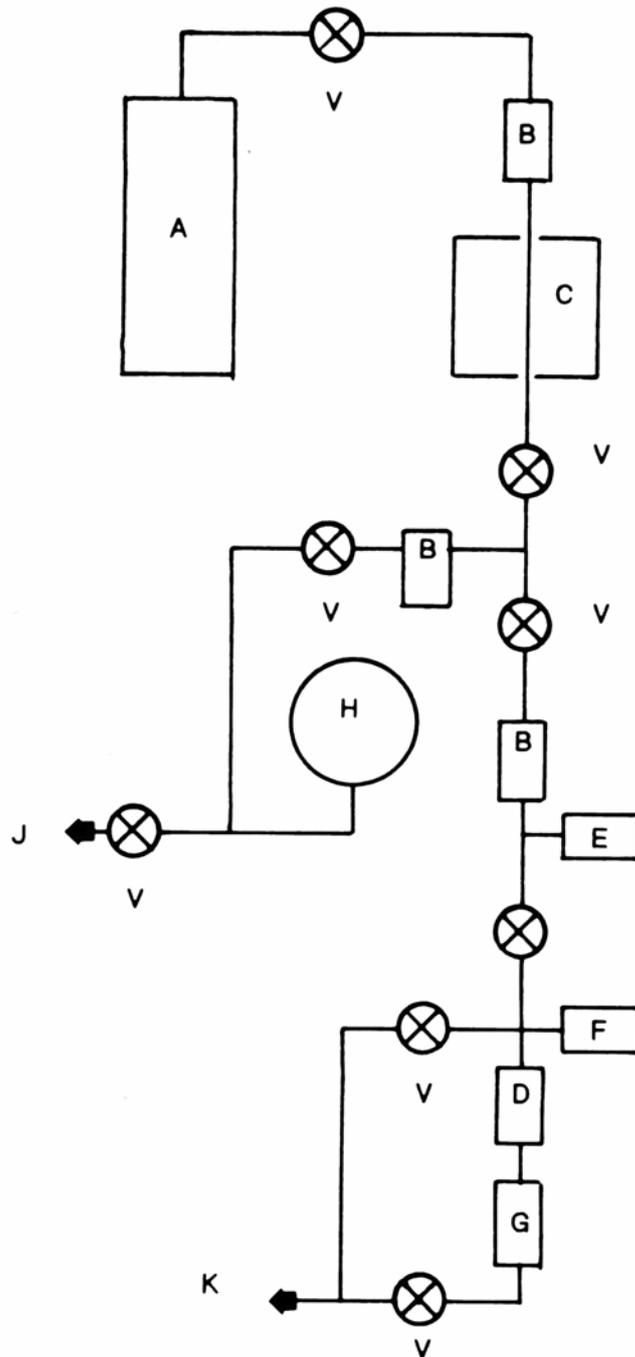
with a massive block, permitting a very slow temperature drop during freezing. Although it has been suggested that it may not be necessary,³ it is a common precaution to blank with argon or helium whenever the metal is above room temperature.

¹ *Metrologia* 5, 35 (1969).

² For example, E. H. McLaren, in *Temperature, its Measurement and Control in Science and Industry* (Reinhold, New York, 1963), Vol. 3, Pt. 2, pp. 185f.

³ H. M. Terwilliger, ISA Report No. 12.11-1-66.

⁴ John P. Evans (private communication).



A = 99.999% pure argon gas in cylinder; B = liquid oxygen cold traps; C = inconel tube furnace packed with copper metal and copper oxide; D = liquid nitrogen cold trap; E = cold-cathode vacuum gage; F = thermocouple vacuum gage; G = oil diffusion pump; H = dial manometer; J = flexible inconel tubing to cell; K = mechanical vacuum pump; V = bellows-type (packless) valve.

THE HEAT PIPE AND ITS USE IN THERMOMETER CALIBRATION

by Henry E. Sostmann

A heat pipe, as its name implies, is a device for transferring heat from one location to another.

Assume a closed shell, a cylinder with the axis vertical. At the lower end is a small quantity of water, which is heated to the point of vaporization. The vapor fills the shell's inner space, rising to the upper, or cold, end, where it is condensed. The condensate is returned by gravity to the lower end where it is again vaporized. Since the latent heat of vaporization is very large, large quantities of heat can be transported.

The system I have just described is called a two-phase thermal syphon, and has been known and used for many years. A deficiency is that the requirement for gravity return requires a specific orientation of the pipe, and is not efficient. This problem is solved by providing a means other than gravity (although it may be assisted by gravity) for returning the condensed liquid to the hot end. While in special designs this means may be centrifugal force, osmosis, etc., the common means is to provide capillary force by including a wick; thus the thermal syphon becomes the modern heat pipe, shown in Bienert's Fig. 1. Since heat is discharged at the condensing surface as a phase change, where vapor and liquid phases are present simultaneously, an essentially isothermal vertical profile is obtained (assuming that the vapor space is isobaric). The departure of the temperature profile from isothermality is almost unmeasurable.

Isothermal Technology uses a heat pipe manufactured by Bienert's Company, Dynatherm, in its high-temperature furnace for realizing the freezing points of high-purity aluminum and silver. The furnace range of use is 500 ° to 1000 °C. The heat pipe is 18 inches long and disposed vertically. The bore diameter is 2 inches.

In addition to providing an isothermal zone for melting and refreezing fixed points, heat pipe techniques lend themselves well to other furnace designs for control and calibration at arbitrary intermediate temperatures. Since the actual temperature is a function of the internal pressure, the temperature can be varied smoothly and continuously by varying the pressure of the vapor. In the paper which follows, Bienert describes a commercial apparatus for the calibration of thermometers over a wide range, which uses the principle of a pressure-controlled heat pipe to realize a comparison calibration furnace of great utility and flexibility.

ISOTHERMAL HEAT PIPES AND PRESSURE CONTROLLED FURNACES

by Walter W. Bienert, Dynatherm Corporation

1. OPERATION OF HEAT PIPES

The principle of a heat pipe is shown in Figure 1. The hermetically sealed envelope, often a tube or a pipe, is evacuated and then partially filled with a suitable fluid. In the absence of heat sources or heat sinks, the liquid and vapor phases of the fluid are in thermal equilibrium and the internal pressure is a function of the temperature only. When heat is applied to any section of the heat pipe (evaporator), vaporization occurs locally, raising the pressure in the vapor phase. The increased pressure causes a flow of vapor to the unheated, cooler sections where the excess vapor condenses (condenser). The cycle is completed by returning the condensate to the evaporator. Most heat pipes utilize capillary pumping by a wick to return the condensate and to compensate for the hydrodynamic flow losses in the liquid and the vapor. In some applications where the heat pipe is favorably oriented, gravity can be substituted for the capillary pump. Heat is transported along the heat pipe in the form of latent heat. Since the hydrodynamic losses in the vapor are usually very small, the pressure, and therefore the temperature, are nearly constant throughout the entire heat pipe. It is this temperature uniformity which makes heat pipes attractive as isothermal enclosures for thermometric applications.

Heat pipes have been built for operation in many temperature regimes, ranging from cryogenic (20 K) to very high temperatures (up to 2300 K). An optimum temperature range exists for each working fluid. Obvious theoretical limits for all fluids are the melting point at the low end and the critical point at the high end. A more practical lower limit is the transition from molecular to continuous flow which typically occurs at vapor pressures of a few Torr; a practical upper limit occurs when the vapor pressure becomes excessive for containing the fluid safely inside the heat pipe. Figure 3 shows the approximate temperature ranges for some working fluids that are of interest in the range from 200 to 1,500°C. The vertical axis in this figure is labeled "Liquid Transport Factor"; it is a Figure of Merit by which fluids can be ranked. A more extensive discussion of the principles of heat pipes can be found in References 1 through 4. The most frequently used fluids in heat pipes designed for thermometry applications are the alkali metals cesium, potassium, sodium, and lithium.

2. HEAT PIPES AS ISOTHERMAL ENCLOSURES

Most heat pipes are designed to transport a maximum amount of heat with the smallest possible temperature loss. However, in thermometry applications, heat transport capability is usually less important than temperature uniformity. The achievable temperature uniformity is directly related to the uniformity of the vapor pressure inside the heat pipe, and is illustrated by the following example: Consider a typical sodium heat pipe with an internal diameter of 2.0 cm (3/4") and a length of 100 cm (40") operating near the boiling point of sodium (1156 K) with a heat load of 1,000 Watts. Under these conditions, the axial pressure gradient in the vapor due to viscous flow losses is approximately 5.5 Pa; using the Clausius-Clapeyron relationship between vapor pressure and temperature, a corresponding axial temperature gradient of 0.006 K can be calculated.

Unfortunately, this small temperature gradient applies only to the heat pipe's vapor space. The temperature distribution on the outside of the pipe, which is of more interest to the user, is influenced by many factors, the most important of them being the local heat flux into and out of the pipe. When heat is applied to or removed from the pipe, it must be conducted through the wall and the wick. The associated ΔT is related to the radial heat flux q by the approximation:

$$q = \left[\frac{k_w}{t_w} \right] \times \Delta T \quad \text{Eq.1}$$

k_w and t_w , are the thermal conductivity of the wall and its thickness, respectively. Equation 1 neglects the ΔT through the wick, which is justifiable for most liquid metal heat pipes. For a typical heat pipe fabricated from Inconel and with a wall thickness of 0.3 cm (1/8") the above relationship reduces to:

$$\Delta T \approx 1 \times q \quad \text{Eq.2}$$

where q is measured in Watt/cm² and ΔT in Kelvins. Referring again to the previous example, assume that the 1,000 Watt heat load is inputted uniformly over one half of the pipe's length (50 cm) and removed from the other half. The resulting radial heat flux is 3.2 Watt/cm² and the wall ΔT becomes 3.2 K. Since the same T will occur at the condenser, the overall ΔT along the heat pipe will be 6.4 K which is 1000 times higher than the internal ΔT in the vapor.

The above example emphasizes the importance of minimizing radial heat fluxes in areas where temperature uniformity is of importance. The working space of heat pipes designed for thermometry applications is usually configured as a cylindrical space surrounded by the heat pipe's vapor; a cavity, or a deep and narrow well. This reduces heat fluxes to the milliwatt/cm² level and the corresponding temperature non-uniformities to the order of a few millidegrees.

Dynatherm's Isothermal Furnace Liners (IFL) are heat pipes designed to minimize radial heat fluxes. Figure 2 shows a few typical configurations. The annular heat pipe in 3a consists of two coaxial pipes in which the wick and the working fluid are located in the annulus between the outer and inner pipe. The heat pipe is normally installed in a cylindrical furnace: thus heat is applied to the outer cylinder. Likewise, heat losses (condensation) occur mostly from the outer cylinder near the end of the furnace. When properly insulated, the inner, cylindrical working space is therefore a region of very low heat flux and good temperature uniformity. The design shown in 3b is a variation of the previous one: when access from both ends is not required, closing one end and surrounding it by the heat pipe further reduces radial heat fluxes and improves temperature uniformity. Figure 3c shows a configuration which is particularly suitable for calibrating radiation sensors. The working space is a cavity with a small aperture which is essentially an isothermal blackbody radiator. Configuration 3d is designed for calibrating thermocouples and resistance thermometers. The small diameters of the individual "wells" reduce heat losses through their openings to an extremely small value. Furthermore, since all wells are surrounded by the nearly isothermal vapor of the heat pipe, temperature uniformity between wells is also guaranteed.

Calculated wall temperature profiles are shown in Figure 4 for a standard IFL which is open on both ends (see Figure 3a) and in Figure 5 for a small diameter thermowell. The calculations assume in each case that the vapor temperature is perfectly uniform at the boiling point of sodium, that all heat losses are by radiation, and that the heat pipes are constructed from Inconel using standard wall thicknesses. The

curve in 4 is symmetrical; the wall temperature is isothermal within 0.1 K over 25 cm (10") and within 1.0 K over 45 cm (18"). In the case of a thermowell, the wall is isothermal within fractions of a millidegree over most of its length and only drops off near the entrance where heat losses become noticeable. Since the open ended IFL exhibits only a modest isothermality (order of 0.1 degree), temperature gradients in the vapor are relatively unimportant. In the case of the thermowell, vapor gradients must also be minimized in order to realize the potential of near perfect isothermality.

Figs. 6 through 8 show measured and calculated temperature profiles in an open-ended IFL such as that shown in Fig. 3a. The heat pipe has an internal diameter of 7.8 cm (3.07") and is 61 cm (24") long. Unlike the previous curves which showed calculated wall temperatures, these Figures show the profile seen by a probe suspended in the center of the working space. Since the probe is coupled by radiation only to the inner walls of the heat pipe, as well as to the ends, the profile is less flat than that of the wall. The last four Figures emphasize the importance of minimizing heat losses; when the ends of the IFL are well insulated (Fig. 6) the temperature is uniform within the 25 mK sensitivity of the measuring instrument. With reduced insulation, or none, the profiles are distinct. The measured profiles are in good agreement with the theoretical prediction, which is based on radiative heat transfer only.

3. THE PRESSURE-CONTROLLED HEAT PIPE

The basic heat pipe I have just described is an entirely passive heat transfer device. The temperature is determined completely by the heat input, the temperature of the heat sink, and the thermal resistances between heat pipe and heat sink. Early in the development of this technology the unique properties of the gas-loaded heat pipe were recognized. While a basic heat pipe contains only a single-component working fluid, the gas-loaded heat pipe, as its name implies, includes also a small quantity of a non-condensable (usually noble) gas. A simplified view of a gas-loaded heat pipe is shown in Fig. 9. The non-condensable gas is swept toward the condenser by the vapor; since it cannot condense, the gas forms a slug which blocks part of the condenser. Thus heat rejection is impeded, and the overall conductance of the heat pipe is reduced.

At first glance, this would appear to be undesirable. However, it turns out that the gas blockage provides a means for controlling the heat pipe's temperature. The total pressure is, of course, the same in the "active region" in which only vapor is present, and in the "inactive" or "blocked" region. If the amount of gas is fixed, the length of the gas slug, and with it the heat pipe's conductance, is a function of the internal pressure. If the heat pipe temperature increases, so does the internal pressure, which compresses the gas slug. A shorter gas slug means increased heat rejection capacity, which counteracts the original temperature rise. Thus a gas-loaded heat pipe is self-compensating; it adjusts its conductance in accordance with the heat load, and achieves a certain degree of temperature stabilization. (Gas loaded heat pipes are frequently referred to as variable-conductance heat pipes (VCHPs). References 5 and 6 provide a detailed discussion of their theory and operation. VCHPs are frequently used in the thermal control of spacecraft, where the temperature of sensitive components needs to be maintained within narrow limits.

The equality of pressures in the vapor zone and in the gas-loaded zone implies that, by controlling the gas pressure, the vapor pressure and consequently the heat pipe temperature can be controlled. The recognition of this fact led to the development of the pressure-controlled heat pipe (PCHP).

Fig. 10 shows the principle. Unlike the conventional heat pipe, which is hermetically sealed, the PCHP is open-ended, with the condenser connected to a large gas buffer

volume. The body of the heat pipe is located inside a furnace, and is well insulated. The condenser is located outside the furnace, and is equipped with a cooling coil. The gas pressure in the buffer volume is adjusted to be such that the interface between vapor and buffering gas is located inside the condenser. (Even without further control of the gas pressure, this device exhibits a very stable temperature). Changes of heater power or changes of the thermal load on the furnace will shift the location of the interface to compensate for varying heat-rejection requirements. However, since the volume of gas in the buffer is many times larger than the amount inside the condenser, the gas pressure is affected very little by changes in the location of the interface. Nearly constant gas pressure is equivalent to nearly constant temperature. (1)

Active control of gas pressure improves the achievable temperature stability with very high precision. Referring again to Fig. 10, active control involves monitoring the gas pressure in the buffer volume with a very sensitive manometer, and making required corrections by adding or removing gas through metering valves. The achievable temperature stability of the PCHP was first analyzed by Busse (Ref. 7). For most heat pipe working fluids of interest for the PCHP, the Clausius-Clapeyron relationship between vapor pressure and temperature is approximated by:

$$\frac{\Delta T}{T} = 0,1 \times \frac{\Delta p_v}{p_v} \quad \text{Eq. 3}$$

For example, if the absolute pressure is controlled within 10 parts per million, the temperature is stable to 1 ppm. At an absolute temperature of 1000 K, this converts to a temperature stability of 1 mK. Busse and Bassani (Ref. 8) analyzed the effects of several parameters on the temperature uniformity and stability practicably achievable. The most important were pressure gradients in the heat pipe, impurities in the working fluid, solubility of the non-condensable gas, and, to a lesser extent, gravity.

The effects of pressure gradients due to viscous and dynamic pressure losses have been discussed earlier. They can be minimized by proper design of the heat pipe and the furnace; e.g., by reducing the axial heat flow.

Impurities in the working fluid affect the equilibrium vapor pressure. They can also result in temperature gradients if they become concentrated in some area, such as the evaporator section.

The effect of dissolved buffer gas is similar to that of impurities. Ideally, the working fluid and the buffer gas are sharply separated at the vapor/gas interface. In actuality, the interface consists of a diffusion zone of finite length. A small amount of buffer gas will dissolve in the condensate and be carried to the evaporator by the liquid. It is returned to the condenser by the vapor, and thus alters its partial pressure slightly.

Gravity also causes slight pressure gradients in the vapor. These can normally be neglected, because the vertical dimensions of most heat pipes are too small for the effect to be measurable. For an in-depth discussion of achievable uniformity and stability, please see References 7 through 10.

1: (In an experimental gas-buffered heat pipe, the late John MacAllan at CSIRO used as his buffer volume an aluminum beer keg buried beneath his laboratory's floor, at the nearly constant earth temperature - HES)

4: DYNATHERM'S COMMERCIAL PRESSURE CONTROLLED HEAT PIPE CALIBRATION FURNACE

The principles just discussed have now been embodied in a commercial pressure-controlled heat pipe calibration system. It includes two separate heat pipe furnaces, one having a cesium and the other a sodium heat pipe. This permits the range of 300 ° to 1100 ° C to be covered, with about 200 ° C overlap between the two ranges, controlled by one control system. The design and dimensions of both heat pipes are shown in Fig. 11. The body of each heat pipe contains a cavity whose opening can be changed by means of screw-in apertures. Four thermowells for the calibration of thermometers are provided. The water-cooled condenser is oriented at 45 ° with respect to the heat pipe, so that the heat pipes can be operated horizontally or vertically with the condenser always in the "reflux" mode. (Please note that this heat pipe design was chosen to meet the requirements of one particular user (NIST); the system is easily adaptable to other heat pipe geometries). The cesium pipe is constructed from 316 stainless steel and the sodium pipe from Inconel. The control gas is helium. The heat pipes are installed in commercial furnaces, modified to accommodate the condensers.

The control system is based on the principle shown in Fig. 10. A diagram showing the major components is shown in Fig. 12. Gas control and furnace heater control is accomplished by a PC, which is also used for data acquisition. There are three pressure transducers, having ranges of 100, 1000 and 5000 Torr respectively. For a given temperature setpoint the appropriate transducer is selected by the software. The algorithm also protects the transducers from over-range. Pressure adjustments are made through proportional metering valves driven by stepper motors. Each furnace has three separate heating zones, each zone controlled by a temperature controller and SCR driver. There is a sensitivity to heat input distribution due to vapor flow dynamics and radiative heat exchange between the hot surfaces and the cavity and wells, thus it is useful to be able to fine-tune the setpoints of the three controllers.

An important function of the automatic control is the handling of setpoint changes. Simply adjusting the gas pressure to a new setpoint could result in spilling the working fluid into the control system. The control algorithm, by temporarily isolating the furnace from the gas control system, assures that the proper pressure balance is maintained during all transients.

A photograph of the PCHP system developed for NIST is shown in Fig. 13. Extensive testing and characterization of the system will be done at NIST, using instrumentation not available at Dynatherm. A system using only a single sodium furnace, but otherwise similar, was tested at NRC in Canada (Ref. 11). After fine-tuning the input to the three heater zones, temperature uniformity of 4 mK was achieved over an 11 cm length within a thermowell.

Preliminary tests of the stability and uniformity of the NIST furnaces were conducted at Dynatherm. Fig. 14 shows the predicted temperature stability to be better than ± 10 mK over the entire range from 378° to 1000° C, calculated, using Eq. 3, from the measured pressure stability. Three data points were taken with the cesium furnace and four with the sodium furnace, with about 200 °C overlap.

A limited number of measurements of the absolute temperature were also made. Figs. 15 through 18 show the measured stability of the cesium furnace at 378° and at 507° C, and of the sodium furnace at 613 ° and at 714° C. During a typical test period of 1.5 hours, temperature fluctuated within a ± 10 mK band; slightly higher than inferred from the pressure stability (Fig. 14). Part of the apparent instability may have been due to the temperature instrumentation; we used a commercial, general purpose 100 SZ RTD

(Thermo Electric) and a readout device with a resolution of only 10 mK (Omega DP14RTD).

Figs. 19 and 20 show the measured profiles in one of the thermowells. The cesium profile at 378° C is uniform within ± 10 mK and the sodium profile at 817° C within ± 35 mK.

Of most significance is the relative temperature uniformity measured between the several thermowells, and between the thermowells and the cavity. These are given here:

RELATIVE TEMPERATURE UNIFORMITY

FLUID	PRESS [Torr]	WELL 1 [°C]	WELL 2 [°C]	WELL 3 [°C]	WELL 4 [°C]	CAVITY [°C]	MAX DELT
Cs	7,7	367,10	367,09	367,12	-	-	0,03
Na	25,0	613,39	613,39	613,38	-	-	0,01
Na	106,0	712,00	712,00	-	712,06	712,03	0,06

The same comments apply relative to the probable inadequacies of test instrumentation.

5. CONCLUSIONS

Annular heat pipes such as Dynatherm's Isothermal Furnace Liners can provide isothermal environments with temperature uniformity in the millidegree range. In order to achieve optimum performance, it is necessary to minimize heat losses and internal pressure gradients. When combined with active pressure control, long-term stability better than ± 10 mK is possible. This capability is beyond the sensitivity and accuracy of commercial industrial instrumentation. The paper describes a complete pressure-controlled furnace system which covers the temperature range from 350° C to 1100 C continuously.

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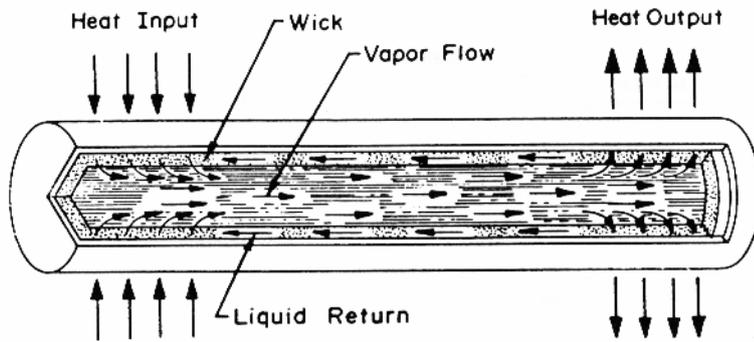
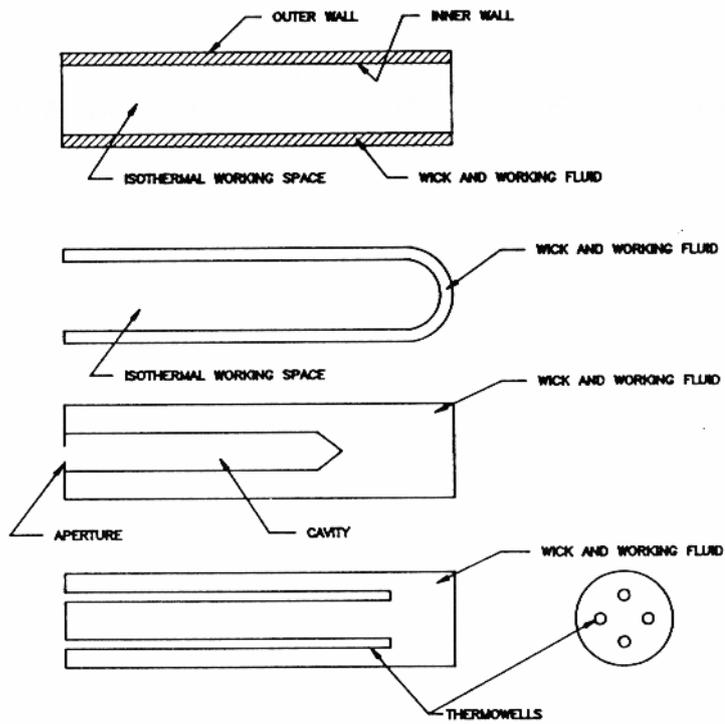


FIGURE 1
SCHEMATIC REPRESENTATION OF A HEAT PIPE



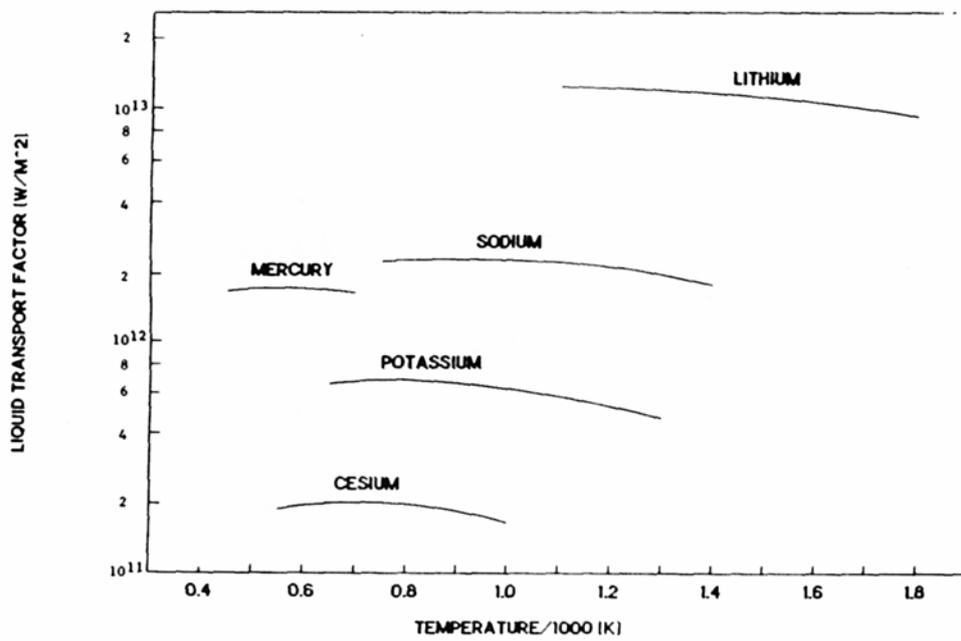


FIGURE 3
LIQUID TRANSPORT FACTOR OF MERCURY AND ALKALAI METALS

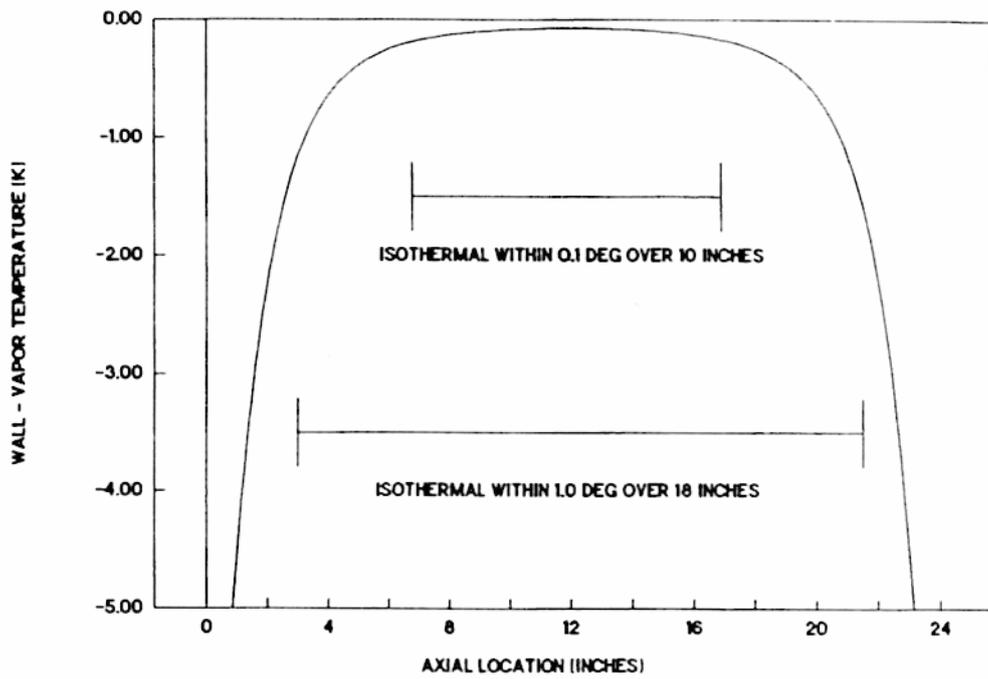


FIGURE 4
 WALL PROFILE IN OPEN-ENDED ISOTHERMAL FURNACE LINER
 Length 24 inches, diameter 3.1 inches

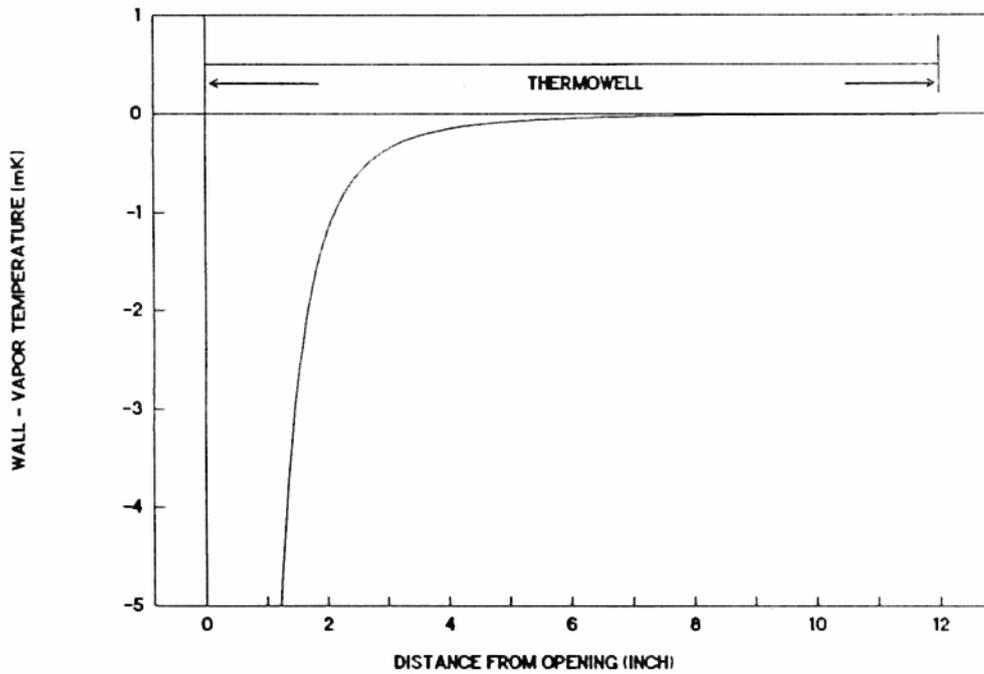


FIGURE 5
 WALL TEMPERATURE IN THERMOWELL
 Well 12 inches deep, 0.313 inches in diameter

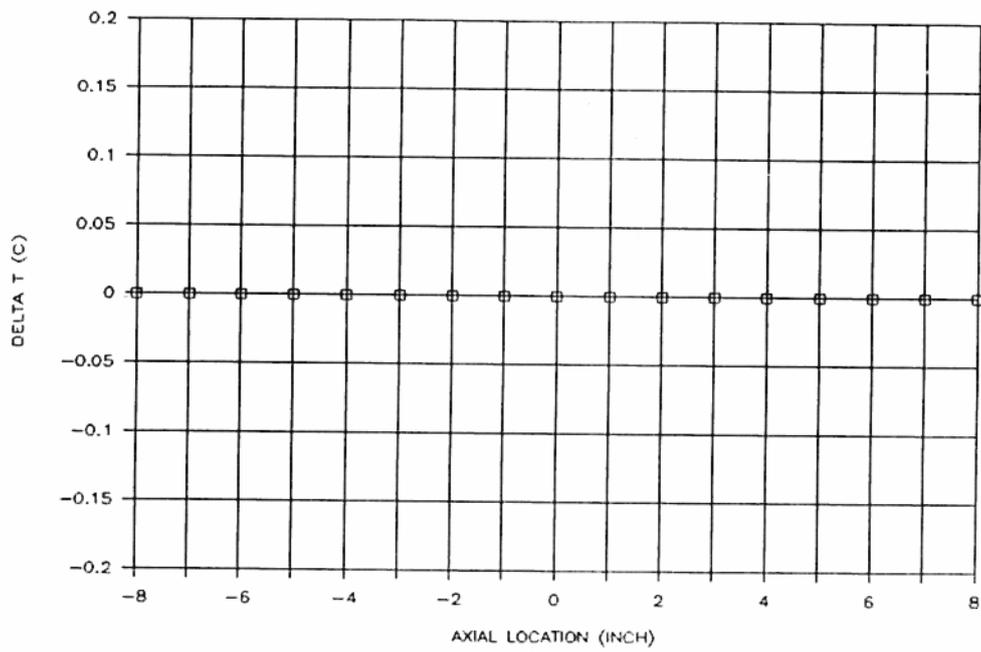


FIGURE 6
ISOTHERMAL FURNACE LINER PROFILE
2 radiation shields, 3 inch insulation

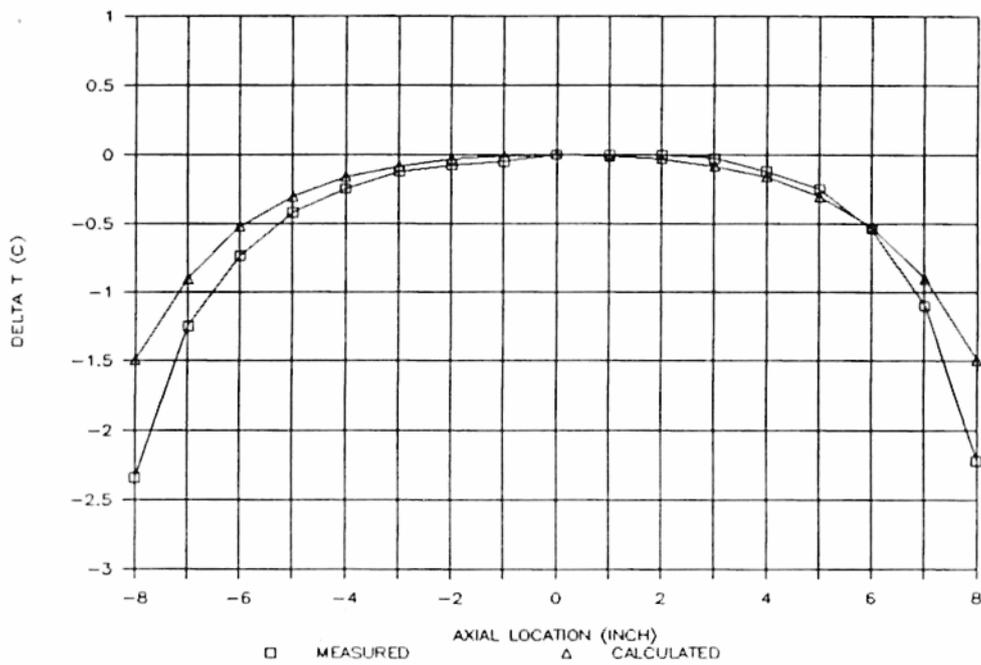


FIGURE 7
ISOTHERMAL FURNACE LINER
2 radiation shields, no insulation

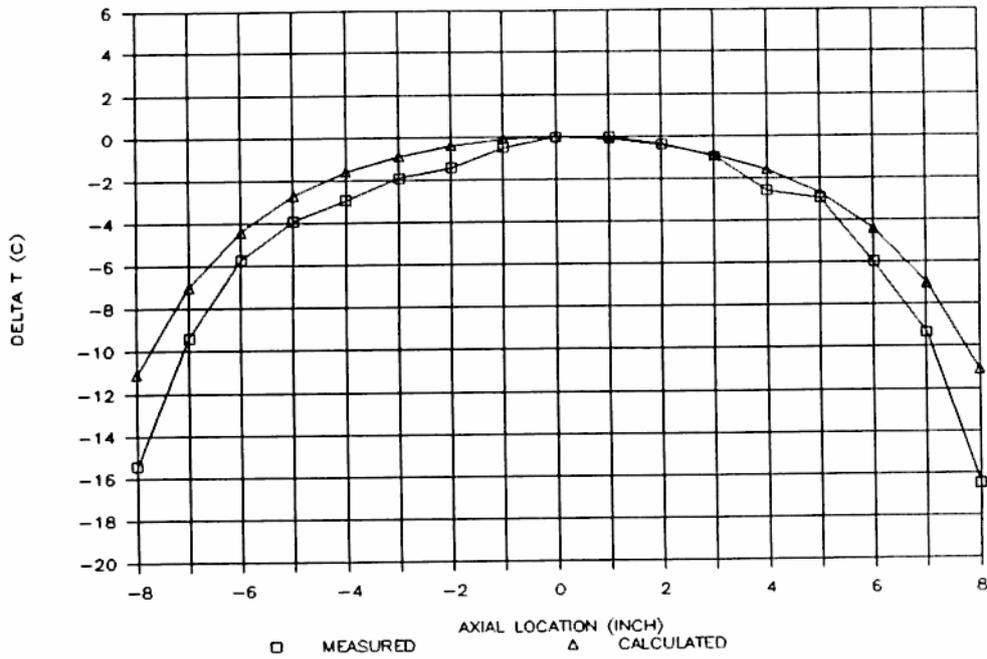


FIGURE 8
ISOTHERMAL FURNACE LINER PROFILE
Quartz tube, no insulation

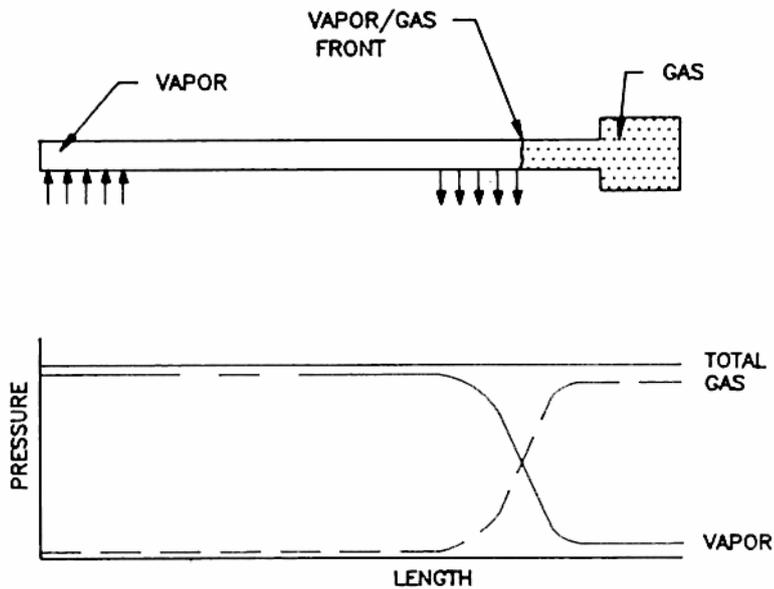


FIGURE 9
PRINCIPLE OF GAS-LOADED HEAT PIPE

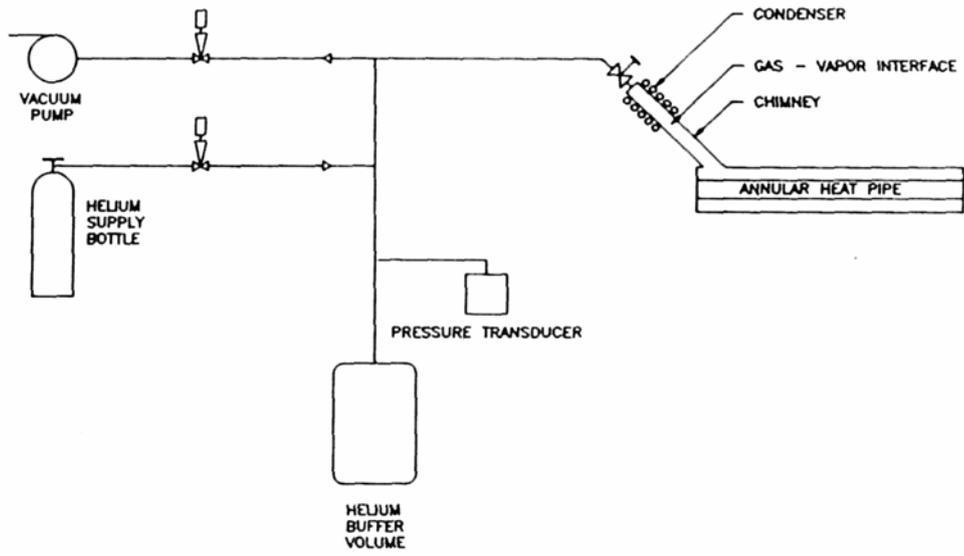


FIGURE 10
PRINCIPLE OF PRESSURE-CONTROLLED HEAT PIPE FURNACE

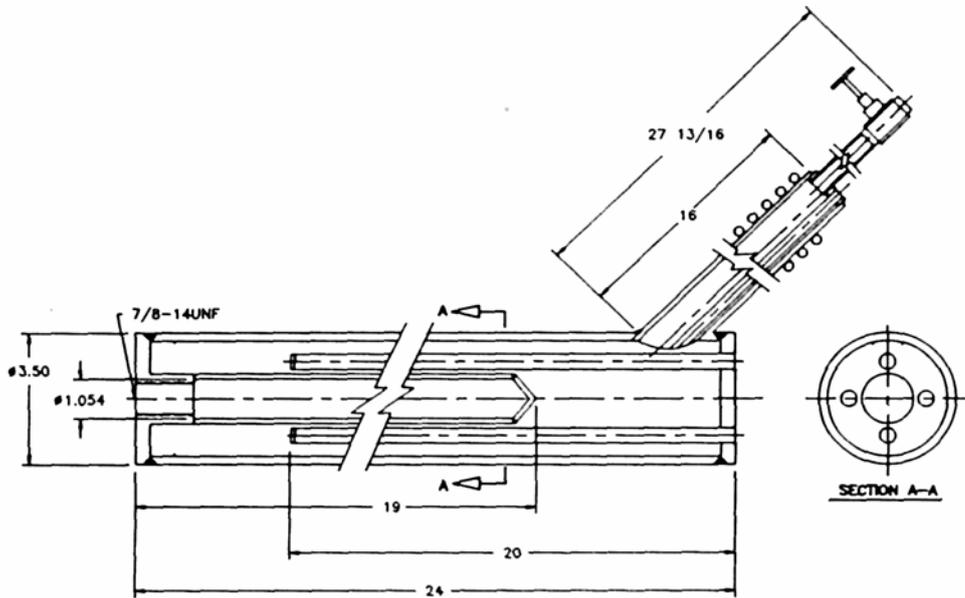


FIGURE 11
DESIGN OF Cs AND Na HEAT PIPES
FOR PRESSURE-CONTROLLED FURNACE

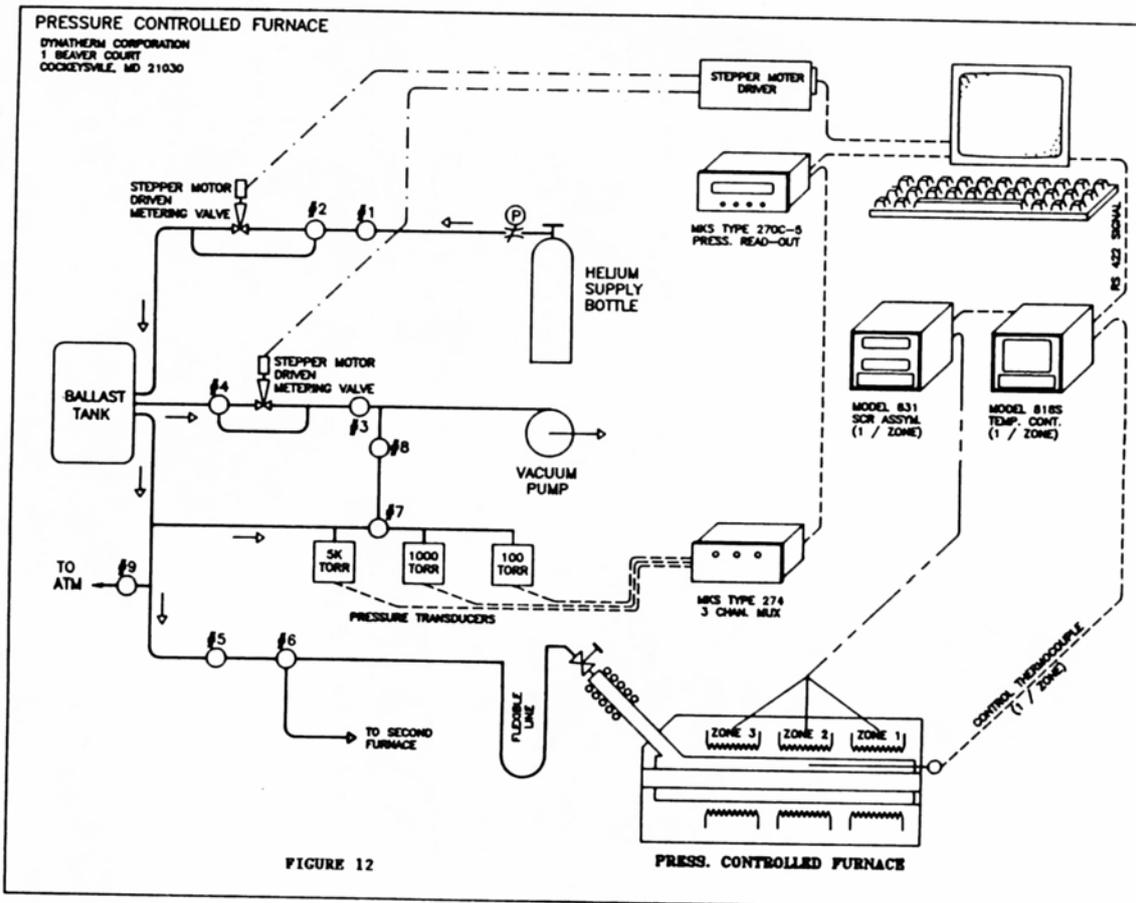


FIGURE 12
SCHEMATIC OF CONTROL ELEMENTS AND SYSTEM

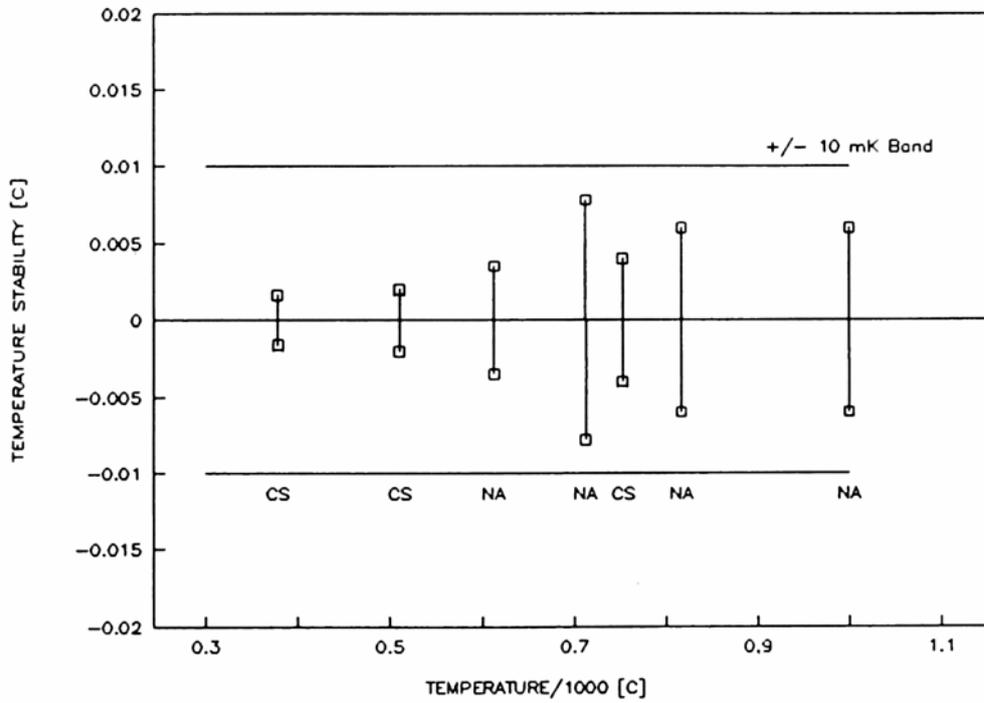


FIGURE 14
TEMPERATURE STABILITY
(CALCULATED FROM PRESSURE STABILITY DATA)

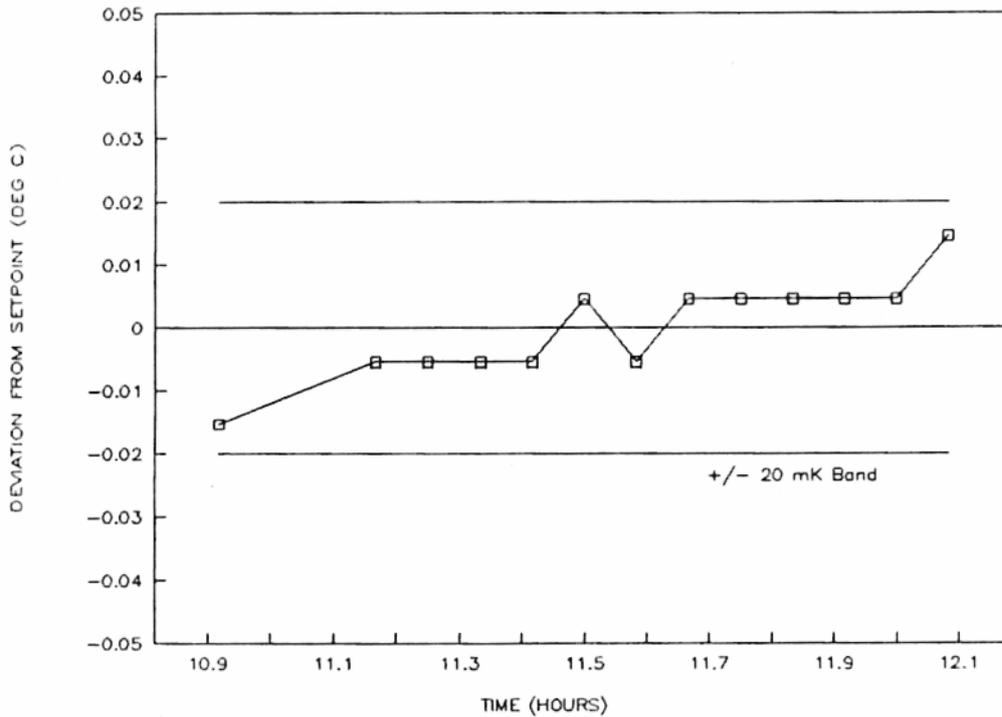


FIGURE 15
TEMPERATURE STABILITY (CESIUM AT 378°C)

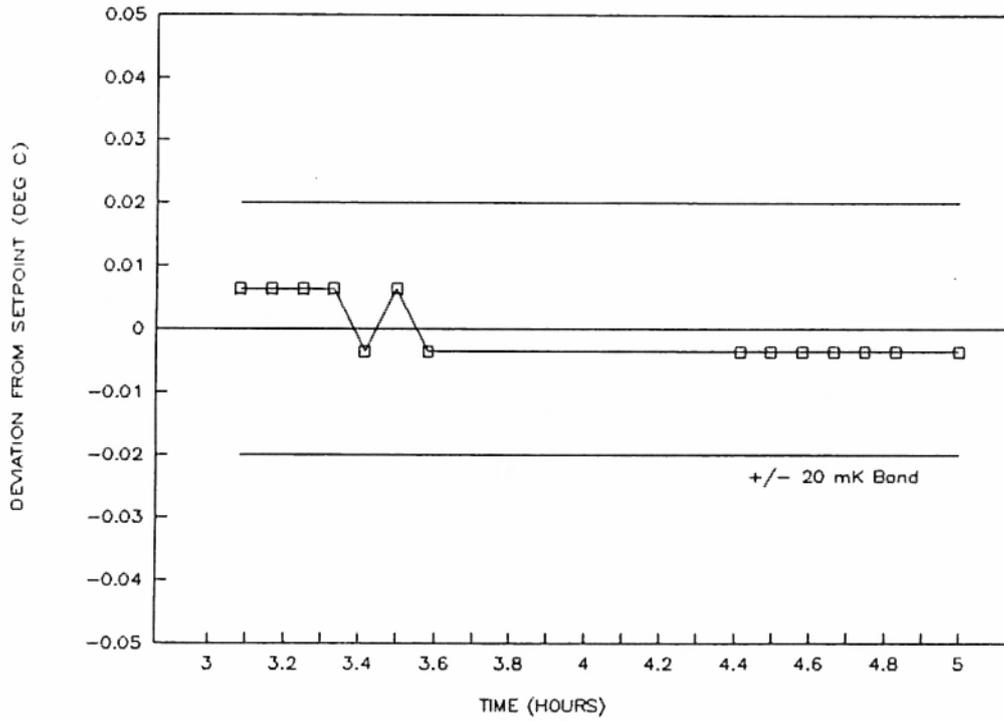


FIGURE 16
TEMPERATURE STABILITY (CESIUM AT 507°C)

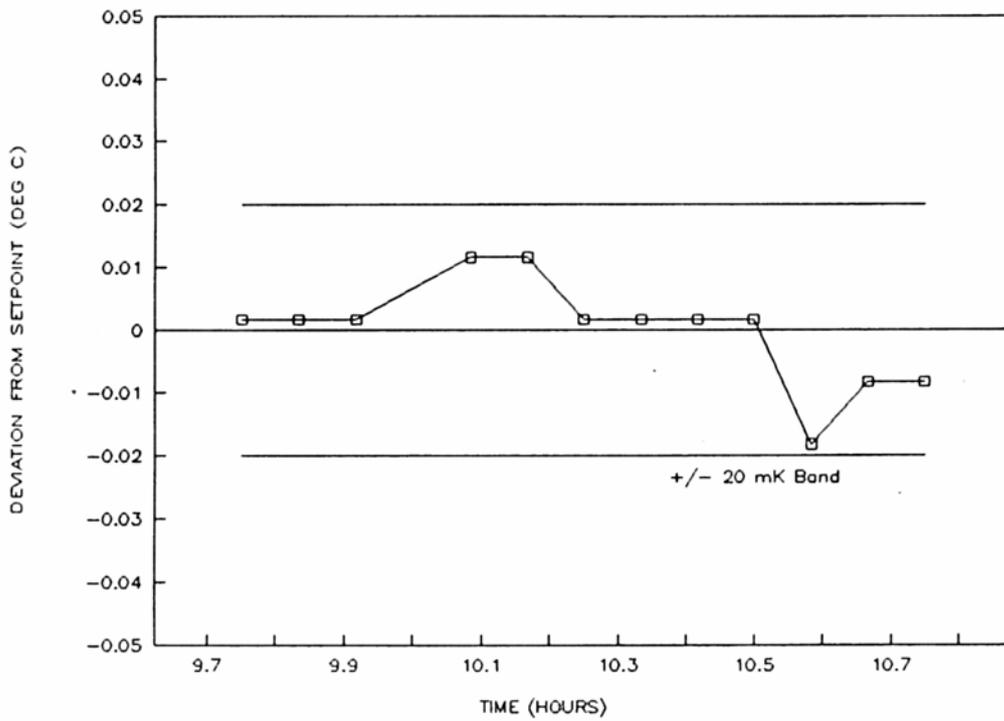


FIGURE 17
TEMPERATURE STABILITY (SODIUM AT 614°C)

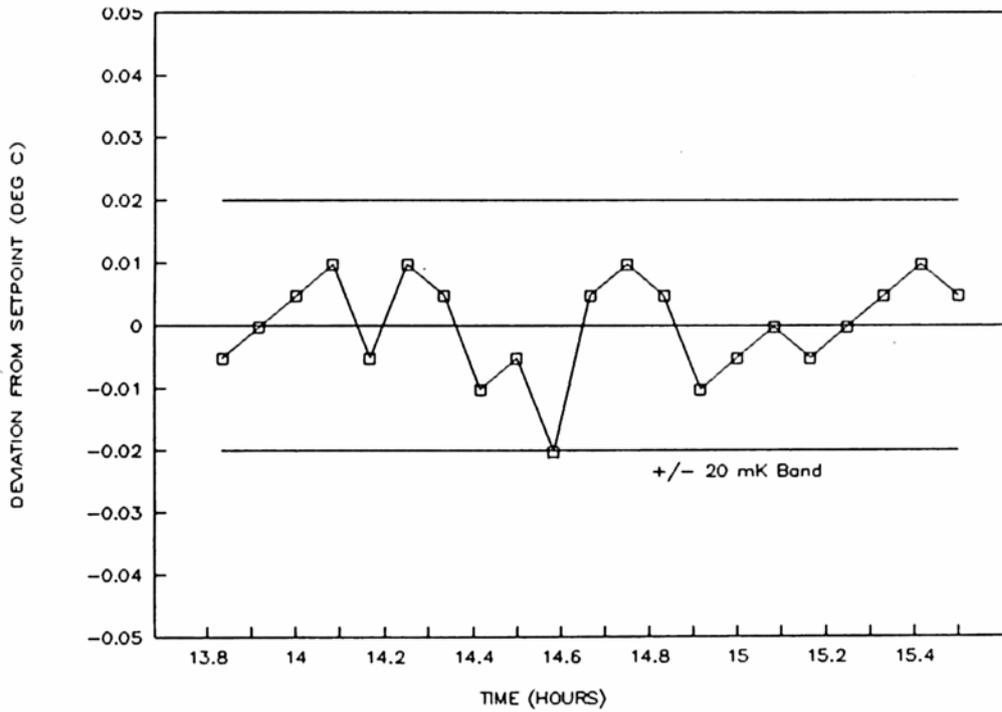


FIGURE 18
TEMPERATURE STABILITY (SODIUM AT 714°C)

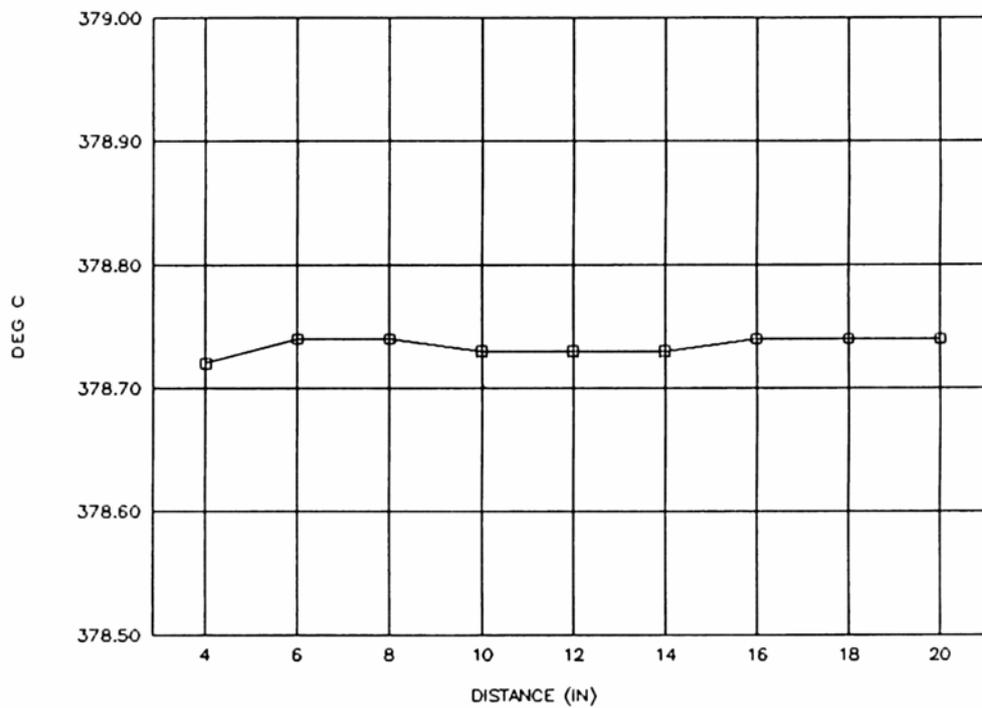


FIGURE 19
PROFILE OF WELL NO. 1 (CESIUM)

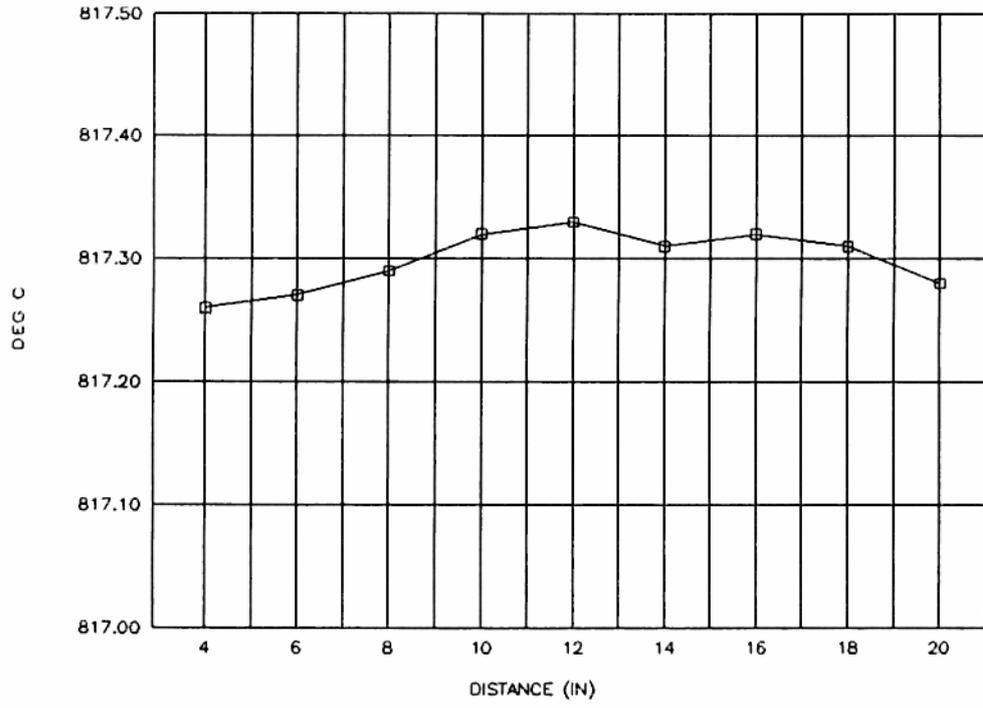


FIGURE 20
PROFILE OF WELL NO. 1 (SODIUM)

TWO NEW BOOKS ON THERMOMETRY

These books have been prepared by members of working groups of the Consultative Committee on Thermometry of the International Bureau of Weights and Measures (BIPM); the same Committee responsible for the International Temperature Scales, including the present International Temperature Scale of 1990. Because of their authorship and publisher, they represent official statements, and are intended as useful amplification of the text of the Scale.

SUPPLEMENTARY INFORMATION FOR THE INTERNATIONAL TEMPERATURE SCALE OF 1990, written by Working Group 1 of the CCT chaired by H. Preston-Thomas of NRC, Canada, is an exhaustive treatment of the realization of the Scale, including a great deal of how-to-do information which the text of the Scale itself does not contain. It begins by providing historical information on the development of temperature Scales beginning with the normal hydrogen Scale and the ITS of 1927. It discusses in useful detail the fixed points and their realization, platinum resistance thermometry, including the Scale algorithms over the resistance thermometer range; the vapor pressure Scales and methods of pressure measurement; gas thermometry and radiation thermometry. There are copious figures, valuable references, and important information about methodology and sources of error, related by eminent practitioners.

TECHNIQUES FOR APPROXIMATING THE INTERNATIONAL TEMPERATURE SCALE OF 1990, written by Working Group 2 chaired by Ronald Bedford of NRC, Canada, is a significant departure from custom (in its recognition of working laboratories) for BIPM, and seems to acknowledge that temperature scales are diverging further, with each revision, from the real world of practical metrology. The subject matter includes specialized fixed points (for example, superconductor and wire-bridge fixed point practice); germanium and rhodium-iron resistance thermometers, vapor pressure, magnetic, platinum and infrared radiation thermometers, and a wide variety of thermometers found seldom in primary standards laboratories and very often used in secondary and working laboratories: carbon, carbon-glass, diode, industrial platinum, thermistors, and base-metal thermocouples. An appendix provides the interpolation polynomials for standard thermocouple reference tables.

Both books are recommended for reading and for reference. NIST (Mangum) has had a limited number of copies for distribution; its is not known whether they are still in stock. Both are available from Bureau International des Poids et Mesures. The price is 95 FF (French Francs) for Supplementary Information, 105 FF for Techniques for Approximating, air post 60 FF, total 260 FF, or about \$50 US.

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CYRIL HOPE MEYERS. National Bureau of Standards, Washington D.C., Physics. Camron, Wisconsin, May 14, 1893. B.S. Oregon 1913, George Washington 1920. From Lab Assistant to Physicist, Bureau of Standards 1914 - Manometers and piston gages up to 200 atmospheres thermodynamics; vapor pressures and PVT gases and liquids, measurement correlation of data; platinum resistance thermometers and bridges. (From American Men of Science, 8th Edition)

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