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ERRATUM — VOL. 1 NO. 1

There are no doubt other errata in the mentioned issue, as there are no doubt typos. One erratum we have identified as potentially serious occurs on Page 28, in the algorithm for conversion between the Scale of 1968 and the Scale of 1990. It should read, completely, as follows:

$$t_{90} - t_{68} = \sum_{i=1}^8 a_i \left(\frac{t}{630}\right)^i$$

t_{90} was erroneously printed as T_{90} . We hope that no one was inconvenienced.

TIME TO SUBSCRIBE TO THE ISOTECH JOURNAL OF THERMOMETRY FOR 1991

Response to our original announcement of the JOURNAL was gratifying. We have distributed almost 400 copies of Vol. 1 No. 1. Since we accept subscriptions on a full-year (two issues) basis only, distribution of Vol. 1 No. 2 will be the same. A few copies of Vol. 1 No. 1 remain, and can be had on a first-come first-served basis.

We hope that the acceptance of Volume 2, for 1991, will be at least equivalent, and we solicit your subscription now, to permit us to plan ahead.

Experience demands a change in our subscription price for Vol. 2. The charge will continue to be \$15.00 or £10 for the two 1991 issues, postage paid, for subscriptions which are accompanied by a cheque and do not require paperwork. For subscriptions by purchase order, which require that we issue an invoice, the charge will be \$25 or £16.

An order form is enclosed so that you need not deface your copy of the Journal. Subscription orders may be sent to either of the following:

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FUNDAMENTALS OF THERMOMETRY

PART II

FIXED POINTS OF THE ITS—90

Henry E. Sostmann

CONFIDENCE IN THE METAL FREEZING POINTS OF ITS—90

John P. Tavener

ABSTRACT

As Part II of the ongoing series generally entitled "Fundamentals of Thermometry", Sostmann discusses the fixed points of the International Temperature Scale of 1990 (ITS-90) which encompass the long stem platinum resistance thermometer range.

A cogent question is: "How does the user know that the equipment he uses to realize these fixed points indeed reproduce the definitions of the Scale?" and because the definitions are superior to any measurement which can be made upon them, there is no direct answer to that question. However there are indirect answers, and this discussion is interspersed with Tavener's description of his own adventures in pursuit of them.

INTRODUCTION (Sostmann)

We saw in the previous article in this series⁽¹⁾ that the International Temperature Scale of 1990⁽²⁾ defines a temperature scale in terms of fixed points; states in nature in which pure materials exhibit equilibrium between two or three phases, during which equilibrium states temperature is invariant. It has become popular to refer to these equilibrium conditions as 'intrinsic standards'.

In the dictionary definition of intrinsic, we have

Intrinsic, (adjective): belonging to the inmost constitution or essential nature of a thing, essential or inherent and not merely relative ... as the very essence of the thing in virtue of the metaphysical structure of the universe ...⁽³⁾

from which we infer that the intrinsic property derives from its fundamental nature and not from an applied attribute, such as the result of a calibration. A committee of the National Conference of Standards Laboratories has achieved this definition of an intrinsic standard, applicable, for example, to the Josephson Volt, the Von Klitzing Ohm, and the equilibrium fixed points of the Temperature Scale:

Intrinsic standard (noun): Designated measurement standard de-rived from the innate value of natural physical constants whose value can be accurately reproduced under carefully controlled conditions.

I believe that this definition is insufficiently pure because of its reference to "accurate reproduction" - the Committee definition includes within its scope the artifact and the experiment with which the value is realized - and I offer the following definition in its place:

Intrinsic standard (noun): a standard of measurement having the property that is the ideal value of the natural physical constant which defines it. The assignment of a numerical value on a Scale may be arbitrary.

My definition differs essentially in that the "standard" is the ideal physical state, approachable only as a limit, and requires that the value which is obtained from the artifact and the experiment must be shown to approach the natural value, preferably within some assignable degree of uncertainty.

The fixed points of the Scale are intrinsic standards; indeed, it would be difficult to build a working temperature scale on any other foundation. The freezing, melting and triple-point temperatures of pure metals and the three-phase equilibria of pure gases, in the ideal case, are constants of nature. As such, they can be approached by measurement, but we recognize that they are superior to any measurement which can be made of them.

Artifact embodiments of intrinsic principles may deviate from ideal from a number of causes. Among them are these:

- (1) Although made from material as pure as obtainable, they are not ideally pure. For example, pure metals used in metal freezing point artifacts are generally impure by less than 1 ppm, but they must still be regarded as very dilute alloys.
- (2) They may be contaminated in manufacture, or in use, perhaps over long periods of time.
- (3) They generally require correction for such things as ambient pressure, hydrostatic head, etc., which are in themselves the results of measurement and subject to measurement error.
- (4) In use, the measuring system may not be in ideal thermal equilibrium with the phase equilibrium temperature.

These, and other effects, lend great importance to the establishment of confidence in the reproducibility and uncertainty assignable to fixed point standards as they are represented by the artifacts we use to calibrate thermometers. The fixed points which are used to define the Scale over the range of the long-stem platinum resistance thermometer and their associated pressure coefficients are shown in Table 1.

We will look first at the temperatures provided by the freezing points of high purity metals, and following that, the triple points of, mercury, argon and water. In this discussion I will relate some of my own experiences as a manufacturer of cells at YSI, and Tavener will describe his further experiences after Isotech acquired this product line in 1987.

THE FREEZING POINTS OF HIGH PURITY METALS (Sostmann)

It is usual practice to confine the metals in cells which contain them during the process of melting and freezing, provide an axial well into which a thermometer may be inserted, include some means (either by gas purging or by a totally sealed enclosure) for ensuring that the metal is not contaminated in use, and which are made of materials which themselves are not sources of contamination. Most impurities (although not all) depress the freezing point of a metal. As a generality, impurities at the level of 1 ppm will depress the freezing point between 0.3 and 1 mK.

Figs. 1(a) and 1(b) show two common designs of cell, used for indium, tin, zinc, aluminum and silver, and the less-often used metals such as bismuth, lead and cadmium. The cell shown in 1(a) contains the metal in a crucible of purified graphite. The well for the thermometer is also sheathed in graphite. The system is surrounded by an envelope of fused quartz, which is continuous and extends to the bottom of the thermometer well. This total enclosure permits the manufacturer to purge the cell with vacuum and inert gas, and finally to seal the cell with a gas content of pure argon at a pressure of 1 standard atmosphere at the freezing point of the metal. Thus the interior of the cell becomes a world in itself, and, unless broken, is not in danger of contamination from outside sources, and does not require correction for the effects of ambient pressure.

I recommend sealed cells for all users. I have used some cells for as much as 20 years without experiencing shifts in the temperature realized. However there is a theoretical objection that the internal pressure cannot be measured in use, and the realized temperature would vary with pressure, if for some reason the built-in pressure of a sealed cell should change. Fig 1(b) shows a cell which is identical except that it is not sealed. Instead, provision is made for gas purging during the heating and cooling of the cell. Some (but by no means all) National Laboratories feel it their legal responsibility to use open cells, and I cannot disagree; they must seek for the ultimate assurance. But the required pressure measurement is a possible source of uncertainty. Also, the blanking gas must be absolutely oxygen-free, since some metal freezing temperatures (silver, for example) are depressed by the presence of oxygen. (This is usually done by passing the gas through a getter, such as copper wool held at about 500°C).

A general discussion of how a metal freeze point is realized, and the equipment and technique necessary for doing so, is given in a previous issue of *The Isotech Journal of Thermometry*⁽⁴⁾. While some metals require detail variations of technique, the general procedure is as follows:

A cell is assured to be in the solid state, as it will be if it has been stored at ambient temperature. The cold cell is inserted into the furnace and the furnace controller is set a few degrees (e.g., 5°C) above the melt temperature.⁽⁵⁾ A monitoring thermometer is placed in the well of the cell. At the onset of melting, the temperature rise will cease (this is called the "melt arrest") because the heat supplied is absorbed in phase change rather than temperature change. When the metal is completely molten, the monitoring thermometer will indicate a rise to the temperature of the controller setting. The controller is then set slightly (e.g. 1°C) below the freeze temperature. As the temperature drops, the monitoring thermometer will indicate a decrease in cell temperature. The metal will remain liquid, briefly, below the temperature at which it will freeze ("supercool", and then, as the first crystal nucleation occurs, will rise to the freeze plateau as the latent heat is released. The slow re-release of latent heat will maintain

the metal on the freeze plateau for some period of time, following which the metal is completely solidified, and the temperature will drop to that of the controller setting. Fig. 2 illustrates this cycle.

More information is given in Tavener's comments in this article. In addition, Isotech furnishes most comprehensive operating instructions with its furnaces and cells.

CONFIDENCE IN THE FREEZING POINTS OF HIGH-PURITY METALS (Tavener)

Isothermal Technology is in the unusual position of being both a Nation-ally accredited laboratory and a producer of commercially-distributed metal equilibrium fixed-point cells and ITS-90 thermometers. We must therefore satisfy ourselves, our customers, and the National Physical Laboratory of England, which supervises us, that the cells we use for our own calibrations, and the cells we sell to others, are acceptable embodiments of the intrinsic standards which define ITS-90.

There are several indirect approaches to the verification of a cell. As part of our application to our National Accreditation and Assessment Authority (NAMAS) to assess our capability, establish evaluation procedures and designate our uncertainty of measurement, our Certificate of Accreditation includes the following text:

In order to evaluate the performance of your fixed-point cells, the following procedure should be carried out:

- (1) Total melt, followed by fast freeze to segregate any impurities
- (2) Slow melt, to be fully plotted from initial undercool to final rise
- (3) Freezing curves, for all cells except the gallium cell, followed by determination of plateau temperature using NPL calibrated Standard Platinum Resistance Thermometers.

The metals used in freezing-point cells are the purest we can obtain; 99.9999% (or in the parlance of the trade, "6 nines) or better. "Better" must be taken qualitatively, since at 6N purity we approach the limit of spectrographic analysis. Nevertheless, these metals must be regarded as having minute fractions of impurity which usually (but not always) de-press the freezing point. Ideally pure materials have unique melting and freezing temperatures, whilst alloys exhibit a melting or freezing range. Examination of the melting range, (2) in our qualification requirements, provides an estimate of the purity of the metal. Fig. 3 shows the typical phase diagram of a dilute binary alloy. Table 2 shows the effects upon melting and freezing range of a selection of possible impurities, taken one at a time in small amounts, and makes clear the sensitivity of the melt plateau slope as opposed to the freeze plateau slope in the detection of impurities and the qualification of cells.

When we use freeze point cells to calibrate thermometers, we first hold the metal molten at about 5°C above the melt temperature overnight, to thoroughly mix the metal by convective stirring, and to distribute any previously segregated pockets of impurity. We then freeze the metal slowly, by setting the surrounding temperature slightly below the metal's freezing temperature and by introducing a cold rod or cold thermometer into the re-entrant well of the

cell. (Tin, because of its large supercool, requires a somewhat different technique). We obtain a very flat freeze plateau, with a thin layer of solid material formed on the outer and inner walls, and the liquid-solid interface progressing away from the thermometer. This is a form of zone-refining, sweeping the minute quantity of impurity ahead of the interface. This method forms the most constant and reproducible plateau for calibration. A brief discussion of freeze and melt curves, and their dependence upon recent history, is given in Annex 2.

However to assess the purity of the metal, we consult the melting curves. Fig. 4 shows that three cells with various levels of impurity all give rather flat freezes. Inspection of the melting curves tells us, immediately, which cell contains the most pure metal.

In melting studies, the metal is held molten at 5°C above the melt temperature for 12 hours, to distribute the impurities homogeneously throughout the ingot, and then frozen rapidly by removing the cell from the furnace into ambient temperature. It is then slowly melted, with a monitoring thermometer connected to a recorder. Informed inspection of the melting curve indicates to what extent the cell is an acceptable embodiment of an intrinsic standard.

Thus, a comparison of the melting curve obtained after a fast freeze with the freezing curve as the metal is frozen slowly (as it is during a thermometer calibration) is one necessarily oblique but useful means of qualifying the purity of the metal in a freeze-point cell⁽⁶⁾. Other methods are described by other authors.

THEORY INTO PRACTICE (Tavener)

I will now discuss our own experience in performing just such analyses upon cells which we have manufactured. I begin with silver and aluminium, because these are new points of the ITS-90, and only now can be studied adequately, with the high-temperature platinum resistance thermometers recently made available.

THE FREEZING POINT OF ALUMINIUM * 660.323°C (Tavener)

To realize the plateau, we allow the molten ingot to cool slowly (≈ 0.1 K/min) until it is below the freezing point and at the temperature of supercool. Aluminium usually supercools 0.4 to 0.6 K. At first, concerned about shocking thermometers, we did not withdraw the thermometer from the hot cell as one does with tin and zinc. This led us to very long recalescent curves. We now believe, at least with our own 0.25Ω thermometers, that a thermometer is restored very nearly to its original state within 30 minutes after it is reinserted into the cell. Also, at first, we tried for long plateaux, and because of the excellent isothermal proper-ties of the sodium heat pipe which is the core of our high-temperature furnace, we obtained 24 hour plateaux. We expected to see flatter plateaux on long freezes and were surprised to find that short ones were flatter. This seems to be not a function of the cell, but

of slow changes in the resistance thermometer over 20-plus hours.
This leads us to the following recommendations:

- (1) Do initiate a freeze by cold-quenching with cold quartz rods after withdrawing the thermometer. Then, after reinserting the thermometer, allow half an hour for it to anneal before believing the measurement.
- (2) Control the plateau length to 4 to 6 hours to minimize drift of the thermometer.

Using these techniques, we expect cells to show slopes flatter than 2 to 4 mK during 50% melt and flatter than 1 to 2 mK during 50% freeze. We have now made measurements on 8 cells, during which time we developed these techniques. Cells 4, 7, 7 and 9 of the table below were done with-out withdrawing the thermometer. Cell 10 used the new technique I have just described.

CELL NO	FREEZE PLATEAU			MELT PLATEAUX			%	SLOPE
	TIME	%	SLOPE	TIME	%	SLOPE		
4	10 h	50	0.9 mK					
7	13 h	70	1.0 mK					
8	24 h	50	1.0 mK					
9	16 h	50	0.9 mK					
10	10 h	50	0.3 mK	18 h	50	0.6 mK	90	3 mK

THE FREEZING POINT OF SILVER * 961.78°C (Tavener)

The freezing point of silver is the most difficult to evaluate, because analysis may be complicated by changes in the characteristics of the monitoring thermometer due to the high temperature. We began by looking for long plateaux, and in our isothermal furnace we can produce plateaux of 24 and longer. However these long plateaux appeared to be inferior in stability to plateaux lasting only 4 to 6 hours. We finally understood that we were seeing thermometer changes rather than plateau temperature changes.

The specification on a respected competitive high-temperature platinum resistance thermometer indicates that a drift of about 43 mK per 100 hours at temperature may be expected, or, if it is linear, about 10 mK for one of our 24 hour plateaux. However it is also accepted that with-drawing a thermometer from 961°C to ambient alters its characteristics. We tried quenching 4 of our Model 961 thermometers from the silver point to ambient, and averaged changes, at the water triple point, of +35 mK equivalent resistance and, at the gallium point, -0.00035 resistance-ratio. It required 24 hours at 650°C followed by slow cooling to 450°C to restore these thermometers to close to their initial values. We now use the following procedure:

- (1) Plateaux should be restricted to 4 to 6 hours.
- (2) In nucleating the melt, remove the thermometer from the silver cell and place it immediately in the preheat tube which is built into the Isotech high-temperature furnace. Cold-rod the cell with quartz tubes rather than quartz-rods, to avoid cold shocks which are too large.
- (3) Place the thermometer in the cell again, and wait 1/2 hour for the thermometer to reanneal before making measurements.

If this procedure is followed, 50% melt should occur over 4 to 6 mK, and 50% freeze over 1 to 2 mK, including thermometer stability over the measured time.

At Isotech, we have evaluated 8 cells during this learning phase, and the following is a representative selection of results:

CELL NO	FREEZE PLATEAUX			MELT PLATEAUX		
	TIME	%	SLOPE	TIME	%	SLOPE
4	2 h	50	0.2 mK	4.5 h	50	1.0 mK
9	24 h	50	5.0 mK	2.5 h	50	0.5 mK
10	3.5 h	50	0.5 mK	6	50	1.0 mK

THE FREEZING POINT OF INDIUM (Sostmann)

Indium is a new defining point of the Scale, although IPTS-68 included it as a secondary reference point. My own production experience includes only 6 indium cells. It is a most satisfactory point to realize, and occupies a position of great utility. The statistics of my 6 indium cells is shown in Fig. 5.

THE FREEZING POINT OF INDIUM * 156.5985 (Tavener)

For evaluation, the metal is melted overnight at 5°C above the melt temperature, removed from the furnace for 30 minutes to fast-freeze the ingot; meanwhile the furnace is set to 5°C above the melt temperature. The cell is placed in the furnace again, and the temperature monitored. We experience a melting range of less than 0.5 mK with a plateau duration of more than 4 hours.

When the metal is completely molten, the furnace is reset 0.5°C below the melt temperature. When the monitoring thermometer indicates that the slight supercool (0.5°C) of indium is achieved, the thermometer is removed, and two cold quartz rods are inserted, in succession, for 2 to 3 minutes each. The thermometer is then returned to the cell. The effect of inserting the quartz rods is to quickly freeze a thin mantle of metal on the thermometer well, releasing sufficient latent heat to raise the melt to the equilibrium temperature. We have now built 5

indium cells from 2 batches of metal, and have found plateaux longer than 6 hours, melting slopes between 0.2 and 0.3 mK, and freeze plateaux between 0.1 and 0.2 mK. The absolute value has been 156.5985°C within our measurement uncertainty of 1 mK, and repeatability better than 0.1 mK.

THE FREEZING POINT OF TIN (Sostmann)

My experience includes the production of 21 tin cells. Statistics of these are shown in Fig. 6. While I believe these to be very good results, I think that the lack of a strong central tendency is the result of thermometer oxidation effects and measurement uncertainty.

THE FREEZING POINT OF TIN * 231.928°C (Tavener)

The cell is taken through an overnight melt and a quick freeze outside the furnace, like indium. When the metal is frozen, the cell is returned to the furnace, which is set 5°C above the melt temperature, and the temperature is monitored.

Achieving the freeze plateau requires a different technique than other metals, because tin supercools as much as 10 to 15°C. If the furnace were set low enough to allow supercooling the furnace, it would not recover in time to allow a reasonably long plateau. Therefore the cell of molten metal is lifted free of the furnace until nucleation is observed, and then quickly returned to the furnace, which is controlled 0.5°C below the freeze temperature. Two cold quartz rods are inserted in succession, to cause nucleation around the thermometer well, and the thermometer replaced to track the tin freeze plateau.

We find long melt and freeze plateaux which correspond closely. The 6 cells produced to date have exhibited melt and freeze plateau slopes of 0.1 to 0.2 mK, with reproducibility of 0.1 mK. There is some difficulty which relates to the measuring thermometer, of minor order, because platinum tends to show an oxidation shift at this temperature⁽⁷⁾. The effect is made negligible by using the ratio W of the resistance at temperature to the resistance of a measurement at the triple point of water immediately following.

THE FREEZING POINT OF ZINC (Sostmann)

I show, in Fig. 7, statistics on 23 zinc cells made before 1987.

THE FREEZING POINT OF ZINC * 419.527°C (Tavener)

Zinc is manipulated in a manner similar to indium. However, for melting curves taken after freezes, zinc seems to show a "memory" of the freeze plateau. In general the melt plateaux are not as constant as the freeze plateaux, but differ by a few tenths of a mK for highest-purity metal to as much as 3 mK for metal less pure. Of the 7 zinc cells we have made to date, from 2 batches of material, we have measured melt slopes from 2 to 3 mK, and freeze slopes from 0.1 to 0.2 mK. Within our measurement uncertainty, all cells have reproduced the ITS-90 value of 419.527°C. If a procedure for freezing is set and rigorously followed, reproducibility is 0.1 to 0.2 mK. Oxidation may here also be a factor, and use of the W ratios is essential for highest precision.

THE MELTING POINT OF GALLIUM (Sostmann)

The melting point of gallium⁽⁸⁾ is also a new defining point on the ITS-90; so new that it was not mentioned even as a secondary point on the IPTS-68. The melt temperature is 29.7646°C. It is an important point, at a temperature useful in many disciplines requiring measurements in the near-ambient or biological range. It was first developed for calibrating the small thermistor sensors used in biomedical studies, because of the proximity to body temperature.

Indium, tin, zinc, aluminum and silver are operated as freezing points, for reasons which were described above. Gallium is used on the melt instead, because, unlike most other metals, it expands when it freezes, and the pressure effects of the metal as it does so interfere with the realization of the equilibrium temperature. Because of the solidus expansion, the cell must be made of a relatively elastic material; Teflon is a common choice. A typical gallium cell is shown in Fig. 9. The interface free energy distribution in Teflon also inhibits the long supercool of gallium (~75 °C) which would be intolerable in practice.

Over the years, I have supervised the manufacture of 141 gallium cells. Measurements were made of these using SPRTs calibrated by the NBS and also by the Physikalisch-Technische Bundesanstalt of West Germany. The statistics of these cells are summarized on the bar chart of Fig. 8.

An anecdote. I once made a gallium cell for the National Institute of Metrology (Beijing). Of course I made a number of measurements of the plateau temperature before it was delivered, and Dr. Ling Shan Kang made a number of measurements after he received it. The two realizations of the Scale, as transferred to thermometers, were entirely independent; nothing was common except the gallium cell. The difference between our measured mean values was 120 pK!

THE MELTING POINT OF GALLIUM * 29.7646°C (Tavener)

We cycle the gallium cell in an essentially automatic apparatus, our Model 17402, which has a two-position switch for MELT and FREEZE. The apparatus is set for MELT and the internal temperature of the thermometer is monitored with a thermometer in its well. Just before the melt point is reached, 17 cm³ of water at about 50°C is introduced into the well in order to start an inner melt and to provide a thermal transfer medium for the thermometer. We have to date produced more than 30 gallium cells, with 0.1 to 0.2 mK stability over 50% of the melt, plateaux of 8 to 20 hours duration with a range of 0.1 to 0.2 mK, and reproducibility of temperature better than 0.1 mK. Absolute values are 29.7646°C within our Laboratory uncertainty of 1 mK.

THE TRIPLE POINT OF MERCURY (Sostmann)

The triple point of mercury, -38.8344°C , appears first as a defining fixed point in ITS-90. (The freezing point, not the triple point, of mercury was a secondary reference point of IPTS-68). It is the only defining point between 0.01°C and the triple point of argon, $-189.3442^{\circ}\text{C}$. It is therefore a point of great value to users whose range of interest extends below 0°C but not into the very cold regions.

The general design of the mercury cell is similar to that of the sealed cell of Fig. 1(a) except that a graphite crucible is not necessary (and, cannot be used, since it would outgas). Mercury triple point cells have been made in envelopes of borosilicate glass and of stainless steel. Since mercury is considered to be a hazard in the workplace, glass envelopes are distinctly contraindicated. The stainless cell, fusion welded at its few joints, evacuated and fusion sealed, reduces the atmospheric contamination probability to nearly zero. The mercury is purified to approximately 10 parts per billion impurities. A typical cell contains about 4 kg of metal.

THE TRIPLE POINT OF MERCURY * -38.8344 (Tavener)

The mercury we use contains impurities of less than 15 parts per billion. It is prepared by chemical washing and triple distillation, and analysis to that level is made possible by boiling off 90% of a sample and analyzing the residue. The mercury is contained in a welded stainless steel cell, which sealed containing only mercury and mercury vapor.

The cell is fast-frozen overnight at about -42°C . It is then placed in our own Model 17724 Mercury Triple Point Apparatus, which allows close control of the refrigeration temperature around the cell. The control setpoint is 0.1°C above the melting point. When the monitoring thermometer indicates that the cell temperature is very close to the melting point, the thermometer is removed, and warm quartz rods are inserted to create an inner melt. The thermometer is then replaced. Melts of 12 hours duration are typical.

When the metal is completely molten, the control temperature is set 0.1°C below the freeze point. The monitoring thermometer is removed, and quartz rods chilled in liquid nitrogen are inserted to form a thin frozen sheath around the thermometer well. The thermometer is replaced, and the plateau tracked. Typical freezes and melts are shown in Fig 10. It should be noted that the triple point can be approached either from the warm or the cold side. I have described the approach from the warm side.

UNCERTAINTIES IN USE (Tavener)

Although Isotech has been making and testing cells since only June, 1989, enough cells have been made to allow us to make some estimates of what can be expected of them.

Of course, as a British Laboratory supervised for our accreditation by NPL, we cannot claim uncertainties smaller than those of our supervising Laboratory. (The same would be true for a manufacturer or a National Calibration Service in any other country.) It is considered appropriate for a NAMAS Laboratory to claim uncertainties no smaller than 1.5 to 2 times those of the National Laboratory. In the United Kingdom, this results in the following table:

Temperature	NPL thermometer calibration uncertainty [\pm mK]	Isotech cell calibration uncertainty [\pm mK]	Isotech thermometer calibration uncertainty [\pm mK]
Hg triple	0.5	1	2
Water triple	0.5	1	1
Ga melt	0.5	1	2
In freeze	1	2	3
Sn freeze	1	2	3.5
Zn freeze	1	2	3.5
Al freeze	4	6	10
Ag freeze	20	30	40

If these appropriately conservative values are compared with reported uncertainties during melt-freeze experiments, it will be realized that the freeze points, melt points or triple points discussed can operate well within the limits of the above Table. The estimates for aluminium and silver given below are based on the techniques described in this paper for 50% melt or freeze, and include the stability of the Isotech Model 962 (Hotspot) High Temperature Platinum Resistance Thermometer over a 4 to 6 hour plateau.

Metal	Melt range [mK]	Freeze range [mK]	Reference
Mercury	0.2	0.2	(9)
Gallium	0.2	Not applic.	(8)
Indium	2	0.2	(6)
Tin	2	0.2	(6)
Zinc	3	0.3	(6)
Aluminium	2-4	1-2	This paper
Silver	4-6	1-2	This paper

ANOTHER USE FOR MELTING POINTS (Sostmann)

There may be a totally different reason to be interested in melting points, rather than freezing points, with the uncertainties Tavener has just described.

We have seen that, to achieve a proper freeze plateau, a good deal of hands-on manipulation is required. Melting points, on the other hand, require only that the furnace controller be set, and the furnace turned on (which can be done by a timer, before the laboratory day begins). Some laboratories, whose charter is for production of calibrations, with a heavy work load and a high throughput, and which do not require the ultimate in accuracy, may choose to operate on the melt plateau instead of at the freeze equilibrium. The equipment is the same, the uncertainty will be higher, and the time consumed in establishing the calibration point is substantially reduced. ⁽¹¹⁾

THE TRIPLE POINT OF ARGON (Sostmann)

There exist in the literature many descriptions of the realization of the triple point of argon⁽¹⁰⁾. The argon triple point requires highly special equipment and a source of a liquid gas which is expensive and not readily available.

Most laboratories will choose to calibrate thermometers at this end of the range by a comparison method, where the thermometer under test is compared to a thermometer of known calibration, usually a valid calibration from a National Laboratory. Indeed, the National Laboratories will most often calibrate thermometers submitted to them by comparison, realizing the argon triple point only for infrequent periodic calibrations of their own standard thermometers. In its publication "Adoption of the ITS-90", the National Physical Laboratory of England makes this statement:

Most thermometers submitted (for calibration) will involve measurements at the tin and zinc freezing points, plus a comparison with NPL standards in a bath of liquid nitrogen (about -195°C)

Fig. 11 shows a bath intended specifically for the comparison calibration of long-stem thermometers, against a reference standard thermometer, in liquid nitrogen; Isotech Model 18205. The apparatus comprises a stainless steel dewar, an inner equalizing block, three wells for thermometers, top connections for filling and monitoring the height of liquid refrigerant, and a manifold by means of which the thermometers may (optionally) be thermally tied down. Using this comparator, calibration uncertainties not larger than 0.002°C can be achieved at about 1/10 the cost of an absolute calibration.

With regard to electrical measurements, using a resistance bridge which employs a single reference resistor, a useful technique is to connect the reference thermometer in place of the reference resistor, and the test thermometer in place of the unknown, and thus read the ratio of the unknown thermometer to the reference thermometer. Since the slopes of standard platinum resistance thermometers are so similar, at least over limited temperature intervals, the ratio of the resistances is representative of the resistance of the unknown, and the resistance of the unknown can be calculated quickly from the calibration table of the calibrated thermometer. The calibration temperature need not be exactly established, nor even need be known precisely.

THE TRIPLE POINT OF WATER * 0.01°C (Sostmann)

Of the defining fixed points of the ITS-90, the triple point of water is the first among equals. It is the sole realizable fixed point common to the Kelvin Thermodynamic Temperature Scale and the ITS-90. Its as-assigned value on these Scales is 273.16 K and 0.01°C, respectively. It is one of the most accurately realizable of the defining points. Properly used, the triple point temperature can be realized with an accuracy of +0.0°, -0.00015°C. (For comparison, it is difficult to set up and use an water and ice bath with accuracy better than 0.002°C). The triple point of water can be maintained easily in a replenished bath of ice and water for a number of days, and in an apparatus dedicated to the purpose, Isotech Model 18233, for weeks or months, making this fundamental point continuously available in the Laboratory.

Also, the triple point of water is the temperature to which the resistance-ratios of SPRT calibration tables are referred:

$$W = \frac{R(t_2)}{R(t_1)}$$

where on IPTS-68 t_1 was 0°C, and on ITS-90, t_1 is 0.01°C.

Furthermore, the triple point of water provides a useful quality-assurance check in verifying the condition of thermometers. A measurement at the triple point of water immediately upon a thermometer's return from a calibration journey will reveal any shift which has occurred in transportation. Valuable continuing history of a thermometer's stability is preserved if measurement results are placed on a control chart each time the thermometer is measured at the triple point of water.

One design of water triple point cell is shown in Fig. 12. There is a glass flask nearly filled with pure water, and sealed containing only water vapor in the headspace. A coaxial well is provided for the thermometer.

In use, the cell is chilled to about 0°C in an ice and water bath, and then a mantle of ice is frozen onto the thermometer well by, for example, filling the well with crushed Dry Ice. When a good mantle has been formed, the well is cleared of any remaining ice and water, and a warm rod inserted to melt a thin film between the ice mantle and the well. (This can be detected by holding the cell upright and giving it a short sharp twist. The mantle should spin freely about the thermometer well). When freshly frozen, the mantle will be under some strain which will tend to elevate its temperature. It will relax in about 24 hours. Specific instructions and precautions are given in manufacturer's literature, and the use and preparation of water triple point cells is the subject of a draft ASTM standard which is expected to be published in 1991.

The problem of verifying the temperature of a water triple point cell to date is intractable. Some manufacturers certify cells by comparing a new cell with a bank of as many as 20 old cells, kept for that purpose; others do not certify their cells. Assuming that a cell contains properly pure water, it is possible to determine that the cell contains only water vapor in the headspace, and that it has not leaked air.

- 1: Holding the cell upright, turn it over slowly. A bubble of water vapor will collect in the upper tubulation or handle, which will compress to the size of a small pea, or even

disappear. This effect is more sensitive when the cell is at room temperature.

2: Again turning the cell over slowly, a sharp click will be heard, similar to water hammer in a pipe, as the gas bubble collapses. The click is more pronounced when the cell is cold.

These two tests are the only verification which a user can perform on the cell.

Laboratories which have space or budget for only one fundamental temperature standard should consider the water triple point favorably.

FINAL WORD

In our next issue, Vol. 2 No. 1, this series will discuss in detail platinum resistance thermometry.

USEFUL REFERENCES

Instruction manuals furnished with Isotech furnaces, fixed point cells and platinum resistance thermometers. These may be purchased separately from a product, if desired. Isothermal Technology Ltd., Southport, England, or Durham, North Carolina.

Mangum, B.W., Platinum Resistance Thermometer Calibrations, NBS Special Publication No. 250-22. Superintendent of Documents, Washington. (IPTS-68 Scale, but still useful).

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FOOTNOTES

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(2) Preston-Thomas, H., *The International Temperature Scale of 1990 (ITS-90)*, Metrologia 27, No. 1 (1990); also *Errata.*, Metrologia 27, No. 2 (1990).

(3) Webster's Third New International Dictionary

(4) Sostmann, H.E., *Practical calibration of thermometers on the International Temperature Scale of 1990*, Isotech Journal of Thermometry, Vol. 1 No. 1, pp. 25-27 (Spring 1990)

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(4) McLaren, E.H., *The freezing points of high-purity metals as precision temperature standards*, Temperature, its measurement and control in science and industry, Vol. 3, pp 185-

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(5) Berry, R.J., *Effect of oxidation on Pt resistance thermometry*, Metrologia 16, p 117 (1980); also *Evaluation and control of platinum oxidation errors in standard platinum resistance thermometers*, Temperature, Its Measurement and Control in Science and Industry, Vol 5, Schooley ed., Am Inst Physics (1982)

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(9) McAllan, J. V., *Sensitive temperature measurements on pure metals during melting and freezing*, J. Crystal Growth, 12, North Holland (1972); also *Practical reference temperatures using melting point techniques*, J. Phys. E., The Institute of Physics, London (1982)

TABLE 1

FIXED POINTS OF THE ITS-90 AND THEIR PRESSURE COEFFICIENTS

Substance	Equilib.	Pressure effects-	
		Atmosph. mK per std. atmos	Hydrostat. Mk per M liquid head
Argon (tp)	13.8033	*	3.3
Mercury (tp)	234.3156	*	7.1
Water (tp)	273.16	*	-0.73
Gallium (mp)	302.9146	-2.0	-1.2
Indium (fp)	429.7485	4.9	3.3
Tin (fp)	505.078	3.3	2.2
Zinc (fp)	692.677	4.3	2.7
Aluminum (fp)	933.473	7.0	1.6
Silver (fp)	1234.93	6.0	5.4

* indicates that a triple point is independent of atmospheric pressure, since it is defined as an

equilibrium under its own vapor pressure.

TABLE 2
PHASE DIAGRAM PARAMETERS

SOLVENT	SOLUTE	m_L m_S		k_o	R	D_L
		10^{-4}C/ppm				
Zn	Cd	-1.9	-77	0.02	75	<0.1
	Cu	2.5	1.7	1.5	1	1
	Fe	-4.1	-33	0.12	27	0.1
	Pb	-1.9	large		large	<0.1
	Sn	-2.4	large		large	<0.1
	Ti	-1.9	large	0.1	large	<0.1
Sn	Bi	-1.6	-4.4	0.36	3	1
	Ca	-20	large		large	<0.1
	Cu	-7.1	large		large	<0.1
	Fe	-33	large		large	<0.1
	In	-2.3	-3.5	0.66	1	1
	Ni	-5	large		large	<0.1
	Pb	-1.3	-20	0.06	19	0.1
	Sb	1.6	1.3	1.2	<1	1
In	Ag	-5	large		large	0.1
	Bi	-2.5	-4.1	0.61	2	<0.1
	Cd	-1	-2	0.5	1	2
	Cu	-10	large		large	0.1
	Pb	<-1	<-1	1.0	<1	2
	Zn	-70	-120	0.6	50	<0.1

Key: m_L = liquidus slope, m_S = solidus slope, k_o = equilibrium distribution coefficient, R = equilibrium alloy melting range, D_L = detection limit (wt ppm) of platinum resistance thermometer.

This table shows the depressing or elevating effects of 1ppm of various solutes on solvents used in ITS-90. It is after McLaren (see Footnote 6). The authors would be pleased to receive information which would extend the table to include mercury, gallium, aluminium and silver.

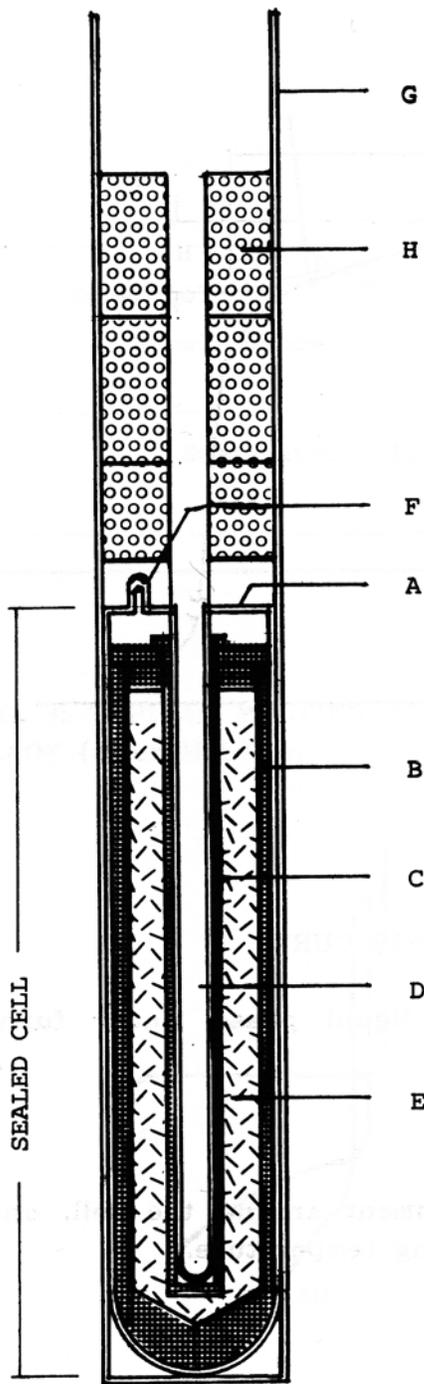


FIG. 1(A)

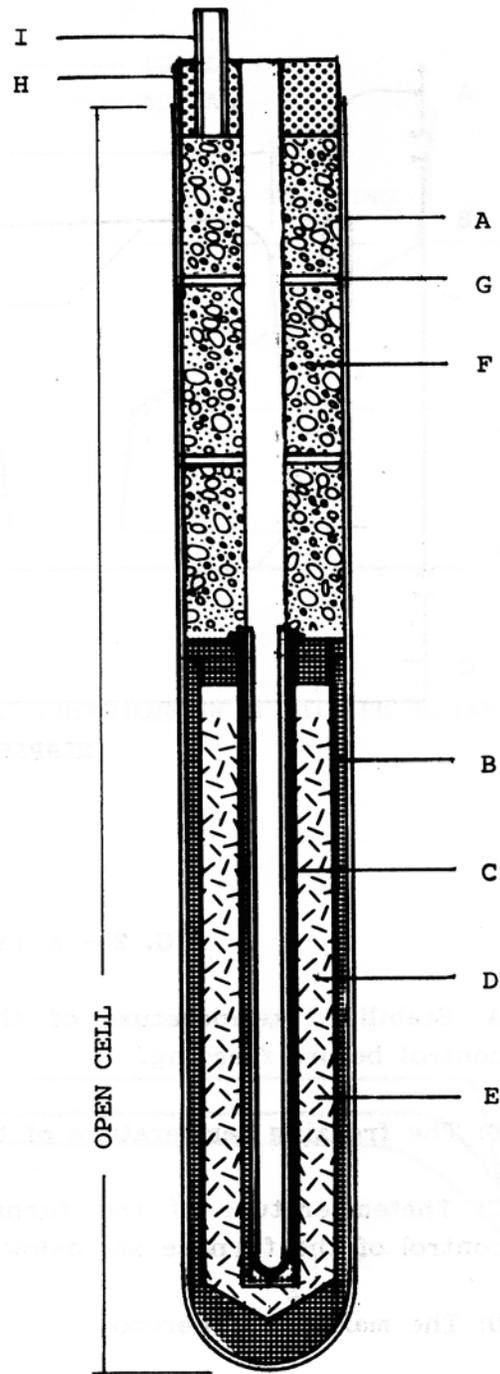


FIG. 1(B)

FIG 1(a): A, quartz envelope; B, graphite crucible; C, graphite sheath over thermometer well; D, thermometer well; E, pure metal; F, fill gas seal-off; G, inconel can for removal of cell; H, insulating bricks. FIG 1(b): A, quartz envelope; B, graphite crucible; (C). graphite sheath over thermometer well; D, thermometer well; E, pure metal; F, thermal insulation; G, graphite heat shunt; H, stopper; I, port for evacuation and fill with blanking gas. (Be careful evacuating over zinc; vapor pressure is $\approx 10^{-4}$)

atmosphere at the melt temperature).

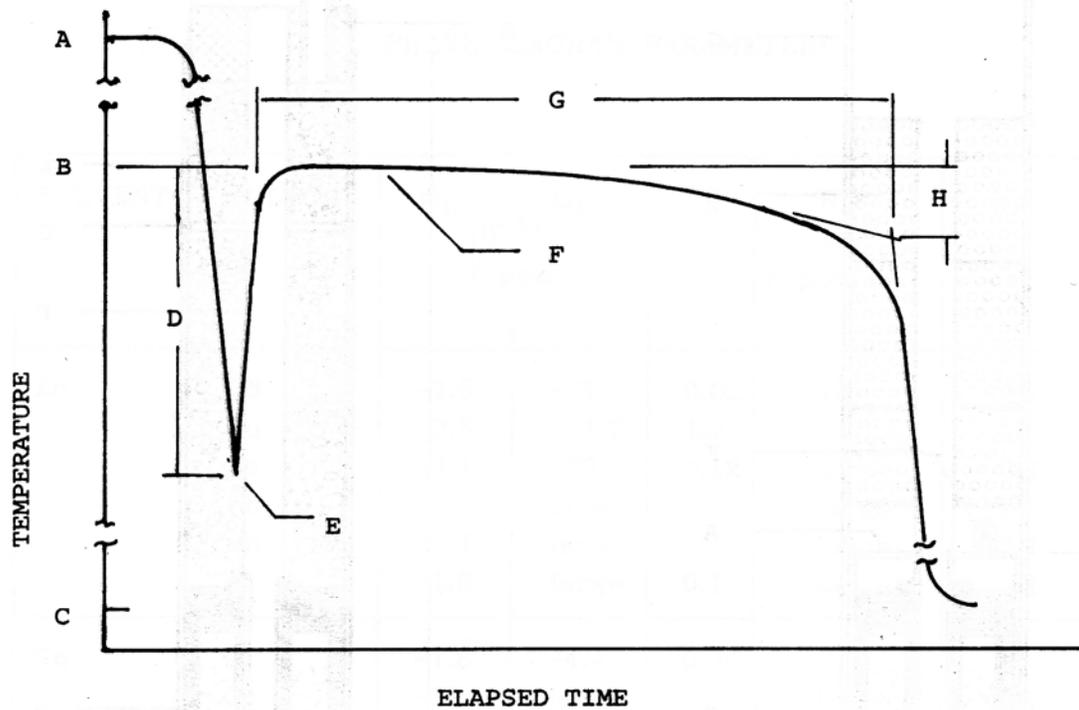


FIG. 2 - A TYPICAL FREEZE CURVE

- A: Stabilized temperature of the cell, in liquid phase, under furnace control before freezing.
- B: The freezing temperature of the cell.
- C: The temperature of the furnace environment around the cell, under control of the furnace set below the freezing temperature.
- D: The maximum supercool.
- E: The onset of recalescence (nucleation).
- F: The freezing plateau.
- G: The total freezing time.
- H: The freezing range.

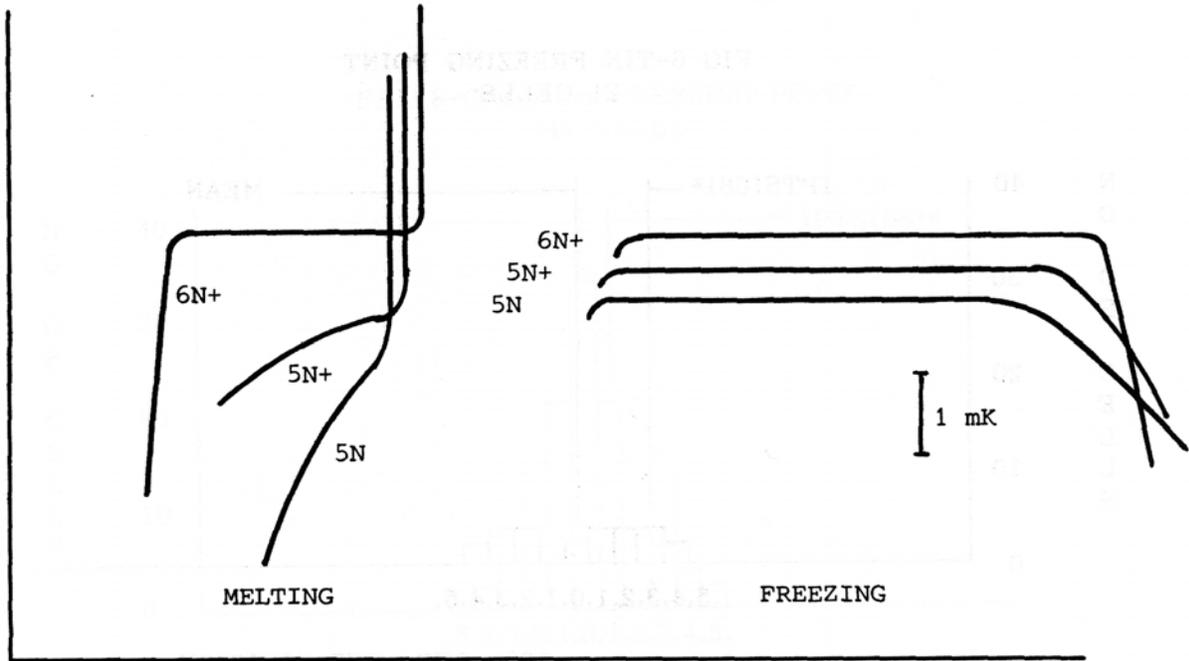


FIG. 3 - TYPICAL MELTING AND FREEZING BEHAVIOUR OF A DILUTE BINARY ALLOY (after McLaren).

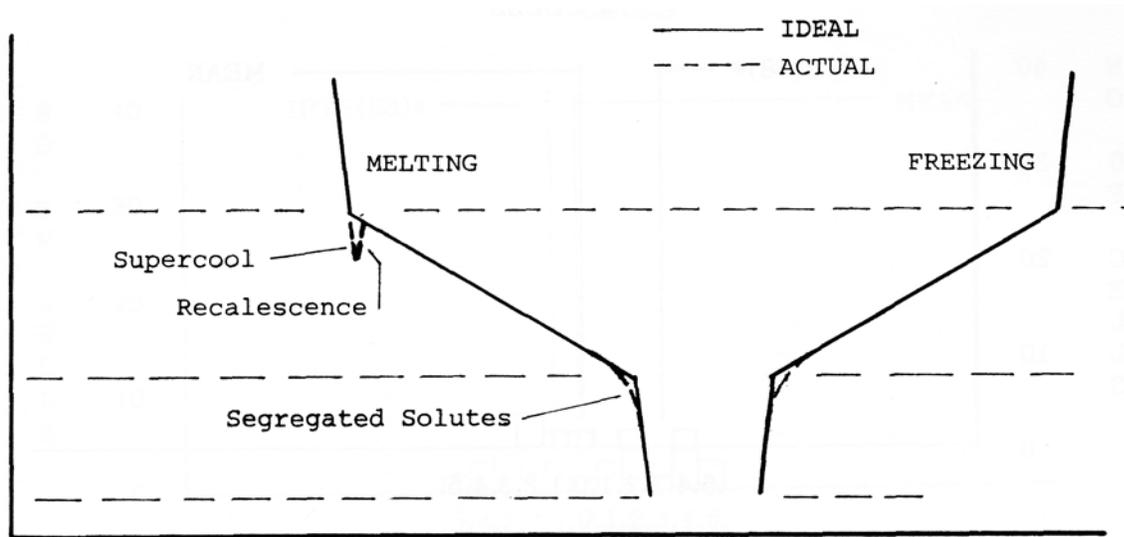
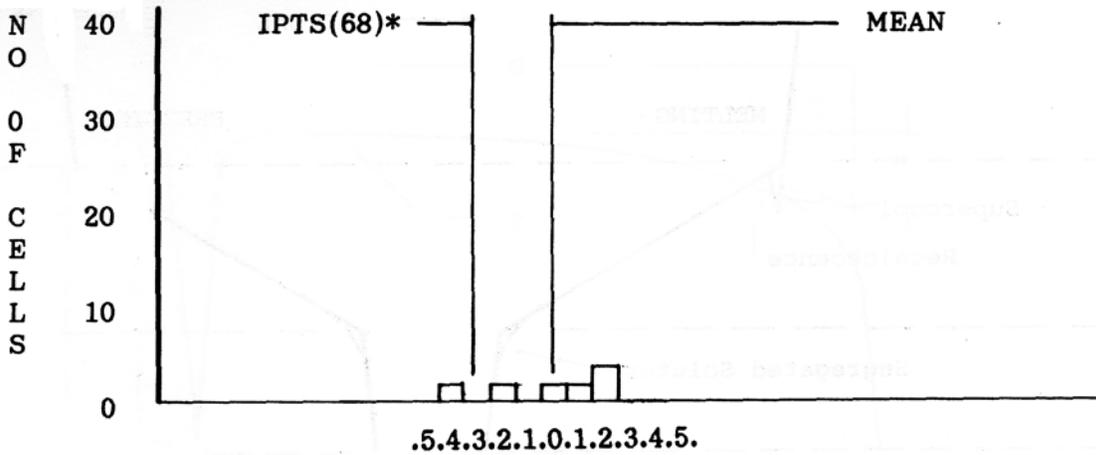


FIG. 4 - TYPICAL MELTING AND FREEZING CURVES OF METALS OF THREE LEVELS OF PURITY

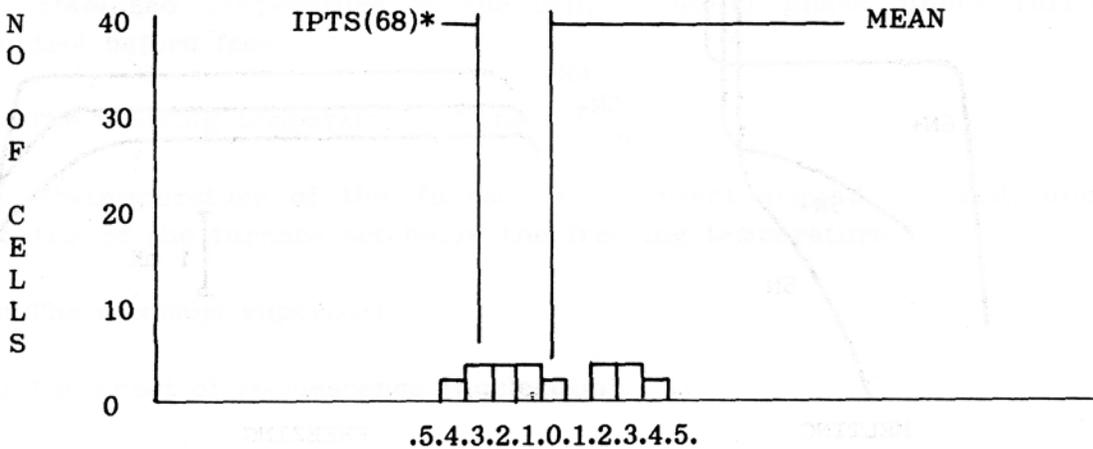
FIG 5-INDIUM FREEZING POINT
6 CELLS



MEAN OF 6 CELLS 156.6348°C
 MAXIMUM CELL 156.6350°C
 *IPTS(68) TEMP 156.6345°C
 CORRECTED FOR HYDROSTATIC HEAD

1 STD DEVIATION 0.0002
 MINIMUM CELL 156.6344°C
 BIAS VS IPTS* +.0003°C *IPTS(68) TEMP IS

FIG 6-TIN FREEZING POINT
21 CELLS

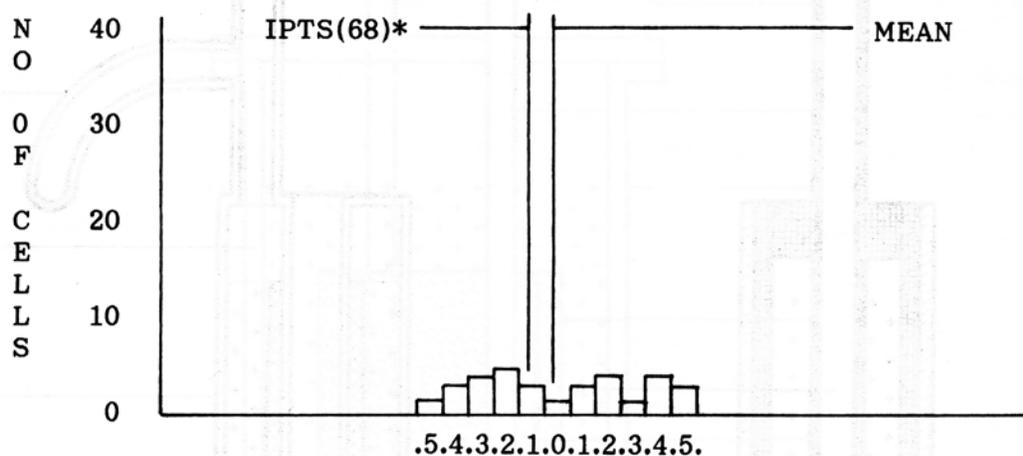


100 pDEG PER DIVISION

MEAN OF 21 CELLS 231.9688°C
 MAXIMUM CELL 231.9692
 *IPST(68) TEMP 231.9685
 TEMP IS CORRECTED FOR HYDROSTATIC HEAD

1 STD DEVIATION 0.0003
 MINIMUM CELL 231.9681°C
 BIAS VS IPTS* 0.0003°C *IPTS(68)

FIG.7-ZINC FREEZING POINT
23 CELLS



100 pDEG PER DIVISION

MEAN OF 23 CELLS 419.5805 °C

MAXIMUM CELL 419.5810°C

*IPTS(68) TEMP 419.5804°C

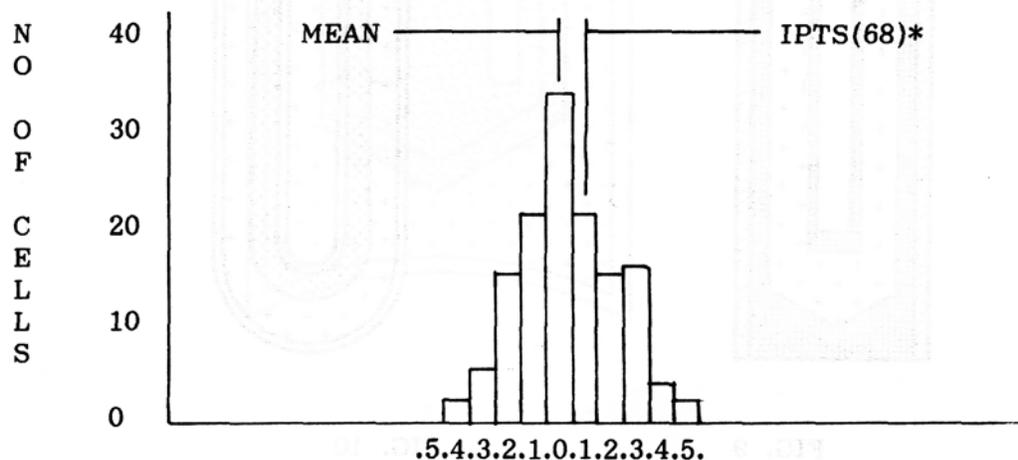
IS CORRECTED FOR HYDROSTATIC HEAD

1 STD DEVIATION 0.0003

MINIMUM CELL 419.5800°C

BIAS VS IPTS* 0.0001 *IPTS(68) TEMP

FIG. 8-GALLIUM FREEZING POINT
141 CELLS



100 pDEG PER DIVISION

MEAN OF 141 CELLS 29.7715°C

MAXIMUM CELL 29.7720°C

*IPTS(68) TEMP 29.7716°C

IS CORRECTED FOR HYDROSTATIC HEAD

1 STD DEVIATION 0.0002

MINIMUM CELL 29.7710°C

BIAS VS IPTS* -.0001°C *IPTS (68) TEMP

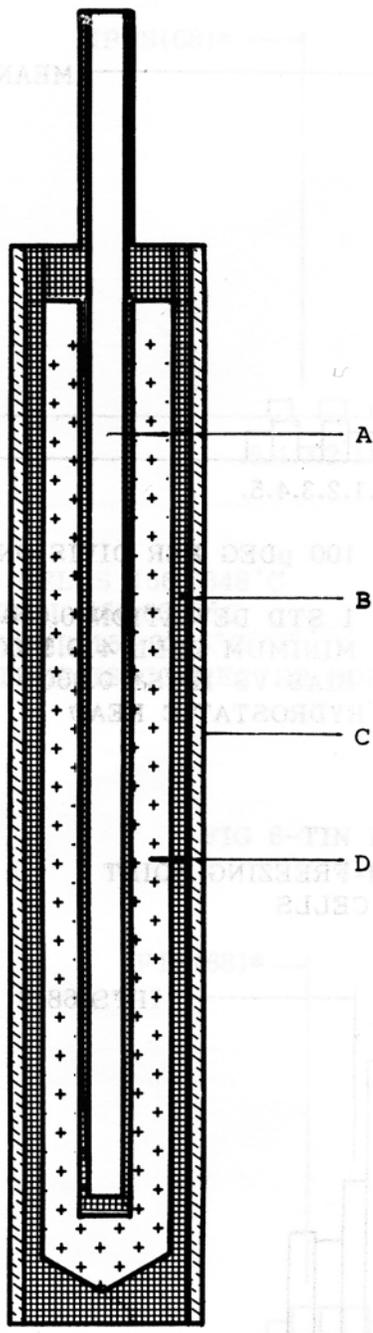


FIG. 9

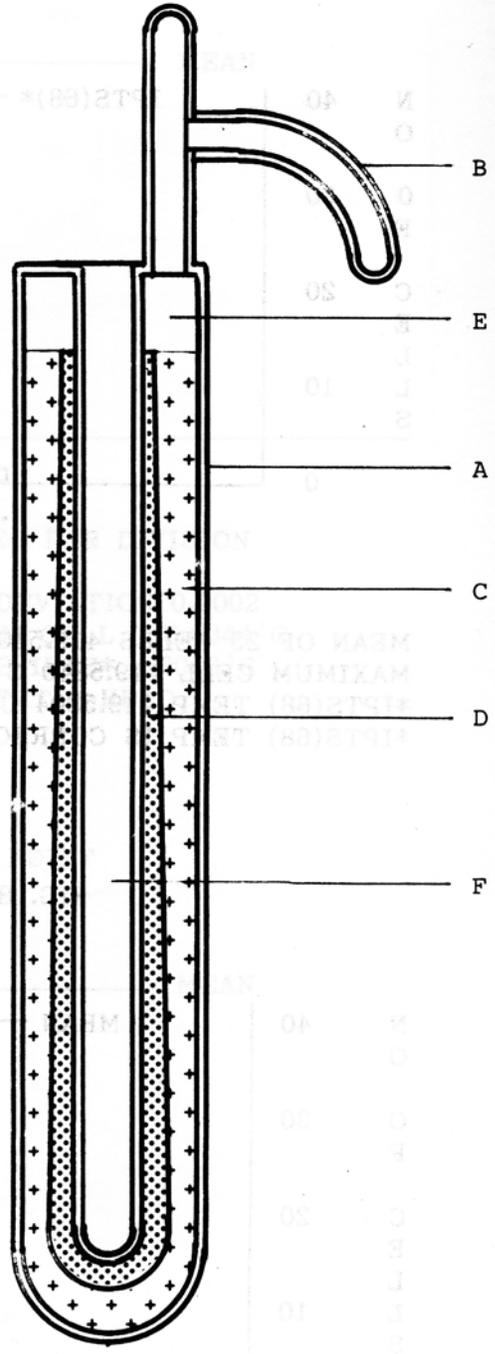


FIG. 10

FIG. 9: A type of gallium cell. A, thermometer well; B, cell wall; C: aluminum sleeve; D, high-purity gallium. A and B are Teflon.

FIG 10: A type of water triple point cell. A, borosilicate glass cell; B, handle; C, liquid water; D, solid water (ice); E, water vapor; F, thermometer well.

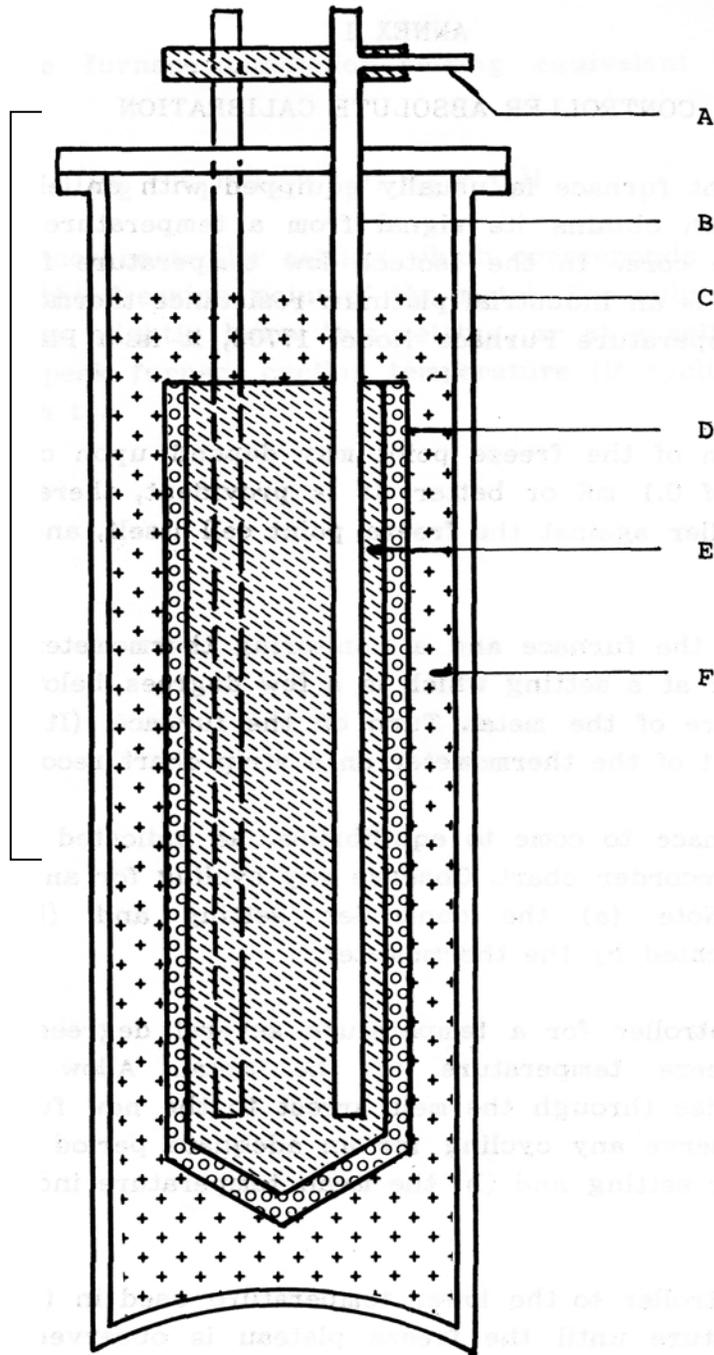


FIG. 11: Comparator for the argon point. A, manifold to surround the thermometers with helium gas (optional); B, thermometer well (three provided); C, stainless steel dewar; D, porous blanket; E,

tellurium copper equalizing block; F, liquid nitrogen cryogen. Not shown: fill port, level control, pressure reliefs.

ANNEX 1

CONTROLLER ABSOLUTE CALIBRATION

The freezing-point furnace is usually equipped with an electronic control system, which obtains its signal from a temperature sensor built into the furnace core. In the Isotech low temperature furnace Model 17701 the sensor is an industrial platinum resistance thermometer. In the Isotech High Temperature Furnace Model 17702, it is a Platinel thermocouple.

Proper realization of the freeze point may depend upon controller set-point accuracy of 0.1 mK or better. It is provident, therefore, to calibrate the controller against the freeze point cell itself, and this can be done as follows:

- 1: With a cell in the furnace and a monitoring thermometer in the well, set the controller at a setting which is a few degrees below the nominal freeze temperature of the metal. Turn on the furnace. (It is helpful to record the output of the thermometer on a strip chart recorder.)
- 2: Allow the furnace to come to equilibrium, as indicated by the thermometer or the recorder chart. Observe any cycling for an adequate period of time. Note (a) the controller setting and (b) the mean temperature indicated by the thermometer.
- 3: Reset the controller for a temperature several degrees higher than the nominal freeze temperature of the metal. Allow the furnace temperature to rise through the melt arrest to the new furnace control temperature. Observe any cycling for an adequate period of time. Note (a) the controller setting and (b) the mean temperature indicated by the thermometer.
- 4: Reset the controller to the lower temperature used in (2). Follow the drop in temperature until the freeze plateau is observed. (Follow instructions for the specific metal on the technique of initiating the freeze plateau.) Note the temperature indicated by the thermometer.

- 5: Using the following notation:

C_1 and t_1 = the controller setting and the temperature, respectively, at the furnace setting below the freeze temperature.

C_2 and t_2 = the controller setting and the temperature, respectively, at the furnace setting above the freeze temperature.

T_3 = the temperature measured on the freeze plateau.

- 6: Calculate the furnace controller setting equivalent to the freeze plateau:

$$CS = C_1 + \left\{ \left[\frac{(c_2 - c_1)}{(t_2 - t_1)} \right] (t_3 - t_1) \right\}$$

This is the furnace controller setting which corresponds to the furnace temperature at the freezing point of the metal. For subsequent freezes, set the controller slightly below this setting, or at a setting which assures that the peak furnace cycling temperature (if cycling is present) will be less than t_3 .

ANNEX 2

The freezing and melting curves of freeze point cells are largely dependant upon their recent history. The charts below illustrate this, and indicate how the best method of working with a cell may be derived and developed.

"Normal freezing" refers to undisturbed natural solidification, whilst "induced freezing" refers to a technique that is used -to shorten the recalescent period. The latter is effected by freezing a thin mantle 'of metal onto the axial thermometer well after nucleation has commenced; this releases sufficient latent heat to raise the metal temperature to the liquidus temperature. The graphs are after McLaren (Footnote 6).

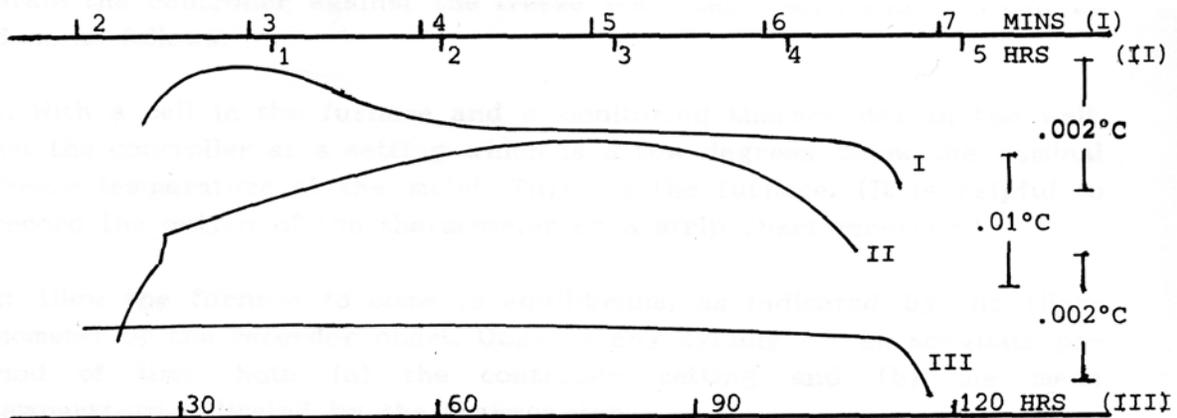


FIG. A: Typical freezing curves of high-purity indium: I, very fast normal; II, very slow normal; III, slow induced

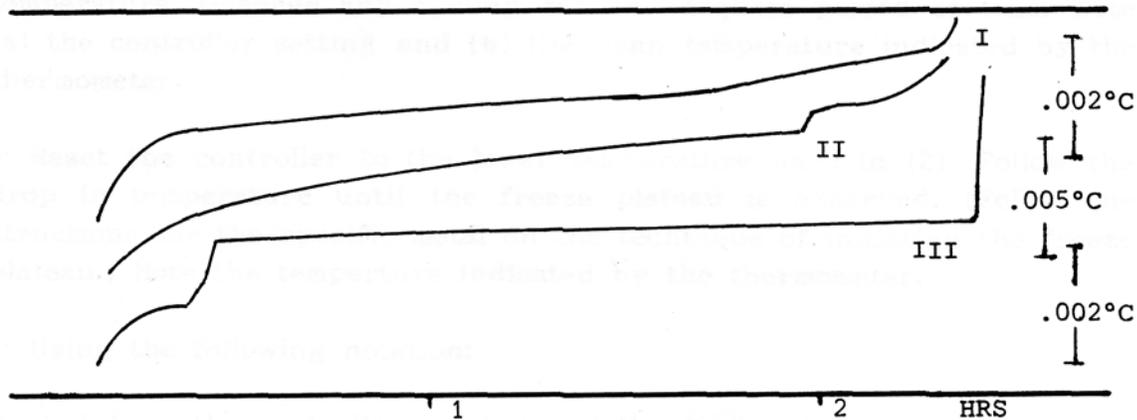


FIG B: Typical melting curves of high-purity indium following: I, very fast normal freeze; II, very slow normal freeze; III, slow induced freeze.

STANDARD PLATINUM RESISTANCE THERMOMETER CALIBRATIONS
ON THE ITS-90 TEMPERATURE SCALE:
HOW TO SPECIFY AND ORDER THEM

Members of the NCSL ad-hoc Committee on ITS-90

ABSTRACT

The new International Temperature Scale of 1990 (ITS-90) has replaced the International Practical Temperature Scale of 1968 (IPTS-68). Purchasers of calibration services or users of in-house calibration laboratories must specify whether an ITS-90 or IPTS-68 calibration is required, and the desired temperature range of the calibration.

Practical temperature measurement in science and virtually all industry is related to a Temperature Scale established by the International Committee on Weights and Measures (CIPM). The International Temperature Scale has been modified from time to time, to include new technological demands and advances, and to take into account new developments in instrumentation and new determinations of thermodynamic temperatures. Thus the International Practical Temperature Scale of 1948 (IPTS-48) was replaced by the International Practical Temperature Scale of 1968 (IPTS-68), and on January 1, 1990, IPTS-68 was replaced by the International Temperature Scale of 1990.

The new scale changes the way thermometer and temperature system calibrations are specified and ordered, from national laboratories, calibration services, and in-house calibration facilities.

Among the features of the ITS-90 that require a new approach to calibration orders are the following:

(a) The ranges over which the Standard Platinum Resistance Thermometer (SPRT) is the prescribed interpolation instrument of the Scale have been extended from the 630.74 °C of IPTS-68 to 961.78°C on ITS-90, (b) the platinum/platinum-rhodium thermocouple has been eliminated as an interpolation instrument, and (c) most values assigned to the calibration fixed-point temperatures have been changed.

No single SPRT can be expected to cover the complete range for which SPRTs are the stipulated interpolation instrument. Capsule thermometers will continue to be used for the cryogenic ranges. SPRTs having a resistance of 25.5 Ω at 0°C, Pyrex sheaths and mica insulation can be damaged by exposure to temperatures higher than 500°C, and quartz sheathed SPRTs with mica insulation should not be used higher than 650°C. New thermometers, usually 0.25 Ω at 0°C, are required for the upper range. Also, although the standard thermocouple has been eliminated as an accepted interpolating instrument, it is anticipated that most industrial temperature measurements in the upper temperature ranges will continue to be made with industrial-grade thermocouples. New tables for all letter-designated thermocouples are in preparation at NIST. These tables will be in terms of the new ITS-90 and the new international volt adopted on January 1, 1990.

There is another major difference which needs to be recognized in specifying calibrations. IPTS-68 provided only three choices of temperature range to those ordering calibration services for SPRTs from a calibration laboratory. These were -200° to 0°C, 0° to either 500°C or 650°C depending upon the construction of the thermometer, or a combination of the ranges above and below 0°C. ITS-90 provides 11 SPRT ranges, and the purchaser of a service must specify the range he wishes. There are several advantages to this change: (1) it is no longer necessary to pay for calibration over a larger range than required by the user (2) ranges are constructed with a calibration fixed point at both ends, so that long extrapolation and consequently increased uncertainty at the temperature extremes is eliminated.

The 11 new ranges for SPRTs are as follows:

- #1. 13.8033 K (-259.3467°C) to 0.01°C
- #2. 24.5561 K (-248.5939°C) to 0.01°C
- #3. 54.3584 K (-218.7916°C) to 0.01°C
- #4. 83.8058 K (-189.3442°C) to 0.01°C
- #5. 0°C to 961.78°C
- #6. 0°C to 660.323°C
- #7. 0°C to 419.5270°C
- #8. 0°C to 231.928°C
- #9. 0°C to 156.5985°C
- #10. 0°C to 29.7646°C
- #11. -38.8344°C to 29.7646°C

An SPRT may be calibrated over any range or over a combination of any range from 1 through 4 and 5 through 10, depending upon the suitability of the type of SPRT to be calibrated. The required range(s) MUST BE SPECIFIED in an order for an ITS-90 calibration. NIST now calibrated only to ITS-90, but some commercial calibration services will be prepared to furnish calibrations in the obsolete IPTS-68 Scale, if requested.

Orders for calibration of industrial thermometers and thermometer systems on the ITS-90 also require clear and distinct specification of the calibration range. These ranges need not be chosen from the 11 ranges of the ITS-90 if they are calibrations to be done by comparison with a standard thermometer, rather than against the fixed points of the Scale. However the calibration laboratory must be told the low and high limits of temperature desired. For systems that include a readout, the same information is necessary unless the readout defines the calibrated range. The ranges of the ITS-90 form a convenient list for choice, and if industry would also adopt these ranges, charges for comparison calibrations might be less than if ranges were arbitrarily specified.

One further note: whenever there is a change of this sort, there is an opportunity for confusion and error. In any work that reports a temperature, it should be made clear in the text or in a footnote whether the value reported is on IPTS-68, ITS-90, or some other Scale.

(This discussion was prepared and reviewed by the following:

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INTERNATIONAL EQUIVALENCE OF THERMOMETER CALIBRATION, TESTING AND CERTIFICATION

by John P. Tavener

INTRODUCTION: International Metrology Equivalence is the recognition of standards and methods of measurement of one country by another. Two levels of agreement exist internationally:

- 1: Agreements between national laboratories
- 2: Agreements recognizing the equivalence of certificates issued by accredited secondary and industrial laboratories.

The International Laboratory Accreditation Conference (ILAC) aims to harmonize criteria and procedures for laboratory accreditation, as required by the General Agreement on Tariffs and Trades (GATT) and to facilitate international acceptance of calibration measurements and data.

1: AGREEMENTS BETWEEN NATIONAL LABORATORIES

A: U.K. (NPL) and U.S.A. (NIST)

A document entitled "The recognition of equivalence of the National Standards of the U.K. and the U.S.A. for the measurement of temperature" was signed on October 14, 1986. This needs updating to ITS-90; however since interpolation uncertainties have been reduced by the new Scale, we can confidently predict that any changes may be a reduction of uncertainties.

Temperature range

Uncertainty of Equivalence

0.5 K to 450°C	0.002 K
450°C to 630°C	0.006 K
630°C to 1500°C	0.3 K
1500°C to 2200°C	1.0 K

B: U.S.A. (NIST) and Australia (CSIRO) - October 1985

1K to 273.5 ° C	0.003 K
0°C to 450 ° C	0.002 K
450°C to 630° C	0.006 K
630°C to 1064° C	0.3 K
1064°C to 2300° C	0.1%

C: U.S.A. and Italy - October 1985

13.8 K to 303 K	0.0005 K at defining fixed points and the freezing point of gallium. 0.003 K between fixed points using SPRTs
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2: MUTUAL RECOGNITION AGREEMENTS

In the United Kingdom, NAMAS (The National Measurement Accreditation Service) a service of the National Physical Laboratory (NPL) has negotiated agreements between its equivalent organizations in many countries; thus each of the countries listed below recognizes the results of a NAMAS calibration and accepts the certificate as if it had been issued by own of its own accredited laboratories. (There is no equivalent to such an accredited calibration laboratory in the United States).

Country,	Accrediting	Calibration	Testing
Australia	NATA	1985	1985
Germany	DKD	1981	
France	BNM, RNE	1988	1986
Hong Kong	HOKLAS	1989	1989
Italy	SIT	1982	
Netherlands	NKO	1988	
New Zealand	Telarc	1985	1985
Sweden	SMO	1988	
Switzerland	SCS	1989	

Copies of the documents describing these mutual agreements are available upon request.

ISOTECH IS AN ACCREDITED NAMAS LABORATORY

IMPROVEMENTS IN METROLOGICAL APPARATUS

Isothermal Technology Ltd. Staff

This is the first of an occasional series keeping the user of Metrological Apparatus up-to-date with latest improvements.

1: THE WATER TRIPLE POINT MAINTENANCE BATH

INTRODUCTION: The triple point of water, 0.01°C by definition, is easily realizable within 0.00015°C. It is the most fundamental fixed point in thermometry.

A prominent use is in ensuring the calibrations of thermometers such as SPRTs. If a thermometer repeats its last water triple point resistance within acceptable limits it has almost certainly not shifted, and the cost of recalibration may be avoided. Also, a water triple point measurement made immediately after a thermometer has been transported will assure that it has not been damaged. It is good practice to convey the water triple point measurement to a control chart whenever that point is measured.

Also, for the most precise work, it is advisable to use the ratios of the calibration report ($W = R_t/R_{wtp}$) rather than the resistances.

DESCRIPTION: Isothermal Technology produce a bath, ITL Model 18233, developed over 8 years by ourselves and predecessors, which will maintain up to 4 water triple point cells. 2 tubes for pre-chilling thermometers are provided. The bath temperature may be set with resolution and accuracy of 1 mK and will keep cells at the water triple point for 4 to 6 weeks or with a little attention much longer. The bath is caster-mounted and completely self-contained. It holds 35 liters of water. It is cooled by 4 Peltier modules, which extract heat from the water and transfer it to heat sinks. Filtered air is blown over the heat sinks to void the unwanted heat.

IMPROVEMENTS: Two improvements have been introduced recently which help the performance of the bath.

Extra insulation and reflecting foil are now used to reduce losses.

An analysis has been made of the air flowing over the heat sinks, and a second fan has been incorporated.

The net result of these changes is that the bath now cools from 20°C to 0.01°C in less than 20 hours compared to the previous 40 hours, and that the bath is more tolerant of changes in ambient temperature. It will work with higher ambients than previously, and cools back to 0.01°C faster after the lid has been lifted allowing heat to be absorbed.

(Isotech does not at this time offer the water triple point cells themselves, but will recommend sources).

2: AYRIES BATH

INTRODUCTION: Fluidized sand baths for higher temperatures have been available for many years. In theory, alumina powder can be levitated at any temperature from liquid nitrogen to over 1600°C! In practice, the concept has proven disappointing, because of large temperature gradients and because powder leaks from the bath into the laboratory environment.

Some 17 years ago, in one of the Electricity Generating Board's Laboratories, attempts were made to resolve these problems. The result of this research is the Isotech Ayries Bath.

DESCRIPTION: The Ayries Bath incorporates three patented features worthy of explanation.

In most fluidized baths, air flows through a porous plate at the bottom of the bath and fluidizes the powder above the plate. Previously, as the temperature increased, so did the flow, according to the gas law

$$P \cdot V = M \cdot R \cdot T$$

where P = pressure, V = volume, M = mass, R is a constant and T is kelvin temperature.

If the flow rate could be set at the working temperature, flow would be constant. The first patent incorporated in the Ayries Bath relates to the flow restriction. Air from a compressor (about 60 l at 1/2 bar) is warmed to the working temperature of the bath and then passed through the flow setting orifice, ensuring an almost constant rate of flow regardless of temperature. (The air flow increase slightly because of a small change in the diameter of the orifice with temperature).

The powder, fluidized by the air, takes on many of the properties of a liquid. Because the powder effectively hovers in the air flow, large thermals exist in a conventional fluidized bath. The second Ayries patent provides for a specially-shaped baffle by means of which the powder flow is forced up and past the furnace heaters, and then down the center of a parallel tube to the bottom of the bath, in a laminar flow pattern. Sensors placed anywhere in the central tube are at the same temperature within ± 0.2 °C.

Air leaving the bath is laden with fine powder, and, unless a separate room is provided for

use of the bath, the powder puts a white layer of alumina all over the calibration laboratory. The third patent relates to this exhaust air. Above the bath is a stainless steel housing containing a porous ceramic filter. The hot, powder-laden air passes through the filter, depositing its powder on the filter wall. As the deposit increases, its own weight causes it to fall back into the bath. What passes into the laboratory environment, or can be ducted outdoors, is powder-free air.

This bath was developed in the early 1980s for the automatic comparison calibration of thermocouples, where $\pm 0.2^\circ\text{C}$ uniformity is more than adequate. However we wish now to use the bath for closer calibration of platinum resistance thermometers, and also for cycling fixed-point cells. Two improvements have recently been incorporated.

IMPROVEMENTS: The original means of calibration was via a multi-tube assembly which bolted to the top of the bath. For fixed-point calibration, this was replaced by a single-well assembly, which proved to have gradients of only 0.1° to 0.2°C , and provided fixed-point plateaux of 20 to 30 hours. The same single tube was left in the bath and an equalizing block holding 1 SPRT and 4 test thermometers was built into the tube. This provides temperature distribution and stability better than 0.01°C .

The second improvement relates to air filtration. If too much air is allowed to flow through the bath, the filter can clog and the pores in the ceramic fill with powder. While cleaning a filter normally involves nothing more than a sharp rap with a wood or plastic rod, it can be a nuisance, and possibly interrupt a calibration run.

We have therefore reversed the flow through the filter so that the air now flows from the outside to the inside surface. Three improvements have resulted. First, the outer surface area of the filter is approximately double that of the inside, so the flow is effectively halved, which reduces clogging. Second, the hot powder-laden air is cooled by the stainless outer wall of the filter housing, further reducing air flow. Third, the outside surface of the filter is easier to clean than the inside surface. The result is that filter cleaning is virtually eliminated. These improvements can be retro-fitted on all existing Ayries baths.

3: LOW TEMPERATURE FIXED-POINT FURNACE, ITL 17701

The Low Temperature Furnace incorporates a massive aluminium bronze furnace core, heated uniformly along its length to create an isothermal zone in which freeze point cells (specifically indium, tin and zinc) can be melted and frozen. Whilst creating excellent conditions for indium and tin, it was found to give plateaux at the zinc point which were shorter than desirable.

A number of small changes have been made, with the result of more than doubling the duration of zinc plateaux. These include improved insulation around the top of the furnace, reducing top end loss from the block; an assembly placed above the zinc cell to reflect heat and reduce possible convection currents, and a heat sink to keep the thermometer handle cool, for comfort and to minimize possible thermal emfs from the platinum-to-copper junctions within the head.

These improvements can be added to existing Low Temperature Furnaces, either by the owner, or by purchase of a conversion kit from Isotech.

Isotech is currently building a special version of this furnace in conjunction with PTB-Braunschweig, which incorporates end heaters to virtually eliminate axial gradients. This will be reported on as results become available.

EVALUATION OF THE GALLIUM MELTING POINT

BY A TWO CELL INTERCOMPARISON

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ABSTRACT

The International Temperature Scale of 1990 (ITS-90) recognizes the melting temperature of pure gallium as one of its defining fixed points. In an effort to determine the practical limits of accuracy in realizing this point, an intercomparison of two commercially available gallium melting point cells was undertaken.

Each cell was measured six times along its melt plateau, and a triple-point-of-water measurement made immediately afterward. A Standard Platinum Resistance Thermometer (SPRT) recently calibrated at the National Institute of Standards and Technology (NIST) on the International Practical Temperature Scale of 1968 (IPTS-68) was used throughout these measurements. It will be shown that, although agreement to better than 0.2 mK was achieved between the cells, modifications in measurement technique are possible which have the potential for increasing measurement accuracy more than twofold.

INTRODUCTION

The Standard Platinum Resistance Thermometer (SPRT) is recognized as the primary interpolation instrument of the IPTS-68 from 13.8 K to 630.74 °C, and of the ITS-90 from 13.8 K to 961.78 °C. The structure of the ITS-90 lends great significance to the gallium point. It is used to determine the suitability of a given SPRT as a primary standard, as well as defining the end point of a range which includes 0°C, and thus links the two reference functions which define the generalized behavior of SPRTs.

Gallium can be refined to very high purities, upward of 99.9999% (six 9's). This ensures that the temperature of phase change equilibria (e.g. a melting point) can be realized very accurately and reproducibly. The melting point of gallium is used, instead of the freezing point used with other met-als, due to the large supercooling necessary (as much as 70°C) to

initiate a gallium freeze, and also, because unlike other metals, gallium expands as it freezes, causing unwanted pressure effects on the freeze temperature. The temperature assigned to the melting point of gallium on the ITS-90 is 29.7646°C. (On the IPTS-68, gallium had a provisional value of 29.7720°C,

and was not a defining point)⁽¹⁾. The proximity to room temperature makes the melt relatively easy to realize, and provides a reliable reference in a region of great importance to medical, pharmaceutical, ecological, geothermal and oceanographic applications.

Automatic systems for realizing this point are readily commercially available. Thus, in terms of application and ease of use, the melting point of gallium is among the most practical of all used for thermometer calibration. As such, an evaluation of the limits of accuracy typical to its realization is of great value.

APPARATUS AND EXPERIMENT DESIGN

The apparatus consists of a self-contained module in which the cell resides (a YSI Model 17402 Gallium Melting Point Standard).⁽²⁾ The cell environment is controlled to the temperatures necessary for initiating the melt and producing long-lived melt plateaux. A gallium cell with a fully frozen sample is placed in the module, and the unit is switched on. Separate indicator lights show when the cell is warming, and when it is ready for use.

An SPRT (YSI Model 8163Q)⁽³⁾ is placed in the thermometer well of the cell, to which a small amount of water has been added as an exchange medium. SPRT behavior is monitored using an ASL F-18 Automatic AC-resistance bridge.⁽⁴⁾ Data is gathered through the IEEE-488 interface of an instrument-controlling computer. The bridge ratio is recorded at a preselected sample rate, and is displayed graphically as a function of real time. The mean ratio can be calculated at any time during the measurement process.

Triple point of water cells are maintained in a bath designed solely for that purpose (YSI Model 18233 Water Triple Point Maintenance Bath)⁽⁶⁾. The gallium apparatus, bridge, and triple point bath are placed in close proximity. Thus, measurements can be made easily without disturbing any electrical connections and with minimum handling of the SPRT. (See Fig. 1).

The gallium cell is removed from the melt module when the plateau has ended, and is placed in an ice bath to facilitate rapid solidification of the sample. The cell is ready to be used again in about 5 hours.

The timing of the experiments is arranged so that the gallium melt plateau (which can last 12 hours or more) occurs overnight. Immediately after a run is finished, a triple point of water value is obtained using the same data-taking procedure described above. This scheme allows one set of measurements to be completed each day.

DATA AND INTERCOMPARISONS

Each of two gallium cells was measured six times along a melt plateau. An SPRT which has been calibrated recently at NIST on the IPTS-68 was used for these measurements. The recent SPRT calibration serves to attest to the integrity of the SPRT used as a primary standard and to allow for temperature intercomparisons to be made. (No statement will be made in this paper as to the absolute or thermodynamic temperature of the gallium melting point).

The measured resistance ratios represent the "hotness" of the phase equilibrium, and so only the temperature value assigned to these ratios is Scale-dependent. Therefore, differences between ITS-90 and IPTS-68 have no direct effect on the results, except with respect to the temperature value assigned to this point. Thus IPTS-68 temperatures, as determined by NIST calibration, will be reported for the gallium melting point.

The gallium melting curves exhibit a typical temperature span of less than 0.5 mK. Therefore, choosing the bridge ratio value which is truly representative of the melting temperature can be somewhat arbitrary. The plateau has a fairly well-defined signature, and typically the value along its flattest portion can be used without undue concern. A typical melting curve is shown in Fig. 2.

It can be seen from this graph that a stable value is not reached until some three hours into the melt plateau. A slow and steady upward drift is seen for the next seven hours as the liquid-solid interface advances toward the thermometer well from the outside of the cell. On the plateau shown in Fig. 2, an extremely flat region can be seen for the last two hours. There is a preponderance of liquid metal and a homogeneous distribution of impurity in the cell at this time. Small heat fluctuations are completely absorbed with no apparent change in temperature, and so the degree of temperature stability is very high.

The ratio value assigned to each melt plateau was taken as the mean value along the portion of the melting curve which appears to represent true phase equilibrium. Table 1 shows the mean value of the ratio indicated by the resistance bridge, and the melting range, for each of the plateaux obtained for these two cells:

TABLE 1: MEAN AND MELTING RANGE

Run # (SN 139)	SPRT mean bridge ratio along plateau	Melting Range (mk)
1	0.28554160	0.15
2	0.28554152	0.33
3	0.28554150	0.32
4	0.28554151	0.35
5	0.28554166	0.24
6	0.28554161	0.32
SN 140		
1	0.28554171	0.38
2	0.28554171	0.33
3	0.28554181	0.32
4	0.28554185	0.34
5	0.28554185	0.32

Following the completion of each melt plateau, the SPRT was measured at the water triple point (TPW). The triple point value is determined by monitoring the bridge ratio graphically in real time, as shown in Fig. 3. It is readily seen when the SPRT reaches a stable state within the cell, and measurements can begin. Very little uncertainty is associated with the realization of the triple point state as such; however, the ultimate determination of the bridge ratio recorded is limited by the accuracy of the measuring instruments.

In accordance with IPTS-68, the bridge ratio at the TPW is used to calculate the bridge ratio at the water freezing point, $R(\text{FPW})$, 0°C . (By definition, these points are separated by exactly 0.01°C). A constant, related to the alpha value of the SPRT and the correction for hydrostatic head of the TPW cell, is used to make the correction from $R(\text{TPW})$ to $R(\text{FPW})$. SPRT resistance ratios are calculated based on the $R(\text{FPW})$.

Col. 2 of Table 2 shows $R(\text{FPW})$ values obtained from TPW measurements made immediately after each of the gallium plateaux had been defined. A single standard resistor was common to all measurements. The SPRT resistance ratio is determined for the gallium point from the bridge ratio recorded at the gallium point $R(\text{Ga})$ divided by $R(\text{FPW})$. Use of ratios eliminates any uncertainty of the standard resistor, provided its short term drift is negligible. Col. 3 of Table 2 shows this ratio for each of the six sets of measurements of the two gallium cells.

A meaningful comparison of data assessing the accuracy of realization of the gallium point should be done in terms of temperature. Due to the fact that the data was gathered with an SPRT calibrated on IPTS-68, the gallium intercomparison data is expressed in terms of IPTS-68 temperatures.

The grand mean of the six measurements for each cell, converted to temperature from the grand mean of the ratios, is used for the inter-comparison. The temperatures appearing in Table 3, as determined from the NIST calibration report, are based on the resistance ratios shown in Table 2. The mean ratios of Table 2 are repeated in Row 1 of Table 3 for convenience.

Table 2 shows the bridge ratio at the FPW for the SPRT, as derived from the TPW measured immediately after a gallium melt. Temperature, as defined by an SPRT, is a function of resistance ratio, not ohmic value; thus ratios are shown for each set of gallium - FPW values.

TABLE 2 - BRIDGE RATIO AT FPW AND SPRT RESISTANCE RATIO AT THE GALLIUM MELTING POINT.

Run #	FPW Bridge Ratio	R(Ga)/R(FPW)
SN 139		
1	0.2553650	1.1181695
2	0.2553652	1.1181695
3	0.2553651	1.1181699
4	0.2553650	1.1181707
5	0.2553650	1.1181708
6	0.2553650	1.1181706
Mean resistance ratio:		1.1181702
Standard deviation (1σ)		4.73×10^{-7}
SN 140		
1	0.2553650	1.1181708
2	0.2553651	1.1181703
3	0.2553651	1.1181709
4	0.2553650	1.1181712
5	0.2553650	1.1181716
6	0.2553652	1.1181707
Mean resistance ratio:		1.1181709
Standard deviation (1σ)		4.68×10^{-7}

TABLE 3: DIFFERENCES IN MEAN TEMPERATURE OF GALLIUM MELTING POINT

	SN 139	SN 140	DELTA
Mean Ratio R(Ga)/R(0)	1.1181702	1.1181709	0.0000007
Mean Ga Temperature (NIST) report	29.771453°C	29.771630°C	0.177 mK
1σ Standard Deviation (temp)	0.120 mK	0.118 mK	0.002 mK

It can readily be seen from Table 3 that the values agree quite well. Although the gallium point is not recognized on the IPTS-68 as a defining point, it is assigned a value of 29.772°C as a secondary point. The temperatures above have not been corrected for hydrostatic head. This correction adds approximately 0.25 mK to the measured temperatures, bringing the values to 29.7717°C and 29.7719°C respectively.

The intercomparison will be no different on ITS-90, where the "hotness" of the gallium point is assigned the value 29.7646°C. The relative differences and standard deviations between cells will not change; they are derived from resistance ratios, which are Scale-independent. The equations describing the relationship between these ratios and temperature are slightly different for the ITS-90 and the IPTS-68 Scales. The functional difference, considering the small change in the assigned value, is negligible in this region.

The advantage of including the gallium melting point as a defining point of the new Scale is in providing a stable reference close to the water triple point. SPRT behavior can be described more fully over a much narrower range bordered by two highly-reproducible fixed points. In addition, the merit of an SPRT as a primary standard becomes much more easily determined and assessed. The IPTS-68 required a priori assumptions about the purity of the platinum wire, and evaluation at difficult-to-realize fixed points (the boiling point of water or the freezing point of tin) to establish SPRT suitability.

ANALYSIS OF UNCERTAINTIES

The standard deviation of the mean of each set of six measurements is nearly the same for both sets. This indicates a high degree of systematic consistency within the measurements. It is also somewhat representative of the limits of accuracy of the measuring system.

The standard resistor used has a nominal value of 100 Ω . Since the nominal resistance of the SPRT was 25 Ω , the ratios measured were typically slightly over 0.28 at the gallium point, and just over 0.25 at the water triple point. The inherent error due to the bridge (which corresponds to 2×10^{-7} at unity ratio) is approximately 0.7 ppm of the bridge ratio at gallium, and approximately 0.8 ppm at the water triple point.

The uncertainty in the SPRT resistance ratio due to the bridge error depends upon the error in the measurements made at these individual points. Propagating these errors in determining the resistance ratio from individual measurements yields an uncertainty in the ratio equivalent to 0.3 mK (see below). This assumes that the 100 Ω standard has negligible drift over the time these measurements were taken. Control charts on the standard show a drift rate of less than 1 ppm per year.

The water freezing point, as the fundamental defining point for all resistance ratios on the IPTS-68, must be expressed in terms of resistance. The TPW resistance, and thus the FPW resistance value, is derived from the bridge ratio using the value of the standard resistor. This leads to contributions to the uncertainty of the FPW resistance from both the error inherent in the bridge, and the resistor uncertainty.

Propagating these errors yields 0.2 mK as the uncertainty associated with the FPW value. However, as determinations are ultimately in terms of SPRT resistance ratio, rather than discrete resistance values at the gallium point and the FPW, the calculated uncertainty in this ratio represents the total systematic error introduced by the inherent inaccuracy of the bridge.

The standard deviations listed in Table 3 are much smaller than the uncertainty of the resistance ratio. The bridge inaccuracy is fixed at two counts in the last digit of the measured ratio. Using a reference resistor of 25 Ω instead of 100 Ω would increase the ratios by a factor of four, with the net effect of reducing the inherent bridge error by a factor of four. Errors due to standard resistor instabilities can be reduced by shortening the time between measurements and making evaluations in terms of resistance ratios only, thus reducing systematic measurement uncertainties.

Ideally, fixed points can be realized better than any system can measure; therefore limitations on accuracy reside within the measurement system. The analysis below will show what effect changing the standard resistor from 100 Ω to 25 Ω would have on the systematic (Type "B") uncertainty when determining SPRT resistance ratios.

R(FPW) = bridge ratio at the FPW

R(Ga) = bridge ratio at the melting point of gallium R(T) = SPRT

resistance ratio $\frac{R(Ga)}{R(FPW)}$

If $F = f(x,y)$ then

$U' =$ uncertainty in $F = dF$

$$= \sqrt{\left\{ \left[\left(\frac{\partial f}{\partial x} \right) dx \right]^2 + \left[\left(\frac{\partial f}{\partial y} \right) dy \right]^2 \right\}} \quad (\text{See footnote 5})$$

therefore

$$dR(T) = \sqrt{\left[\left(\frac{dR(Ga)}{R(FPW)} \right)^2 + (R(Ga)dR(FPW))^2 \right]}$$

$$dR(Ga) = dR(FPW) = 2 \cdot 10^{-7}$$

Using a 100 Ω standard resistor:

R(FPW) = 0.25536503, R(Ga) = 0.28554160

dR(T) = 1.18×10^{-6}

for SPRT SN 1120 this corresponds to dT = 0.3 mK = 'B'

Using a 25 Ω standard resistor:

R(FPW) = 1.0214601, R(Ga) = 1.1421664

dR(T) = 2.9×10^{-7}

for SPRT SN 1220 this corresponds to dT = 0.07 mK = 'B'

The random or Type 'A' uncertainty is taken as the standard deviation of the mean of the six measurements taken on a given cell. As this is the deviation from the mean ratio, changing the standard resistor would also have an effect. The magnitude of this effect can only be determined empirically, as the systematic contribution to the random error is not well defined.

The random error is taken as 0.120 mK, and is almost the same for both cells, as shown in Table 3.

Combining the Type 'A' and Type 'B' uncertainties using the 100 Ω resistor yields:

$$U = \sqrt{(A^2 + B^2)} \\ = 0.323 \text{ mK } (1\sigma)$$

Using a 25 Ω standard resistor and assuming the worst-case scenario, which is no change in the standard deviation:

$$U = 0.0140 \text{ mK } (1\sigma)$$

The use of a 25 Ω standard may in fact increase the noise in the measuring system. The reduction in uncertainty indicated above should be thought ideal. In actual practice, a twofold decrease in uncertainty is a more realistic expectation.

CONCLUSIONS AND FUTURE PLANS

Two identical, commercially available, gallium melting point cells have been measured in the Primary Standards Laboratory for the purpose of inter-comparison. Using a NIST calibrated SPRT, the resistance ratios measured for the two cells, referred to the freezing point of water, agree to within the basic measurement uncertainty of the measuring system. The results have been presented in terms of temperature for convenience.

Assuming that the accuracy of fixed point realizations is limited by measurement uncertainties, it has been shown that uncertainties can be de-created by at least a factor of two by relatively simple means. The practical limits of accuracy depend most strongly upon the measuring system.

A 25 Ω standard resistor is currently on order. As time allows, the measurements will be repeated to determine the decrease in uncertainty due to reduction in systematic error.

FOOTNOTES:

(1) Bedford, R.E., Bonnier, G., Maas, H., Pavese, F., *Recommended Values of Temperature for a Selected Set of Secondary Reference Points*, Metrologia 20, 4 (1984)

(2) and (6) are now manufactured by Isothermal Technology Ltd., South-port, England, and available from it or from Isothermal (USA), 250 West 57 Street, New York, NY 10170.

(3) is available from YSI, Yellow Springs, Ohio

(4) is available from Automatic Systems Laboratories, Milton Keynes, England.

(5) Jaeger, K.B., Rangan, L.K., *Procedures for Calculating and Reporting Calibration Uncertainties*, Lockheed Primary Standards Laboratory, internal communication, 1988, pp 2-4

Names of manufacturers of equipment are given for the sake of completeness. Mention of these products and manufacturers in no way constitutes an endorsement of the product.

REFERENCES TO THE LITERATURE

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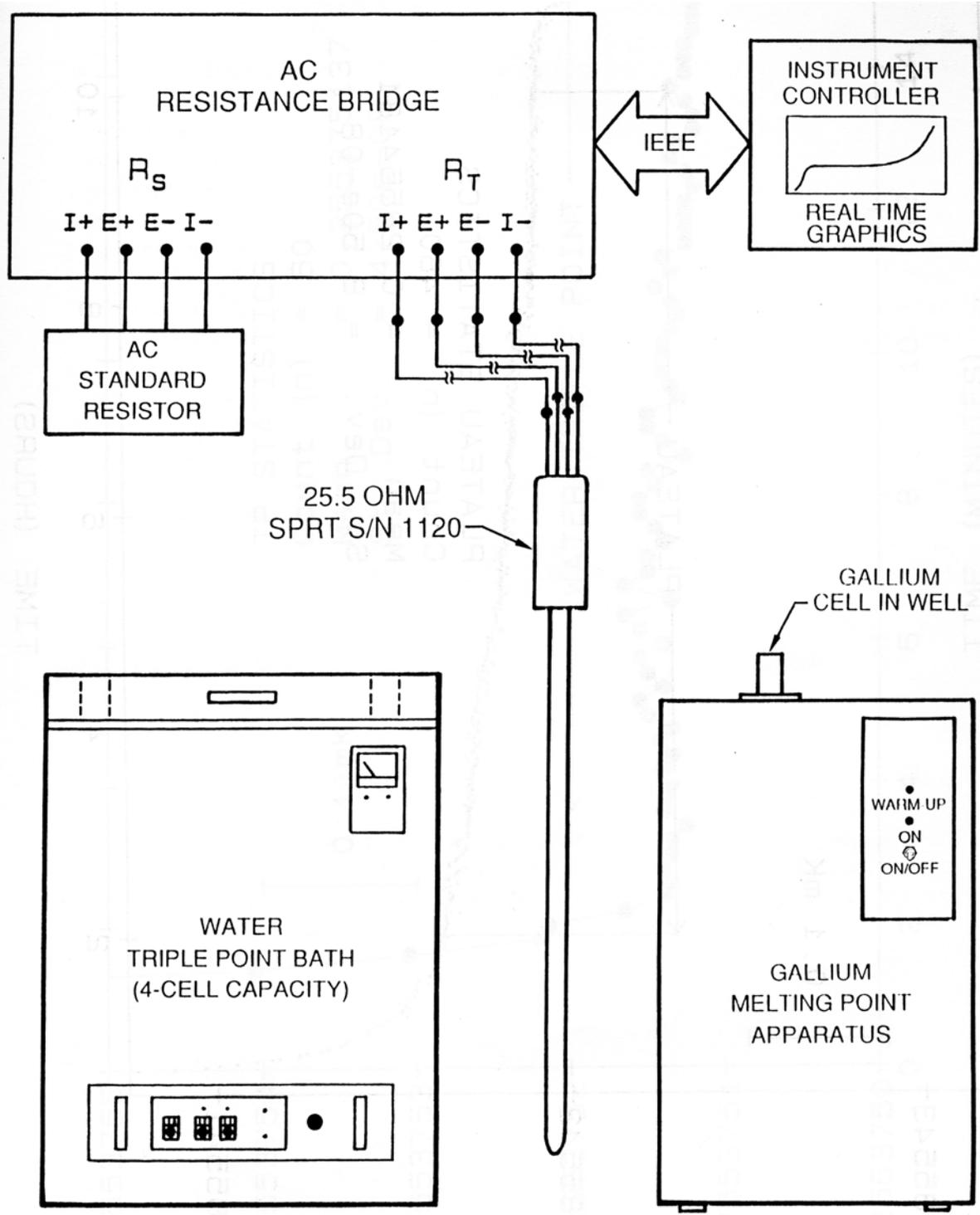


FIG 1 – EXPERIMENTAL ARRANGEMENT FOR REALIZING SPRT RESISTANCE RATIO AT THE GALLIUM POINT

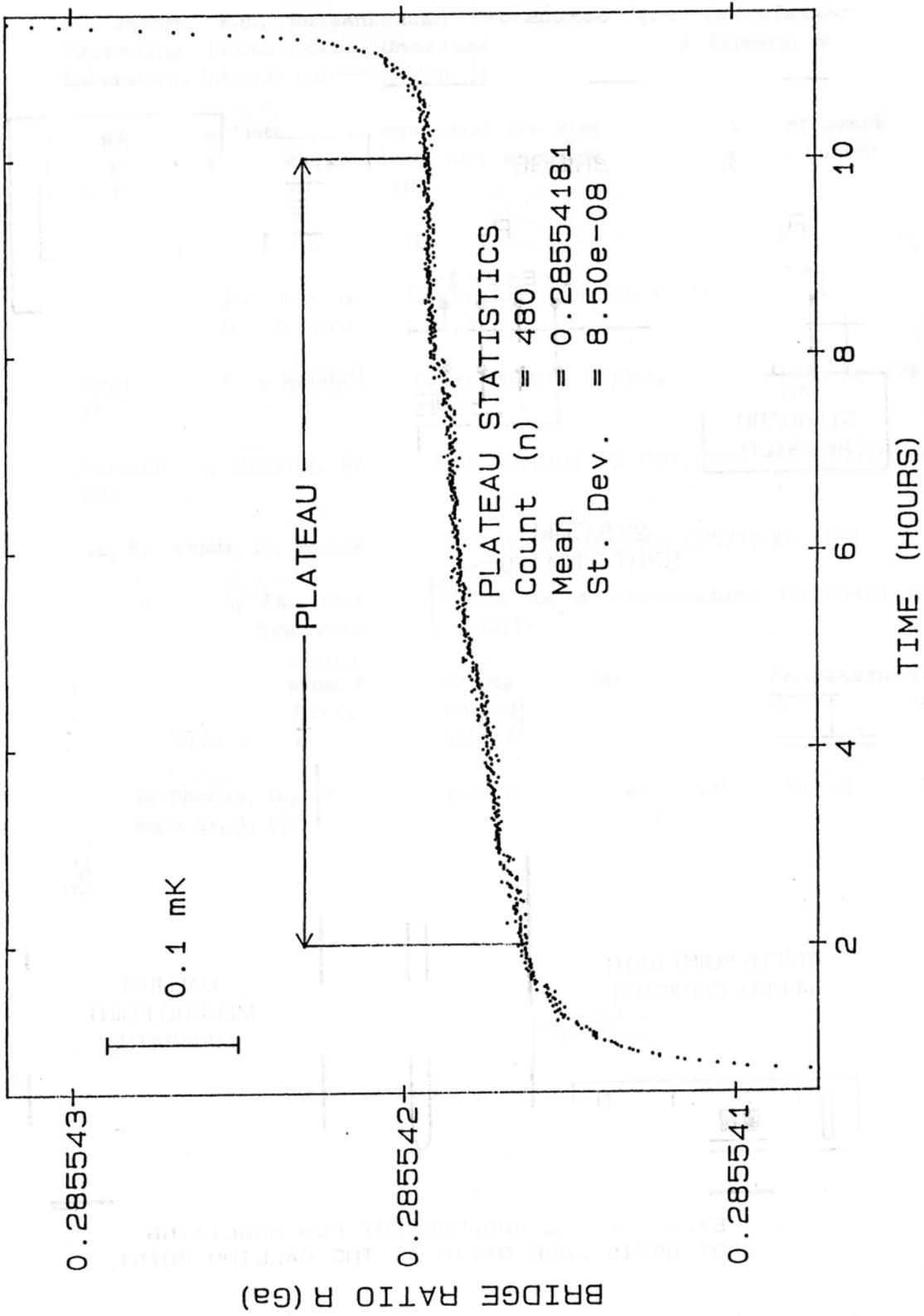


FIG. 2 - GALLIUM MELTING CURVE

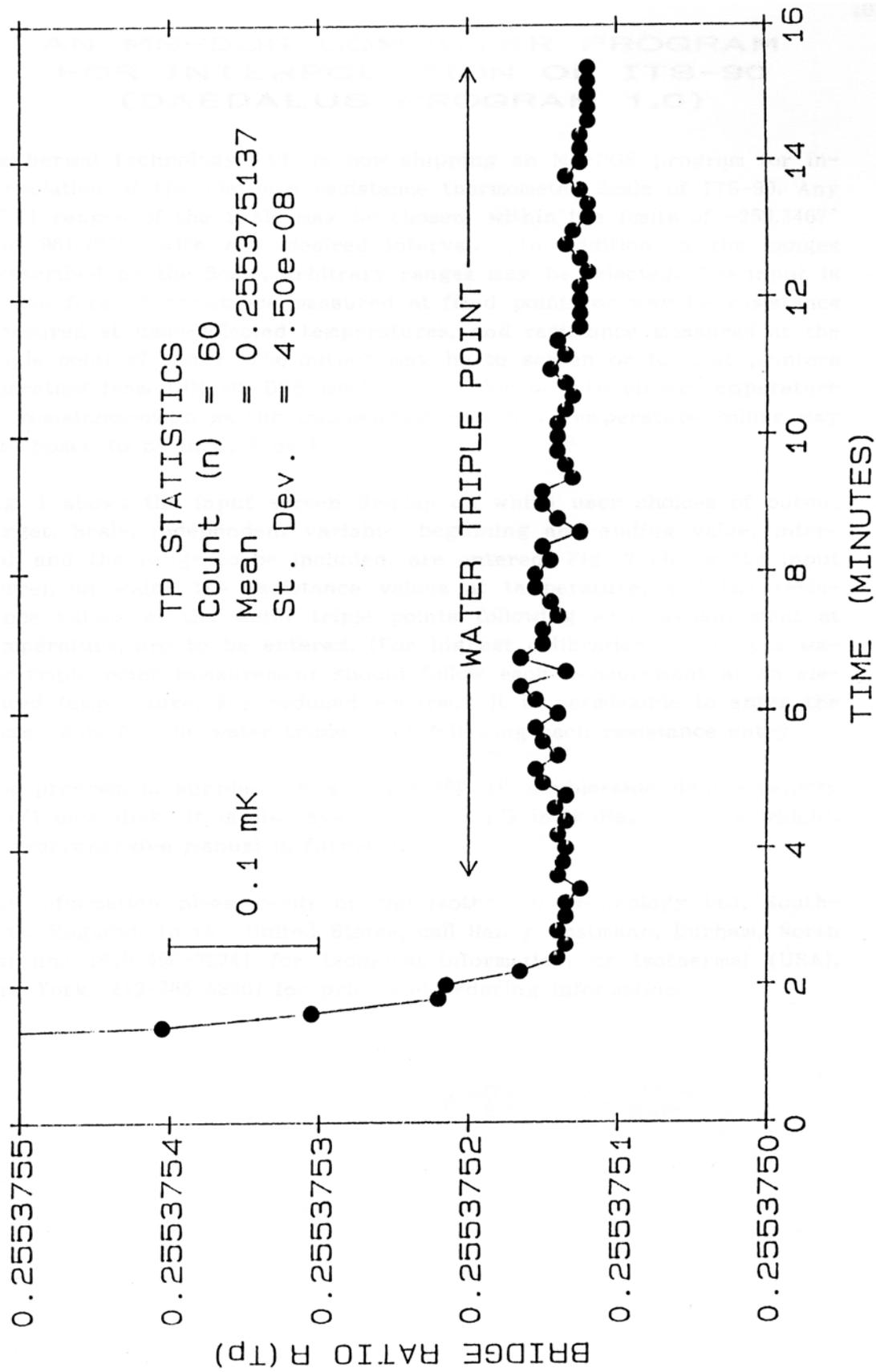


FIG. 3 - DETERMINATION OF WATER TRIPLE POINT

AN MS-DOS COMPUTER PROGRAM
FOR INTERPOLATION OF ITS—90
(DAEDALUS PROGRAM 1-0)

Isothermal Technology Ltd. is now shipping an MS-DOS program for interpolation of the platinum resistance thermometer Scale of ITS-90. Any of 11 ranges of the Scale may be chosen, within the limits of -259.3467° and 961.78°C , with any desired interval. In addition to the ranges prescribed by the Scale, arbitrary ranges may be selected. The input is in the form of resistance measured at fixed points or may be resistance measured at user-selected temperatures, and resistance measured at the triple point of water. The output may be to screen or to most printers operating from IBM MS-DOS machines or clones, with either temperature or resistance-ratio as the independent variable. Temperature values may be chosen to be in C, F or K.

Fig. 1 shows the input screen display on which user choices of output target, Scale, independent variable, beginning and ending value, interval, and the range to be included, are entered. Fig. 2 shows the input screen on which the resistance values at temperature, and the resistance values at the water triple points following each measurement at temperature, are to be entered. (For highest calibration accuracy a water triple point measurement should follow each measurement at an elevated temperature. For reduced accuracy it is permissible to enter the same value for the water triple point following each resistance entry.)

The program is supplied on a single 360 kb double-side double-density 5-1/4 inch disk. It is also available on 3-1/2 inch disk (specify which). A comprehensive manual is furnished.

For information please write or call Isothermal Technology Ltd, South-port, England. In the United States, call Henry Sostmann, Durham, North Carolina (919-490-3234) for technical information, or Isothermal (USA), New York (212-765-5290) for price and ordering information.

Fig 1- ENTRY SCREEN 1

Display to screen/printer S/P ()

Temperature Scale C/F/K ()

Plot temperature against W Y/N () Calculate coefficients Y/N ()

Start Value _____ End Value _____ Interval _____

EQUATIONS

Equations below 0 C (1-11) ()

Equations above 0 C (1-11) ()

1: -259.3467 C to 0.01 C
2: -248.5939 C to 0.01 C
3: -218.7916 C to 0.01 C
4: -189.3442 C to 0.01 C
11: - 38.8344 C to 29.7646 C

5: 0.0 C to 961.78 C
6: 0.0 C to 660.323 C
7: 0.0 C to 419.527 C
8: 0.0 C to 231.928 C
9: 0.0 C to 156.5985 C
10: 0.0 C to 29.7646 C

Fig. 2 DATA ENTRY SCREEN 2

OUTPUT TO SCREEN
EQUATIONS 2, 5

TEMP SCALE CELSIUS

CALIBRATION POINTS

Initial R(0.01) _____

Temp 1: -259.3467	R(1) _____	R(0.01) _____
Temp 2: -248.5939	R(2) _____	R(0.01) _____
Temp 3: -218.7916	R(3) _____	R(0.01) _____
Temp 4: -189.3442	R(4) _____	R(0.01) _____
Temp 5: - 38.8344	R(5) _____	R(0.01) _____
Temp 6: 231.928	R(6) _____	R(0.01) _____
Temp 7: 419.527	R(7) _____	R(0.01) _____
Temp 8: 660.323	R(8) _____	R(0.01) _____
Temp 9: 961.78	R(9) _____	R(0.01) _____

EXAMPLES OF THE MS-DOS 'DAEDALUS' PROGRAM ENTRY SCREENS. THIS BROAD RANGE OF CALIBRATION TEMPERATURES WOULD BE INAPPROPRIATE FOR ANY SINGLE REAL THERMOMETER.

ABOUT THE AUTHORS

HENRY E. SOSTMANN is a Consulting Metrologist practicing in temperature metrology, laboratory management and international legal metrological standards. He is a graduate of Rutgers University with graduate studies at Drew University, the Polytechnic Institute of Brooklyn, New York University and Wright State University. He was founder and President of H.E.Sostmann & Co., and later Vice President, Basic Metrology, of YSI. His present activities include U.S. representation on various secretariats of the International Organization for Legal Metrology, concerned with international harmonization of metrology product standards.

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BRAD MELLONS joined the Primary Standards Laboratory at Lockheed in 1985, and has been involved in all aspects of the development of primary temperature standards, including software design for automated data acquisition and analysis, development of measuring techniques and procedures for thermometry at the primary and secondary level, and assistance in the design of a primary cryogenic calibration station and a pyrometer calibration station. His experience spans the temperature range from cryogenics to 3000°C.