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THE EDITORS

HENRY E. SOSTMANN
JOHN P. TAVENER

FUNDAMENTALS OF THERMOMETRY PART V

INDUSTRIAL (USE) PLATINUM RESISTANCE THERMOMETERS

by Henry E. Sostmann, and

COMMON ERRORS IN INDUSTRIAL TEMPERATURE MEASUREMENT

by John P. Tavener

INDUSTRIAL PLATINUM RESISTANCE THERMOMETERS

ABSTRACT

The interpolation standard stipulated in the International Temperature Scale of 1990, over the temperature range from -189° to $+962^{\circ}\text{C}$, is a long-stem Standard Platinum Resistance Thermometer (SPRT); or rather, a pair of SPRTs, since no one thermometer can encompass the entire range [1]. Some characteristics required of an SPRT make it unsuitable and unfit for use outside the standardizing or calibration laboratory; therefore a variety of industrial-grade platinum resistance thermometers, of high quality, have been developed for use as field working standards, and in applications such as process monitoring and control.

DISCUSSION

The specific constraints on the SPRT, stipulated in the text of the ITS(90) [2], are that it be strain-free, that the ratio of resistances at the gallium point and the water triple point, or the mercury triple point and the water triple point, be

$$W_{ga} = R_{ga}/R_{wt} \geq 1.118\ 07 \quad \text{Eq. 1}$$

$$W_{hg} = R_{hg}/R_{wt} \geq 0.844235 \quad \text{Eq. 2}$$

and, in addition, if the thermometer is to be used to the silver point (962°C), that the ratio of the silver point and the water triple point resistances be

$$W_{ag} = R_{ag}/R_{tp} \geq 4.2844 \quad \text{Eq. 3}$$

(These constraints replace the requirement of the previous Scale, the IPTS(68), that the ratio of the resistances at 100° and at 0°C be

$$W = R_{100}/R_0 \geq .0032950 \quad)$$

Eq. 4

These requirements are a well-meant but unfortunately indirect attempt to specify the purity and strain-freedom of the platinum resistance element (it is not difficult to think of a better formulation). In order to conform to this requirement, the platinum wire must be almost ideally pure, and it must be mounted on some support in a fashion which (contradictorily) provides only a minimum of support, and no constraint. Thus, while the well-designed and constructed SPRT [3] is an instrument of extraordinary sensitivity and repeatability, it is delicate, and can be easily knocked out of calibration. It has been said, correctly, that if an SPRT is put down onto a surface with enough force so that it can be heard to touch it, it may be strained out of calibration. Ironically, it may be strained due to shipping shock as it is returned from a Laboratory where it has been newly calibrated! Precautions regarding the quality assurance of SPRTs are provided throughout the literature; e.g., [4].

In addition, SPRTs are physically fragile. The best of them contain the platinum element inside a sheath of fused quartz. Quartz is an unforgiving material, in that its elastic limit and its breaking point are the same, but its use is justified by its physical tolerance of high temperatures (its softening point is about 1500°C), its available purity, its transparency, which allows the platinum element to be viewed, its impermeability to gasses (except under special circumstances) and its obvious indication that it and the construction it contains are fragile and must be treated with respect. Metallic sheaths, on the other hand, can emit vapors which are poisonous to the platinum element, and provide a spurious sense of robustness. One manufacturer provides a quartz sheath inside a metal sheath, with the net effect that the protective inside quartz can be broken by a slight bending, and the platinum element exposed to the metallic vapors, with no visible indication to the user that the system is corrupt.

"INDUSTRIAL GRADE" RESISTANCE THERMOMETERS

I have provided quotation marks around the word "Industrial" in this heading to indicate customary terminology. The use of such thermometers is by no means limited to industry, but extends into numerous other fields of science, technology, and broad common application.

Industrial resistance thermometers (IRTs) are compromise devices, where the compromise is struck between the requirement (for resistance stability) that it be as free from strain as may be compatible with use, and the requirement that it be rugged and durable in the environment for which it is intended.

It should be mentioned, briefly, that not all industrial resistance thermometers are made of platinum wire. Other constructions are:

(a) Nickel, copper, and alloy wire. Nickel and copper wire were once very popular as thermometer materials. Both have temperature coefficients of resistance higher than that of platinum; approximately 6.9×10^{-3} and $6.5 \times 10^{-3} \Omega/\Omega/^\circ\text{C}$, respectively, as opposed to $3.9 \times 10^{-3} \Omega/\Omega/^\circ\text{C}$ for platinum. This higher change of resistance with temperature reduced the burden on earlier signal amplifiers; with modern solid state electronics, the advantage is negligible. Nickel is highly non-linear and passes through a sharp change in coefficient in the vicinity of 370°C where its magnetic characteristics change. Copper has a very low specific resistance, a disadvantage for thermometry. Both materials, since they are base metals, are more susceptible to contamination and oxidation than platinum. Both cost less than platinum, but the fraction of the cost of an IPRT which represents platinum wire is small. Both have faded from popularity as resistance thermometer materials (although copper is still employed in some in-slot temperature monitors in the protective circuits for electric motors).

(b) Platinum films, thin and thick. These will be included in this discussion.

(c) Non-metallic resistance thermometers; e.g., thermistors and other semiconductors. These will be discussed in a future issue of this series.

"INDUSTRIAL" PLATINUM RESISTANCE THERMOMETERS

Thus the industrial resistance thermometer of today is likely to be an Industrial Platinum Resistance Thermometer (IPRT).

The IPRT has a longer history than its most refined relative, the SPRT. Werner von Siemens is generally acknowledged to have made the first, proposing it in his Bakerian Lecture of 1871, and advancing a three-term interpolation algorithm. It came rapidly into use, largely because of its inventor's reputation, and declined as rapidly, because of inherent problems of stability. The Siemens thermometer comprised 1

meter of 0.1 mm (0.004 inch) diameter platinum wire wound on a porcelain or fire-clay tube, the whole assembly enclosed in an iron tube for protection. (Siemens also experimented with sensing elements of ceramic impregnated with platinum group metals).

A committee of the British Association for the Advancement of Science found that the resistance of the Siemens thermometer increased upon each heating, making it necessary to calibrate the thermometer each time it was used (and a calibration is a use). The change in resistance, reported to reach 15% after repeated heatings to 900°C, was assigned to chemical alteration in the platinum. (Two probable other reasons for increase in resistance are (a) gradual volatilization of some platinum, resulting in a decrease in sectional area of the wire, and (b) the growth of intergranular boundaries, affecting the conduction mechanism at these points).

About 20 years later, Callendar, and Callendar and Griffiths, revived the platinum thermometer for laboratory use over moderate temperature ranges. Callendar found that the clay substrate was a major cause of the variation of resistance; that the platinum wire "became brittle and stuck to the clay". We can guess now at gross silica contamination. On the other hand, a mica strip, that the platinum touched only at the edges, appeared to be "perfect" insulation in that it did not cause contamination. Callendar also stipulated that all joints with platinum be autogenous fusion weldments without foreign material such as solders; that pressure joints (screws or torsion) be avoided, and that copper conductors in the heated zone be eschewed, because of the volatility of the material; and we observe these strictures today.

As a generality, the work of Callendar and Griffiths was confined to the range 0° to 550°C. Over this range, they found that a third-order parabolic equation using three fixed points, ice, steam, and the boiling point of sulfur, was adequate to establish an interpolation scheme. Of the famous Callendar equation, which was the basis for International temperature Scales until 1968, more later.

PLATINUM AS A THERMALLY SENSITIVE MATERIAL

The development of the platinum resistance thermometer paralleled, in time, the development of platinum itself as a workable and pure material. Callendar's platinum was certainly not equivalent to pure platinum and the SPRT as we think of it today.

Platinum, as first refined, occurs as a spongy mass. In the mid years of the 19th century, it was beyond technology to force this mass

into an ingot by methods which retained its purity, and the wrought metal was obtained, essentially, by hammering. Today, it can be melted into an ingot which is then further compacted by successive swagings and annealings, until it is finally in the form of a square-sectioned bar ready to roll into strip, and eventually to draw into wire. Modern techniques for managing platinum, from the sponge to wrought wire, are generally proprietary [5].

A CONFUSION OF INDUSTRIAL STANDARDS

A corollary of the improvement in metallurgical techniques is increase in the temperature coefficient of resistance exhibited by the wire. The purer the material, the higher the temperature coefficient. Let us recall that most useful coefficient, α , a regrettable victim of the IPTS(68) and its subsequent replacement Scales:

$$\alpha = R_{100}/R_0 \qquad \text{Eq. 5}$$

α is, therefore, the measure of the sensitivity of platinum wire stated as the slope of a straight line between 0°C and 100°C, and is expected to remain a useful and popular way of classifying IPRTs.

For platinum of purity approaching ideal, α is higher than $3.928 \times 10^{-3} \Omega/\Omega/^\circ\text{C}$ (assuming always the absence of strain). The best of production SPRTs today have α coefficients between 3.925×10^{-3} (the minimum α permitted on the IPTS-68) and 3.928×10^{-3} .

Industrial resistance thermometry has a much longer history of use in Europe than it has in the United States. I cannot trace this history, but certainly resistance thermometers were commonplace in measurement and control early in the 20th century. This period predates the preparation of modern platinum, and, in consequence, the European standard for α has been fixed, before recent memory, at 3.850×10^{-3} ; quite probably, the best, but hardly ideal, wire that could then be obtained.

The migration of European process engineers to the United States after the war turned Western Hemisphere attention to IPRTs as more precise devices than process thermocouples. In the mid-1950s, I made 400 process-control IPRTs for a duPont textile fiber plant; the largest order ever placed, at that time, in the U. S. I made these from the best Cohn wire, and achieved α coefficients of about 3.916×10^{-3} . Obviously, these elements were not strain free; a compromise was necessary in order that they be stable under conditions of industrial use.

In the absence of an American standard for α , 3.915×10^{-3} became the *de facto* U. S. standard, while the European standard, cast in concrete, remained 3.850×10^{-3} . The American *de facto* standard reflected pure platinum wire in a less than ideal physical structure, while the European coefficient, once derived from platinum wire less than ideally pure, now reflected pure platinum wire doped with specific impurities [6]. The situation of two (and there were more than two) accepted coefficients would obviously cause problems. For example, a controller scaled for one coefficient would indicate (except at 0°C) improperly if the sensor were of the other coefficient. Many unsuccessful attempts at compromise were made, by many standards-writing bodies. SAMA, for example, promulgated a standard in which the sensing element α was required to be 3.923×10^{-3} (a completely unrealistic number for an IPRT) and was then shunted with prescribed shunts to an effective coefficient of either 3.915×10^{-3} or 3.850×10^{-3} (which incidentally altered the shape of the characteristic curve). The response of U. S. manufacturers to the confused situation was to produce sensors of both coefficients.

SPRTs are considered by their users to be individual instruments, whose characteristics are completely and adequately described by individual calibration constants and printed interpolation tables. IPRTs, however, are specified not as individual sensors, but as members of a group performing within certain limits. The primary emphasis here is on the interchangeability of like sensors, so that field replacements may be made without the necessity of recalibrating systems or processes. Elements may be checked by the manufacturer on an individual or on a statistical basis, and, often, the difference between thermometers of several accuracy classes represents sorting at inspection. An individual calibration of such a sensor is almost never done except in batch qualification, and is never offered to the user except as an extra-cost option.

It seems today that the confusion will eventually be resolved, not on technical grounds, but by demands of the marketplace for harmonization of standards on a global basis. A task group of the International Electrotechnic Commission (IEC) is formulating a revision of the IEC document on industrial resistance thermometers, and it will be based on the European α coefficient only. The IEC standard will be adopted rapidly and verbatim into the European Community standards documentation, as a DIN, BSA, etc. National standard, and the need to comply will be persuasive to all manufacturers, including those in the United States who wish to export.

In the absence of a uniform standard at this time, we offer Table 1, which lists tolerances and coefficients for IPRTs promulgated as National regulations within IPTS(68). (Note that the tolerances shown in

TABLE 1

INTERNATIONALLY AGREED UPON SPECIFICATIONS (PRIOR TO ITS-90)

IEC PUB 751 1983 OIML 1985 BS 1904:1984 DIN 43760 GOST 6651-84 JEMIMA SAMA RC4(1966)

	100	5 to 1000	100	100	10, 46, 100	100	100	100
R(O DEG C) OHMS TOLERANCE AT 0								
CLASS A	+/-0.06		+/-0.075	+/-0.06	+/-0.15	0.03 appr		
CLASS B	+/-0.12		+/-0.1	+/-0.12	+/-0.3	0.5 appr		special standard
ALPHA	0.00385	0.00385 0.00391	0.00385	0.00385	0.00385 0.00391	0.003916		0.003923
TOLERANCE								
CLASS A	+/-1.3E-05	+/-0.7E-05	+/-0.7E-05	+/-1.3E-05	+/-0.7E-05			
CLASS B	+/-3.0E-05	+/-1.2E-05	+/-2.0E-05	+/-3.0E-05	+/-1.1E-05			
RANGES, DEG C								
CLASS A	-200 TO 650	-200 TO 850	-183 TO 630	-200 TO 850	-200 TO 600	-200 TO 600		-200 TO 600
CLASS B	-200 TO 850		-220 TO 1050		-200 TO 850			
COEFF TYPE I								
A	3.90802E-03	3.90802E-03	3.96835E-03	3.90802E-03	3.90802E-03	3.97478E-03		3.98153E-03
B	-5.802E-07	-5.802E-07	-5.8349E-07	-5.80195E-07	-5.802E-07	-5.8775E-07		-5.8531E-07
C	-4.2375E-12	-4.274E-12	-4.3557E-12	-4.2735E-12	-4.2735E-12	-3.4813E-12		-4.3545E-12
COEFF TYPE II								
A		3.96968E-03			3.96847E-03			
B		-5.8677E-07			-5.847E-07			
		-4.141E-12			-4.3558E-12			

NOTE: IEC = INTERNATIONAL ELECTROTECHNICAL COMMISSION; OIML = INTERNATIONAL ORGANIZATION FOR LEGAL METROLOGY; BS = BRITISH STANDARD; DIN = DEUTSCHE INSTITUT FÜR NORMUNG; GOST = GOSSANDARD (ALL-UNION STANDARD, USSR); JEMIMA = JAPANESE STANDARD; SAMA = SCIENTIFIC APPARATUS MAKERS ASSOCIATION (UNITED STATES)

Table 1 are at 0°C. Fig. 1 shows the IEC 1983 tolerances at other temperatures). The Table suggests that almost any single international standard will be welcome, in place of these many conflicting standards.

A Table of values of resistance versus temperature may be calculated from any of these using the following algorithms (stipulated in the standards), which closely resemble the formulations of Callendar (above 0°C) and Callendar-Van Dusen (below 0°C). (In these equations, A, B and C are equivalent to but not numerically the same as Callendar-Van Dusen's α , δ and β).

Above 0°C:

$$R(t) = 1 + At + Bt^2 \quad \text{Eq. 6}$$

Below 0°C:

$$R(t) = 1 + At + Bt^2 + C(t - 100)t^3 \quad \text{Eq. 7}$$

Tables calculated using these equations will not be identically in accord with IPTS(68), since these equations represent mathematically continuous functions, and the IPTS(68) interpolation equations do not. (Think of the IPTS(68), and also the ITS(90), as a long clothesline hung between two supports, in a catenary curve, but interrupted at intermediate points by the fixed points which enter into the equation, giving the curvature of a clothesline supported by intermediate props). However it is stated by the designers of IPRT standards that the differences are not "industrially significant". (The major difference from IPTS(68) was about 15 mK plus the difference of the individual thermometer from the nominal curve). Until we have seen the equivalent constructions for ITS(90), it is not possible to estimate the divergence between the industrial scale and the ITS(90). Our prediction is that it will be much larger, because of the nature of the ITS(90) algorithms. Perhaps what we need (I appreciate what heresy I speak) is a quite distinct Industrial Platinum Resistance Thermometer Scale; an International Practical Temperature Scale.

THE CONSTRUCTION OF IPRT SENSING ELEMENTS

Most manufacturers of IPRTs do not stint on the quality of the platinum wire used for the sensing element, but commonly purchase "reference grade" wire when the α coefficient is to be 3.92×10^{-3} or higher, and the best wire when α is to be 3.85×10^{-2} . Lead wires may be commercial grade platinum. Occasionally other materials than platinum are used for lead wires, but platinum is preferable because it avoids ac-

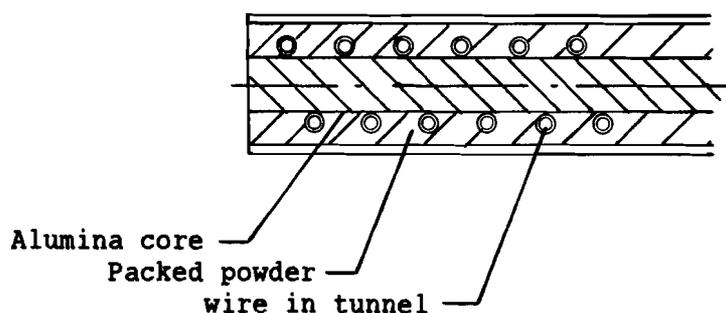
cidental thermoelectric junctions in the hot zone of the finished thermometer.

The resistance at 0°C may be 100Ω, 200Ω, 500Ω or some other value. For the 100Ω element a typical wire diameter is 0.025 mm (0.001 inch) for the realistic reason that this is the diameter at which the sum of the cost of the platinum metal and the cost of drawing the wire is a minimum. Higher 0°C resistance elements may employ even smaller diameter wire, 0.015 mm (0.0006 inch) being a realistic lower limit imposed by physical handleability. The wire is usually supplied in the hard-drawn condition to avoid stretching it during manufacture.

There have been a number of schemes for the disposition of the platinum wire upon the supporting structure. All are contrived to provide some freedom for the wire to expand, contract, etc., with minimum induction of mechanical strain, while still restraining the wire so that the physical motion of the wire as an accelerated mass will not in itself induce strain, or in the extreme example, cause unwanted turn-to-turn contact.

A design I used for many years is as follows. .001 inch diameter wire, coated with a film of Isonel varnish 0.0001 inch thick, was wound onto a high-purity alumina mandrel, and the ends spot welded to platinum lead wires fixed within the mandrel. After trimming to resistance tolerance, the system was dip-coated with ceramic, dried, and fired to anneal and to vaporize and drive off the varnish. (In later production, the winding was surrounded by a loose-fitting ceramic tube, and the interspace filled with ceramic powder compacted centrifugally). This left the wire free in a helical tunnel perhaps 0.0002 inch larger than the wire diameter. Since the thermal expansion of platinum is higher than that of the ceramic mandrel, it was necessary, first to stretch the wire by several cycles from room temperature to liquid nitrogen temperature, and then to re-anneal the wire above its upper temperature of use.

FIGURE 2

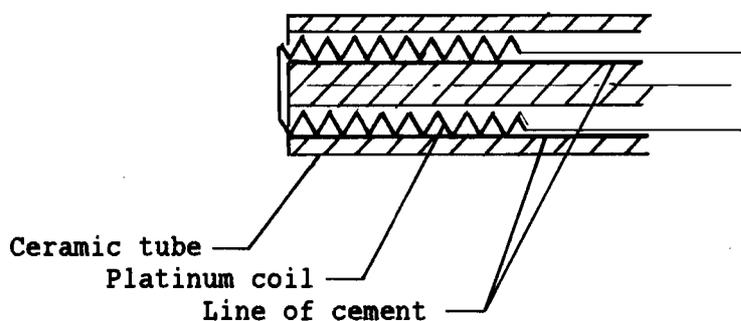


The "tunnel" construction of an element

In an experiment to determine the effectiveness of this construction, a large number of thermometers (ca 100) were made using bare wire, which would be closely captured by the coating, and Isonel-coated wire, which could be expected to have the freedom described above. All other details were common to both sets. The close-captured thermometers exhibited an a coefficient closely grouped about the mean of 3.915×10^{-3} , while the loosely-retained thermometers were all about 3.920^{-3} . Obviously, the latter were freer from strain. Fig. 2 indicates the tunnel construction.

The most usual design, due to Curtis and other workers, is to prepare two fine-diameter coils of platinum wire, and place these within two bores of a four-bore ceramic insulator, the other two bores being used to capture lead wires. The coils are welded together at one end and to the lead wires at the other end. A cement, ceramic or glass-based is introduced into the tubes holding the coils, in such manner that the cement contacts and secures only some specific portion of each turn. Fig. 3 indicates this construction.

FIGURE 3



The "2-coil" construction of an element

Stability with temperature change, and stability with mechanical shock and vibration, are directly contradictory requirements, and one may be satisfied only at the expense of the other. In the construction described above, the manufacturer has a wide range of choice between sticking down only a very small fraction of each turn of wire, for best thermal stability, a very large fraction (or all) of each turn for best mechanical stability, or any desired compromise between these.

These basic designs have been in use for many years. This is not to say that there has been no improvement over these years. The modern IPRT is superior for the following reasons, among others:

a: The fine platinum wire employed is now drawn through laser-drilled sapphire or diamond dies, which give repeatable results without contaminating the platinum.

b: Ceramic materials, mandrels, substrata and cements, have reached a development stage of purity undreamed of even 10 years ago.

c: Techniques have been developed which permit sensing elements to be thin and long, short and fat, very small, flat and rectangular, to include two or more electrically separate matched elements, etc. Examples of available shapes and sizes are shown in Table 2 [7].

FILM ELEMENTS

Much work has been done to develop platinum sensors based on thin- and thick-film technology, but the results have been to date disappointing. The hoped for results of lower cost and performance equivalent to that of wrought-wire sensors have simply not been realized, and there is now reason to doubt that they can be.

(a) Film thermometers must be mounted intimately to a substrate, but a substrate material of identical mechanical expansion properties has not yet been identified. Many film elements are excellent thermal expansion strain gages!

(b) The paste or film, which contains very little metal, is easily contaminated, and therefore usually is sealed under a glassy coating, which is subject to the problems of (a) and also may contaminate the platinum.

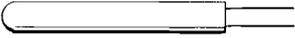
(c) The inhomogeneity of the paste provides characteristics entirely different from wrought wire; resembling, rather, spongy platinum.

(d) Characteristics vary from batch to batch, as a function of sintering temperature, etc.

(e) The thin film units are so small that self-heat is a problem not solved.

(f) mechanical attachments of leads, etc., are weak and susceptible to the generation of thermal emfs.

All dimensions in mm
Detectors shown same size

		Resistance tolerance at 0°C	Ceramic length	Ceramic diameter	Sensing length
	P100/7040	0,1%	70mm	4mm	65mm
	P100/5024 P2100/5024	0,1%	50 + 0 - 0,5	2,4 + 0 - 0,03	47 ± 1
	P100/5015 P2100/5015	0,1%	50 + 0 - 0,5	1,5 + 0 - 0,03	48 ± 1
	P100/3045 P2100/3045	0,1%	30 + 0 - 0,5	4,5 + 0 - 0,03	27 ± 1
	P100/3038 P2100/3038	0,1%	30 + 0 - 0,5	3,8 + 0 - 0,03	28 ± 1
	P100/2532 P2100/2532	0,1%	25 + 0 - 0,5	3,2 + 0 - 0,03	22 ± 1
	P100/2528 P2100/2528	0,1%	25 + 0 - 0,5	2,8 + 0 - 0,03	22 ± 1
	P100/2524 P2100/2524	0,1%	25 + 0 - 0,5	2,4 + 0 - 0,03	22 ± 1
	P100/2515 P2100/2515	0,1%	25 + 0 - 0,5	1,5 + 0 - 0,03	22 ± 1
	P100/2516 P2100/2516	0,1%	25 + 0 - 0,5	1,6 + 0 - 0,03	22 ± 1
	P100/2020 P2100/2020	0,1%	20 + 0 - 0,5	2,0 + 0 - 0,03	17 ± 1
	P100/1545 P2100/1545	0,1%	15 + 0 - 0,5	4,5 + 0 - 0,03	12 ± 1
	P100/1532 P2100/1532	0,1%	15 + 0 - 0,5	3,2 + 0 - 0,03	12 ± 1
	P100/1528 P2100/1528	0,1%	15 + 0 - 0,5	2,8 + 0 - 0,03	12 ± 1
	P100/1524 P2100/1524	0,1%	15 + 0 - 0,5	2,4 + 0 - 0,03	12 ± 1
	P100/1520 P2100/1520	0,1%	15 + 0 - 0,5	2,0 + 0 - 0,03	12 ± 1
	P100/1516 P2100/1516	0,1%	15 + 0 - 0,5	1,6 + 0 - 0,03	12 ± 1
	P100/1515 P2100/1515	0,1%	15 + 0 - 0,5	1,5 + 0 - 0,03	12 ± 1
	P100/1512	0,1%	15 + 0 - 0,5	1,2 ± 5%	12 ± 1
	P100/1509	0,1%	15 + 0 - 0,5	0,9 ± 5%	12 ± 1
	P100/1012	0,1%	10 + 0 - 0,5	1,2 ± 5%	8 + 0 - 1

Nevertheless film sensors seem adaptable to uses which require no great accuracy or stability, and where cost is a paramount consideration; e.g., home heating and air conditioning systems.

HOUSING THE IPRT SENSOR

Almost all IPRT sensors require some sort of enclosure when they are put to use, to protect the sensor, provide for the securing of external connecting wires, close off the spaces into which they are inserted, couple them to a process or an environment, etc. The variety is limited only by the configuration of the sensor chosen, and the ingenuity of the designer.

For those who need only one or a few special configurations, it is not excessively difficult to buy sensors and assemble thermometers on a do-it-one's-self basis. Precautions include the necessity to preserve cleanliness, to make impeccable joinings of wires, to pay attention to thermal coupling between the sensor and the outside environment, and to provide adequate electrical isolation. For those who require more than a few thermometers, it is usually more effective to adapt to the many commercial configurations available, or to seek an assembler willing to make up special configurations. It is possible to mount IPRT sensors in tubes, wells, drilled holes in casings, machine screws, etc.

CALIBRATING THE IPRT

Accurate calibration of an SPRT, in at least one sense, is simple compared to calibrating an IPRT. One has only to have a suitable set of fixed points into which an SPRT fits, diametrically and with sufficient immersion, and the rest is straightforward.

The majority of IPRTs cannot be calibrated in the usual sort of ITS(90) fixed point cell, because they won't fit, or they are not designed for sufficient immersion, or because they won't tolerate the temperatures along the length of the lead wires.

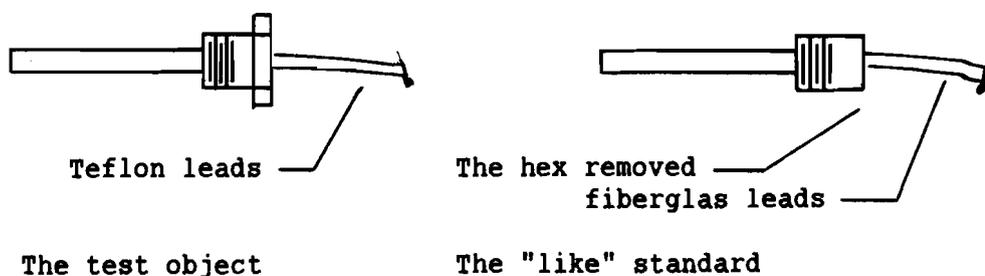
The concept of the "Like Standard" is a useful one in the calibration of IPRTs. It comprises the creation of a reference thermometer, as "like" as possible to the thermometer to be calibrated, but altered in whatever manner is necessary to adapt it to a fixed point cell.

An example is shown in Fig. 4. This small sheathed thermometer is intended to be screwed into the hot zone of a spinnerette for making a textile fiber. In use, the tip projects into a stream of high-velocity steam. Since the leads are external to the heated system, they are Tel-

fon insulated. The hexagonal nut is too large to fit a fixed-point cell, and the leads will not tolerate the temperature.

A "like standard" was made by (a) reducing the diameter of the hex nut and the threaded portion (b) substituting glass-insulated leads for the Teflon leads of the working thermometers. It was then possible to obtain calibrations of the "like standard" at fixed points, so that the "like standard" could serve as a reference thermometer for comparison calibrations of the working thermometers.

FIGURE 4



The short length of the thermometer meant that the immersion depth would not be sufficient. The paper by John Tavener which immediately follows will suggest the errors inherent in the system. However the "like standard" had, intentionally, the identical deficiencies, and when the standard and a working thermometer were screwed side-by-side into a copper block, and the block immersed in a constant-temperature bath, these deficiencies cancelled almost exactly.

One might well ask: but in use, did the working thermometer accurately realize the temperature of the steam? In this real-world example, no one really cared. The relevant matters were (a) once the process had been optimized, the working thermometer maintained the preset temperature (b) any replacement of the working thermometer (say in the event of its failure) had the same calibration and characteristics.

COMMON ERRORS IN TEMPERATURE MEASUREMENT

ABSTRACT - JOHN P. TAVENER

It is the first law of thermometry that a thermometer senses no temperature except its own. While it is easy to obtain steady readings of temperature from a resistance thermometer, it is much more difficult to be sure that these readings indicate the temperature of the object or environment of interest.

Of the almost infinite number of ways in which errors can occur, some of the more obvious are discussed here, together with an estimate of the magnitude, and corrective measures which may be applied.

INTRODUCTION

If two systems in thermal equilibrium are each the same temperature as a third, then they are also the same temperature as each other. But no perfect system exists. The temperature of an object is affected by the thermometer, and the thermometer may be temporarily or permanently affected by the system into which the thermometer is immersed.

In obtaining a steady reading with a thermometer, the following need to be considered, before one can accept the reading as the correct system temperature.

1: Thermal lag; the delay caused by the thermometer's thermal or electrical inertia

2: The thermometer's thermal capacity

3: Immersion error, caused by heat transfer from the system (including the environment) to the thermometer

4: Self-heating, caused by the necessity of passing a current through the thermometer

5: DC errors; effects of emfs caused by junctions between dissimilar metals

6: Effects of lead resistance

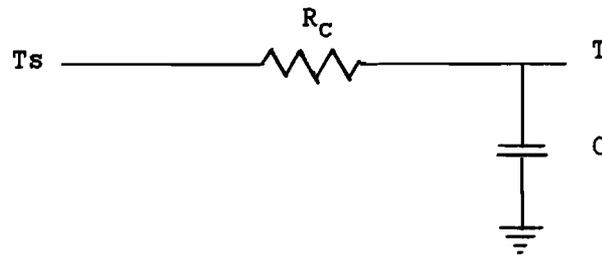
1: Thermal lag. If a small-diameter thermometer is inserted loosely into a large diameter pocket or well, one feels instinctively that the thermometer immersion should be deeper than if it were a tight fit, or the pocket or well were absent. Not necessarily true: the size of an air gap around the thermometer is almost irrelevant, compared to the fact that there *is* an air gap.

(I once had the problem of designing a thermometer for a reactor bypass cooling water loop, which had to show a 63% response time of 2 seconds in water flowing turbulently at 2000 feet per minute at 3000 psig, so that the fast response had to be achieved in a mechanically very rugged construction. The sensing element was mounted in a hole drilled in a taper pin, and embedded in a beryllium oxide paste. The

interior of the sheath was reamed to hold the taper pin. The response time was 8 times longer with the pin loosely in place than when it was driven tight, as a result of the very slight air gap! - HES)

However the combination of thermal resistivity between the thermometer and the temperature to be measured, and the heat capacity of the thermometer itself, give rise to an effect known as thermal lag. The heat flow required to warm or cool the thermometer causes a finite response time. I describe this effect with an electrical model in Fig. 5:

FIGURE 5



An electrical analogue model of thermal lag

Fig 5: Thermal lag. T_i = the initial temperature of the thermometer, T_s and T are the temperatures of the system and the thermometer respectively, R_C is the thermal resistance between the thermometer and the system, C is the heat capacity of the thermometer, and τ is time to achieve a 63% response.

Then the error in the temperature measurement (the shaded area of Fig. 6) is

$$T_e = -(T_s - T_i) \exp(-\tau/T) \quad \text{Eq. 1}$$

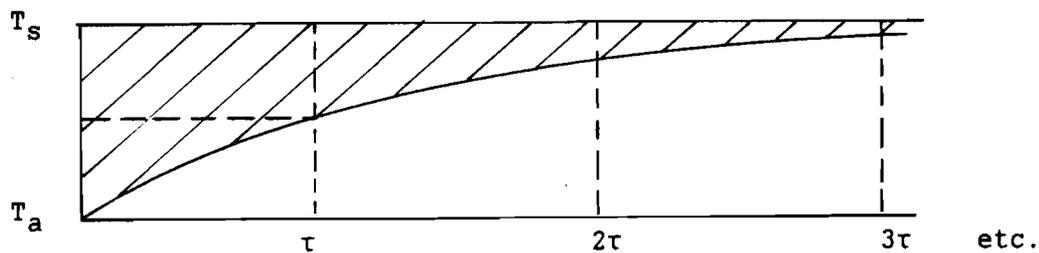
Other ways of expressing time constant are commonly found in the literature, and there appears to be no general consensus on how to present this information. Usually, however, a manufacturer specifies how the information he provides was determined.

In a practical situation, the user can make the error arbitrarily small by waiting for a sufficient time. Fig. 6 shows the factor

$$T_e/(T_s - T_i) = -\exp(-\tau/T) \quad \text{Eq. 2}$$

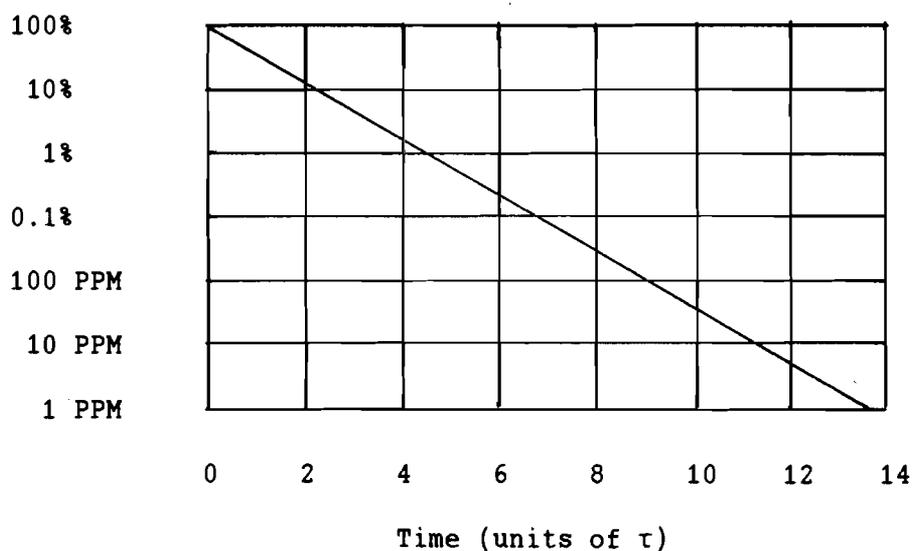
in multiples of the response time.

FIGURE 6



The effect of thermometer response time on a measurement

FIGURE 7



Temperature error $T_e/(T_s - T_i)$ plotted against measurement time in multiples of τ

EXAMPLE: Estimate the minimum measuring time to achieve an accuracy of better than 0.1% when measuring a temperature near 150°C with a thermometer whose response time is 15 seconds.

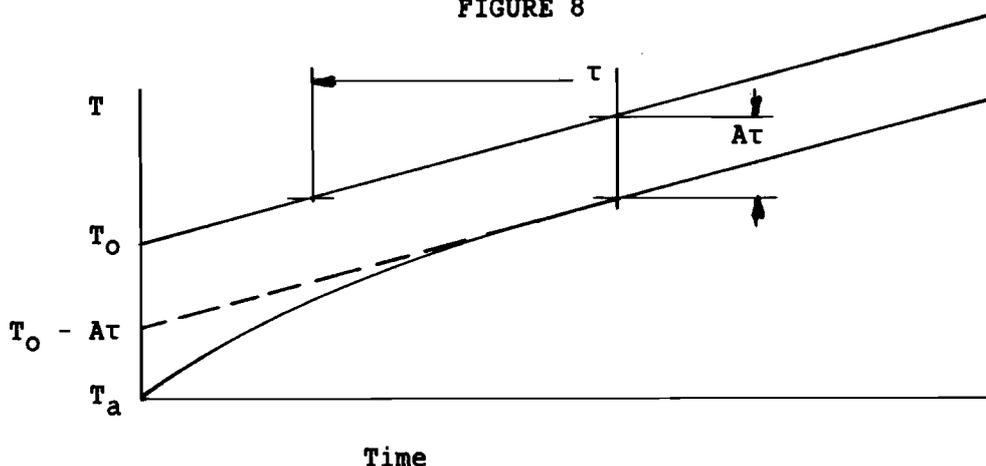
The maximum error is

$$0.1^\circ\text{C}/(150^\circ - 20^\circ) = 0.07\% \quad \text{Eq. 3}$$

From Fig. 7, the minimum measurement time is $7\tau = 105$ sec.

In systems where the temperature is not constant, the measurement errors become more complex. Consider the situation in which the

FIGURE 8



Temperature error due to the thermometer time constant in a system with the system temperature constantly rising

In systems where the temperature is not constant, the measurement errors become more complex. Consider the situation in which the system temperature rises continually, as in Fig. 8. Here there are two error components, an exponential component

$$T_{e1} = -(T_0 - AT - T_i) \exp(-\tau/T) \quad \text{Eq. 4}$$

and a constant component

$$T_{e2} = -AT \quad \text{Eq. 5}$$

where the system temperature $T_s = T_0 + A\tau$ and A is the rate of rise of the system temperature.

The exponential component, T_{e1} , can be dealt with by waiting for a long enough time. The constant component T_{e2} can be dealt with only by using a thermometer of shorter time constant.

2: Thermal capacity. When a thermometer is immersed into a system, heat will flow between the system and the thermometer until equilibrium is reached. Unless the system temperature is under external control, a permanent change in the system temperature will result. The smaller the heat capacity of the thermometer, the smaller will be the effect upon the system temperature. A simple model of the measurement process provides an estimate of the resultant temperature error:

Let C_t and C_s be the heat capacities of the thermometer and the system respectively, T_s be the final system temperature, and T_i and T_f be the initial and final temperatures of the thermometer. Then

$$T_e = -[C_t/(C_s + C_t)][T_s - T_i] \quad \text{Eq. 6}$$

and rearranging,

$$T_e/(T_s - T_i) = C_t/(C_s + C_t) \quad \text{Eq. 7}$$

Therefore, to achieve less than 1% error, the heat capacity of the thermometer C_t should be at least 100 times smaller than the heat capacity of the system C_s .

EXAMPLE: Suppose a thermometer has a heat capacity of $5\text{J}^\circ\text{C}^{-1}$, and is used to measure the temperature of a cup of coffee. Estimate the temperature error due to heat capacity. Assume that the temperature of the coffee is 80°C , the initial temperature of the thermometer is 20°C , and the heat capacity of coffee = the heat capacity of 250 ml of water = $1000\text{J}^\circ\text{C}^{-1}$.

From Eq. 5,

$$T_e = [5\text{J}^\circ\text{C}^{-1}/1000\text{J}^\circ\text{C}^{-1}][80^\circ\text{C} - 20^\circ\text{C}] = 0.3^\circ\text{C} \quad \text{Eq. 8}$$

The thermometer used in this Example is a small sheathed IPRT. Small thermistors and very fine thermocouples can have heat capacities less than $0.02\text{J}^\circ\text{C}^{-1}$, while mercury-in-glass thermometers may have heat capacities of as much as $200\text{J}^\circ\text{C}^{-1}$. Such a mercury-in-glass thermometer would show a 12°C error; even in 30 liters of coffee, the error would be 0.1°C .

In many measurements, it is possible to preheat or precool the thermometer, so that the initial temperature of the thermometer is close to the system temperature.

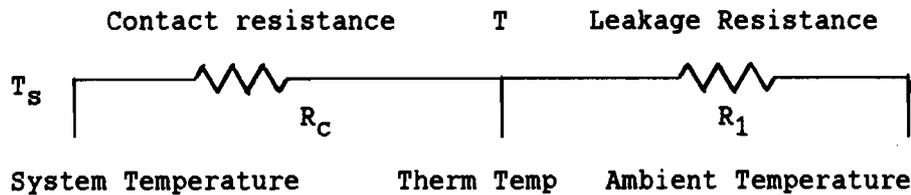
3: Thermometer immersion depth. A definition: A thermometer is sufficiently immersed in a system when there is zero heat flow between the sensor and the external non-system environment through the leads, sheath or other thermometer parts that extend from the sensor to ambient temperature.

Heat flowing through the thermometer from or to ambient is absorbed or replaced by the system in the forms of conduction, convection and radiation. A simple model:

$$\delta T = qR \quad \text{Eq. 9}$$

where δT = temperature difference, q = heat flow and R = thermal resistance. This can be compared to Ohm's Law, and represented shown in Fig. 9:

FIGURE 9



a: The greater the immersion depth, the greater the resistance to leakage; therefore the thermometer should be immersed as far as may be practicable.

b: The greater the immersion depth, the smaller the contact resistance is likely to be.

c: The contact resistance also depends upon the thermal conductivity of the system, if a fluid how fast the fluid is flowing, whether the flow is laminar or turbulent, and whether there is cavitation behind the thermometer.

A simple formula is:

$$T_e = (T_a - T_s) K_o \exp(-L/L_o) \quad \text{Eq. 10}$$

where T_e = temperature error, T_s = system temperature, L = immersion length of the thermometer, L_o a constant called the "characteristic length of the thermometer", T_a = ambient temperature, and K_o = a constant always less than 1.

In instances where the conductivity of the system is poor, or where high precision is required, a simple experiment will determine L_o and estimate the magnitude of T_e . At least three measurements must be made. at immersion depths of L_1 , L_2 and $L_3 = \delta L$. Temperatures of T_1 , T_2 and T_3 are obtained.

$$T_s = T_1 + [T_2 - T_1]^2 / [2(T_2 - T_1) - (T_3 - T_1)] \quad \text{Eq. 11}$$

and, rearranging Eq. 10,

$$L_o = [\delta L] / \ln[(T_s - T_1)/(T_s - T_2)] \quad \text{Eq. 12}$$

EXAMPLE: Suppose that measurements at immersion depths of 3, 4 and 5 cm give measured temperatures of 115°, 119° and 121°C. What is the system temperature and the characteristic length of the thermometer?

$$T_s = [119 - 115]^2 / [2(119 - 115) - (121 - 115)] \quad \text{Eq. 13}$$

$$= 123^\circ\text{C}$$

$$L_o = [4\text{cm} - 3\text{cm}] / \ln[(123 - 115)/(123 - 119)] = 1.44 \text{ cm} \quad \text{Eq. 14}$$

4: Self-heating error. A resistance thermometer is a passive electrical element; in order to make a measurement of its resistance, a current must be passed through it. This inevitably results in some heating of the thermometer, which is inevitable:

$$P = I^2R \quad \text{Eq. 15}$$

This results in an elevation of the apparent temperature of the thermometer (which measures only its own temperature) and an elevation of the system temperature. The problem is to evaluate whether this heating is significant in terms of the accuracy required of the measurement.

The self-heating effect is readily determined by placing the thermometer in a controlled environment and making measurements at at least two impressed currents. From this the so-called "zero power resistance"; that resistance which would be measured if it could be measured with no impressed power; and the actual resistance measured at any current may be estimated as a difference from R_o :

$$R_o = [R_1 - i_1^2] [(R_2 - R_1) / (i_2^2 - i_1^2)] \quad \text{Eq. 16}$$

5: Voltage errors. Most resistance-measuring systems compare the voltage across the unknown resistor with that across a reference resistor. Therefore any extraneous voltages which arise in the measurement path are a source of error.

The largest DC error is caused by imperfect amplifiers. The ideal amplifier develops zero voltage when both inputs are at zero potential. Any output voltage which occurs under these conditions can be replaced by an equivalent DC input voltage V_{OS} , referred to as the input offset voltage of the amplifier. Most manufacturers of amplifiers supply data

sheets giving a typical or maximum value for V_{OS} , which may typically range from 20 μV to 5 mV depending upon the type or quality of the amplifier. The offset voltage and its sensitivity to various parameters (e.g., temperature, power, supply voltage and time) represent the most important sources of error in operational amplifier circuits.

Thermal emfs are another important type of DC error. They are generated when junctions of leads of dissimilar materials act as thermocouple junctions. (Even joints in copper wire from two different manufacturers may produce emfs as high as $0.2 \mu\text{V}^\circ\text{C}^{-1}$. The emf of a copper-platinum junction is typically 6 to 8 $\mu\text{V}^\circ\text{C}^{-1}$.)

In assembling a thermometer, the maker should avoid making lead junctions from different types of wire, especially wire that may have become contaminated. The net emf can be further reduced by keeping pairs of junctions close to each other (i.e., at the same temperature), and, if solders are used at all, employing "low thermal" solders.

Voltaic emfs arise from electrochemical activity between dissimilar metals, in the same way that batteries generate voltage. The problem should not occur if the same wire is used throughout, the joints are perfect, and the environment is clean and dry.

Temperature error caused by DC voltage errors is given by

$$T_e = V_e/S \quad \text{Eq. 17}$$

where V_e is the input voltage error (mV) and S is the sensitivity of the instrument ($\text{mV}^\circ\text{C}^{-1}$).

Although it is possible to reduce some of these errors by good technique (e.g., by exchanging leads and averaging measurements) voltage errors limit the practical accuracy of a DC resistance thermometer. AC techniques are free from voltage errors but may be subject to resistance time constant limitations [8].

6: Lead resistance errors. Industrial PRTs may be connected as 2-wire, 3-wire and 4-wire systems. The reference provides a discussion of these errors and their mitigation [9].

FOOTNOTES

[1] John P. Tavener, Platinum Resistance Thermometers as interpolation standards for ITS-90, *Isotech Jour Therm* V1 N1 pp 31-37

[2] H. Preston-Thomas, The International Temperature Scale of 1990 (ITS-90), *Metrologia* 27, pp 3-10 (1990)

[3] C. H. Meyers, Coiled-Filament platinum resistance thermometers, *Isotech Jour Therm* V2 N1 pp 16-24

[4] Henry E. Sostmann, Standard platinum resistance thermometers, *Isotech Jour Therm* V2 N1 p 10 et seq.

[5] I am indebted for a conversation with Michael O'Shaughnessy, Sigmund Cohn Corporation, Mount Vernon, New York, for information about the preparation of pure platinum wire.

[6] Dr. Bert Brenner, for many years the revered metallurgist of the Sigmund Cohn Corporation (the world's premier supplier of thermometer wire) favored the pure platinum coefficient. He said to me once: "I have not spent my life learning how to purify platinum in order then to contaminate it!".

[7] From the catalog of TDI, Southport, England. For the addresses of TDI and its Western Hemisphere agent, please see Page 3 of this issue.

[8] See H. E. Sostmann, Fundamentals of Thermometry Part IV, Standard resistors, resistance bridges, thermometer measurements, *Isotech Jour Therm* V2 N2 pp 67-69

[9] *Ibid.*, Pages 72-73

I BLAME THE MOTHER-IN-LAW

by David Ayres

ABSTRACT

The temperature bath in which the bath medium is finely-divided alumina, "fluidized" or levitated by flowing air, has been in use for a number of years. Its advantages are well known, and so are its disadvantages. Among the former are (a) the temperature range can be large without the need to change medium (b) the medium, unlike molten salts, is not in itself hazardous (c) the rate of temperature change can be relatively rapid. Set against these are some very cogent objections, such as the infiltration of slippery powder throughout the entire laboratory.

This paper describes Dave Ayres' frustrations with fluidized baths as he found them, and the stages of development of a system which retains the advantages and eliminates the objections.

DISCUSSION

It was the fault of my mother-in-law that I found myself frustrated with a fluidized-bed calibration bath that refused to give repeatable results.

It began when she spotted an advert for a job with the Central Electricity Generating Board (as it was then called) of the United Kingdom, and sent me off in response, to win the hearts, as I did, of the interview Board. Thus it came to be that I was thrown into the broad and bottomless pond of Metrology, with enthusiasm my only life vest.

The fluidized-bed furnace causing problems was part of an automatic thermocouple calibration system. This new boy had the bright notion of repeating a calibration to see "what would happen" - a practice which does not necessarily lead to tranquillity - and of course, the two calibrations disagreed one with the other. My next mistake was to let this be known to my superiors, who, masters of the art of delegation, saw their role in the matter to convince me that it would be a splendid idea if I were to sort it all out. I had rubbed the bottle; I was now to persuade the Genie to pop back inside.

This is what I found out. A fluidized furnace consists, usually, of a container of aluminum oxide powder. The container has a porous base

plate, and sufficient air is passed through the porous plate to motivate the powder into a fluid-like state, so that it will flow under gravity, display buoyancy effects, and transfer heat well. However, unlike a fluid, it can be compressed. Also, it does not have a freezing or boiling point, as fluids do, and therefore is usable over a wide temperature range. The heat to the bath is supplied by heaters immersed in the powder, and for below-ambient baths there are provided external cooling coils.

The advantages of the fluidized bed furnace over more conventional baths and furnaces are these:

- (a) No toxic or flammable fumes are produced
- (b) The temperature range covered can be very wide
- (c) The working volume can be large
- (d) The fluidized medium can transfer heat ten or more times faster than a circulated-air furnace
- (e) When changing temperature, the transit time between setpoints can be quite short, and unattended automated control is easily provided.

The disadvantages of fluidized baths, at that stage of their development, were:

(a) Good temperature stability and uniformity could not be achieved in the medium, but were obtained by using large metal soaking blocks. These tended to inhibit the fluidizing of the powder around the workpiece, collapsing the bath either locally or completely.

(b) The furnaces leaked powder, which migrated to other work areas, got into places where abrasive dust was not welcome, and turned floors into sliding ponds.

(c) Some with air-assisted exhaust systems required large air flows (e.g., 125 L/min at 50 psig).

Very interesting, but more to the point: why was my fluidized calibration furnace giving me odd results?

Upon examining the intended operating cycle, I realized that the fluidization was not continuous, but instead was what was called the

"dead-bed" technique. The temperature of the fluidized powder was monitored until it was stable at the setpoint, when the air and heaters were turned off, collapsing the bed. This turns a bath with good heat-transfer properties into exactly the reverse. Therefore any immersed object was then surrounded by a thermal insulation at the same temperature as itself; a state said to allow isothermal and electrically noise-free periods suitable for calibration.

But calibrations couldn't be repeated, and an examination of the temperatures seen by the control thermocouples and the SPRT which was the subject of the calibration showed me why. During the dead-bed period the rates at which the thermocouples and the SPRT cooled was not the same. I could get repeatable calibrations only after I had modified the software controlling the operation, and was calibrating using continuous fluidization.

I still wasn't happy. The bath allowed powder to escape. It did not allow adequate immersion. It was not designed like conventional liquid calibration baths. It used more air on the powder recovery system (which didn't work adequately) than it did on fluidizing the powder. The air control system seemed complicated and inefficient. So I said to myself, as we all do from time to time, "I can do better than that!"

Now, bath development wasn't part of my job description, and so it had to be done at home. Our first baby had recently arrived, Dawn was tearing down walls between nappy changes, and there wasn't any spare money at all. With scrap stainless steel tubing from the local cement works, the shell of an old washing tub, an eagle eye out for any discarded thing which could be used, I built my first home fire hazard. It was crude, but it resolved most of the deficiencies I had identified in fluidized baths then available.

Many good liquid baths employ a concentric-tube scheme, in which the inner tube encloses the working space, and the next annular shell is the fluid return past the heaters. I used the same idea, spacing three 1 KW heaters between the working volume and the outer shell. I planned to force the powder up the center tube and down the satellite tube past the heaters, as in a liquid system, by some means I hadn't quite thought of yet. However I turned it on whilst I was thinking about this, and, as Henry likes to say, "physics worked"; a combination of the effects of the heaters and the ratio of the areas fluidized inside and outside the concentric tubes caused the powder to flow the other way 'round, and I left it like that. The tubes were long enough to allow a 450 mm (17.7 inch) immersion depth for thermometers in the working space. The concentric tubes help produce isothermal conditions around

the thermometers because there is no direct path to the heaters, and because there is time for proper mixing and thermal equilibration of the powder.

I very much wanted a sealed system, from which no powder could escape. Dawn frowned on any addition to our dusting load; but there were also technical reasons. A sealed system would allow finer powders to be used, which give better fluidization and heat transfer. A sealed system would allow the fluidization to be more vigorous; the best mixing, and thus the best condition for isothermality, occurs when "bubbles" pass up through the powder. I sealed the working space with a top plate, which also carried the wells for the thermometers under test. Waste air was taken away through an exhaust pipe and passed through a ceramic filter to trap entrained powder.

The most challenging problem, which eventually has the simplest solution, was the fluidizing air control system. It requires less air to fluidize the powder at high temperatures than at low, because the air expands when it enters a hot volume, and this must be countered by reducing the supply flow proportionally. There are, obviously, several ways this can be done:

- (a) Have an operator watch the fluidization, and adjust a valve
- (b) Include a stepped set of solenoid valves, operated by comparing the resistance of a resistance thermometer with that of a ladder of graduated resistance values, and closing that specific valve when a match is found
- (c) Space two resistance thermometers in the bath, with one in a self-heating condition. Let these be two legs of a control circuit which adjusts the fluidization for maximum heat transfer between them (indicated by minimum resistance difference).

But I was working at home, and needed a simpler solution. It eventually suggested itself. The air supply was first heated by passing it through a long tube immersed in the bath, and then forced through an orifice. If the pressure is kept constant above the orifice, the flow through it will remain constant, and so will the level of fluidization.

I borrowed some instruments from work, and set about measuring the thermal characteristics of my furnace. Using various formulae for setting up Proportional-Integral-Derivative values for the temperature controllers got me nowhere, so I used the "best guess" technique, and eventually found a set of values which held over the whole temperature

range from 50° to 700°C. Thermal surveys in the working volume showed that the temperature was uniform both horizontally and vertically within $\pm 0.1^\circ\text{C}$ at 100°C and $\pm 0.02^\circ\text{C}$ at 600°C. These measurements were carried out in a 9-tube probe holder 380 mm long, and were valid for the bottom 200 mm. The temperature in the tubes was hotter near the powder's surface, because of the direction of powder flow. Stability over a 10 minute interval was also $\pm 0.1^\circ\text{C}$ at 100°C and $\pm 0.2^\circ\text{C}$ at the bottom of the tubes. The gradient and stability figures made the furnace useful for thermocouple calibration, and since I did not need to use massive equalization blocks, the time was shorter than in other systems. It was during these studies that my furnace, made from scrap, reached its maximum temperature of 700°C, unquestionably signalled when the wooden top began to smoulder, and when Dawn, shortly afterward, did the same.

I was now very happy with my creation. I filed a patent application, which was promptly rejected for style. Then I met George Carcasson, a retired patent writer in his seventies, to mutual benefit: I taught him how to roll a kayak, and he wrote a successful patent application.

A friend suggested that I discuss my furnace with John Tavener of Isothermal Technology Ltd. John sponsored the first properly engineered version, made of fireproof materials, which is still in use, 10 years later, in my home lab. A second unit was built for a commercial customer in France, and is still in daily use. The bath then entered the Isothermal product line, with the name changed from "Ayres" to "Ayries", to better fit Isotech's style of product name. Over the years, the working volume has increased and the case and the controls have been improved. A recent change in design bears mention. Some customers have had problems with exhaust filters blocking. We have altered the filter assemblies to give far better performance, permitting also use of a finer powder, and all baths made in 1992 and later will have this change. Older furnaces can be updated easily to the new system.

The furnace was originally meant for thermocouple calibration, but is finding a new and significant use as a furnace for operating freezing point cells, using an equalizing block mounted with a top plate and re-entrant tube. Wolfgang Merz, Director of a calibration station of the German Calibration Service in Munich, has developed a completely automatic computer-controlled system for cycling cells [1].

Also, SPRTs may be calibrated by comparison. Isotech's NAMAS Laboratory uses one for comparison calibrations at 420°C and 660°C with

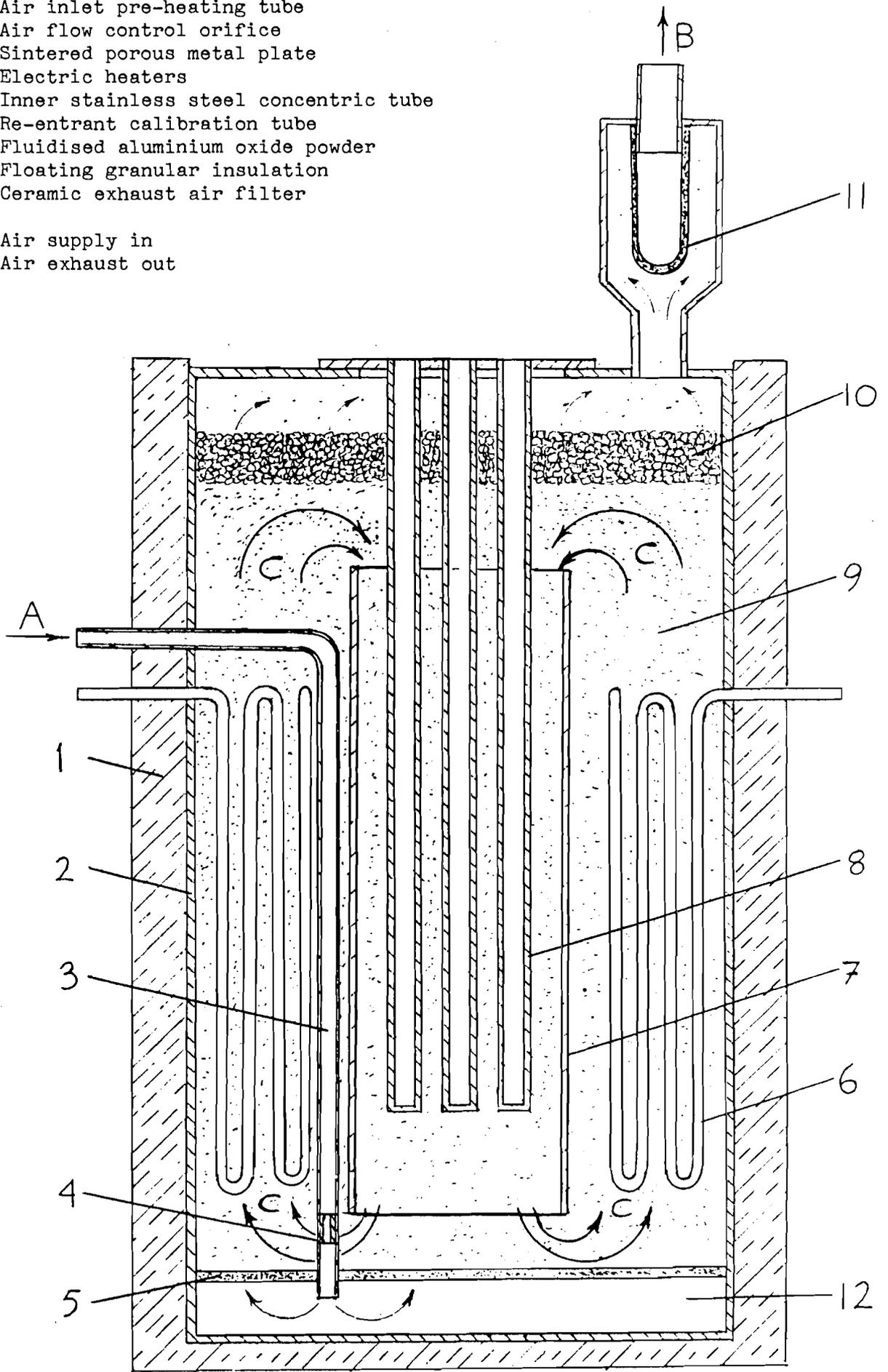
an uncertainty of $\pm 0.05^{\circ}\text{C}$. The furnace has few moving parts, and now that everything is made from best quality materials and components, the life expectancy is equivalent to that of the purchaser.

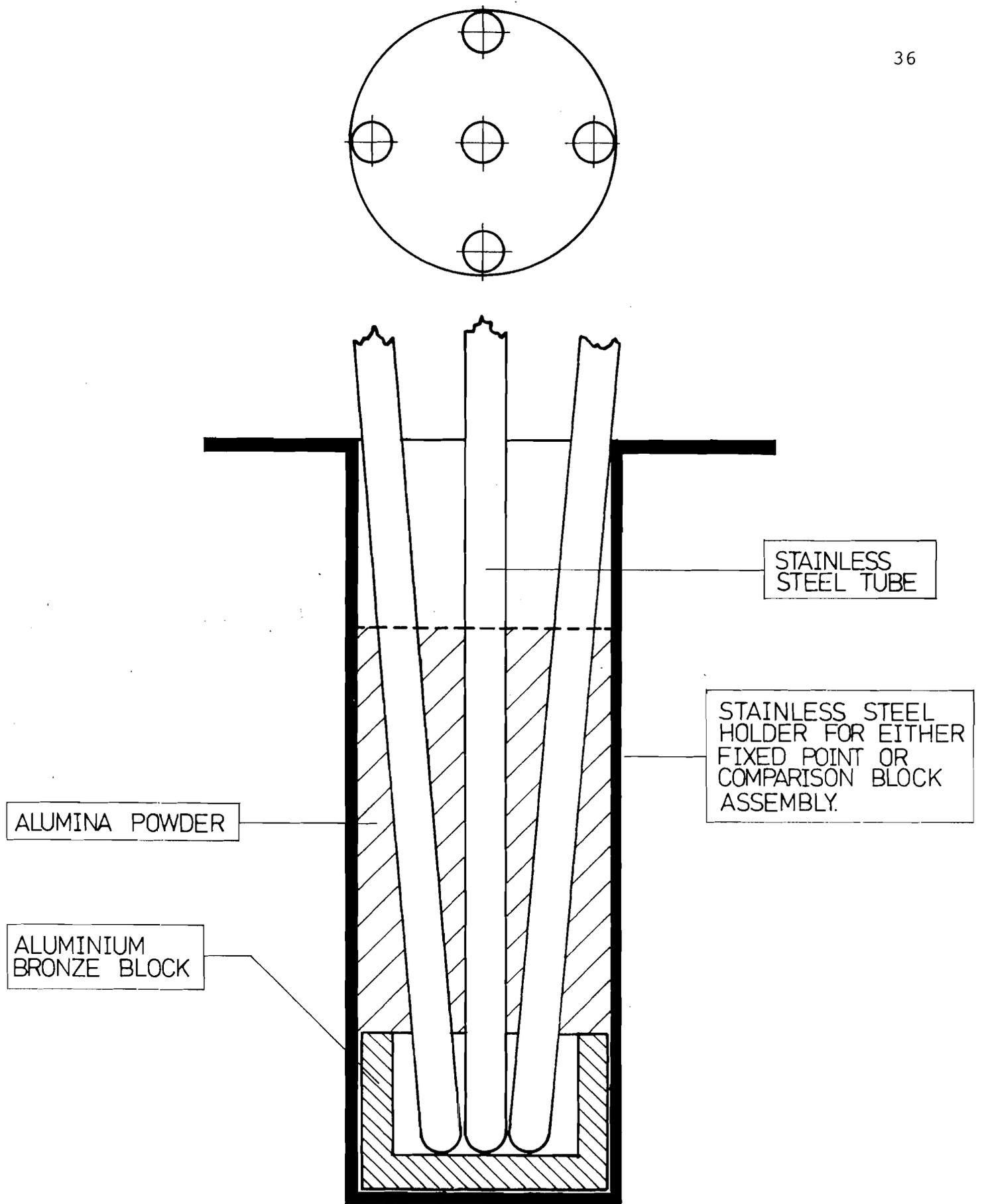
In 1986, after 9 years of labor for the large organization which is the Central Electricity Generating Board, I began to run out of enthusiasm. As in Neil Young's song "My Country Home", I had to think to smile. Dawn wanted to head west into the hills, and when John offered me half the salary and twice the fun, I responded with no resistance; to find that life can begin at 38, when you abandon the cool and anonymous shelter of a National Institution, and become part of The Family.

[1] Details are available from Wolfgang Merz, Merz Meßfühlertechnik GmbH, Bahnhofstraße 3, D-8024 Deisenhofen, Germany, or from Isotech

1. Ceramic insulation
2. Stainless steel case
3. Air inlet pre-heating tube
4. Air flow control orifice
5. Sintered porous metal plate
6. Electric heaters
7. Inner stainless steel concentric tube
8. Re-entrant calibration tube
9. Fluidised aluminium oxide powder
10. Floating granular insulation
11. Ceramic exhaust air filter

- A. Air supply in
 B. Air exhaust out





AYRIES BATH EQUALIZING BLOCK ASSEMBLY DIAGRAM.

THE WATER TRIPLE POINT AND GALLIUM POINT IN SECONDARY LABORATORIES IN GERMANY

By Peter Klasmeier

ABSTRACT

Many secondary Laboratories in Germany, such as those that operate calibration stations of the German Calibration Service, or those that function as the measurement base for public utilities, are required to conform to regulations regarding their practice and procedures. These Laboratories are required to maintain the water triple point, and find it convenient to maintain the gallium point, in support of their calibration systems. However these are not used at frequent intervals, and can be realized in simpler and less costly equipment than in primary laboratories. This paper describes the use of the Isotech OCEANUS dry calibration bath with water triple point and gallium melt point cells.

DISCUSSION

In Germany, the National Laboratory, responsible for maintaining the International Temperature Scale, is the Physikalisch-Technische Bundesanstalt (PTB). Like many European National Laboratories, and unlike the United States practice, the PTB does not calibrate or recalibrate all thermometers in the hands of users, but, as a generality, calibrates only thermometers owned by the secondary laboratories which serve commercial and industrial users.

One such group of secondary laboratories are those accredited as calibration stations (Kalibrierstellen) of the Deutsche Kalibrierdienst (German Calibration Service; DKD). DKD Laboratories are accredited and supervised by the PTB, and are authorized to perform specific ranges of calibrations for users, and to issue legally-recognized calibration certificates. Usually the DKD Laboratories maintain appropriate fixed points of the ITS-90. (Many other countries have equivalent calibration services; for example, NAMAS, in England. Isothermal Technology is a NAMAS Laboratory).

Another such group of secondary laboratories are those that maintain the quality and accuracy of temperature measurement for public utilities. In many newer cities and towns of Germany, heat to buildings and homes is supplied from a central heat-generating facility, and

distributed, usually as steam [1]. The distribution system is known as the Fernwärmedienst, or remote heating service. Accuracy of temperature measurement is required for the computation of heat use by the subscriber, who is charged on this basis. The range of calibrations performed by the Fernwärmedienst Laboratories is commonly limited to 0° to 140°C.

Both types of Laboratory are required to maintain the absolute thermometric standard represented by the water triple point, as a reference base for their working thermometers. The water triple point alone does not provide an assurance of calibration slope; two points at least are required for that; and with the introduction into common use of the melting point of gallium, a slope assurance can be obtained.

The water triple point is the equilibrium state of pure water in its liquid and solid phases under its own vapor pressure, and is 0.01°C, with an uncertainty of less than 150 μ° [2]. It is established in a familiar pattern of glass cell, which contains high-purity water sealed under its own vapor, and provides a re-entrant well for the thermometer under test. To prepare the cell for use, a portion of the water is frozen into the solid phase, and in traditional practice, this is done by introducing into the thermometer well a charge of liquid nitrogen or crushed dry ice. This procedure is tedious to carry out, and requires liquid nitrogen or dry ice as consumables. Therefore it is not ideally suited to Laboratories that prepare and use their cells only occasionally.

A method described by the National Physical Laboratory of England (the "slush" method") is simpler for these Laboratories. Like all pure materials, pure water will exhibit undercool; that is, when cooled it will remain in its liquid phase at temperatures below that at which it would melt if it were in the solid phase. In the slush method, the water in the cell is cooled as a liquid to -6°C. When the cell is removed from the cooling environment and shaken, solid crystals form immediately throughout the liquid. If the cell is returned to the cooler and held at -6°C for another hour, a solid mantle of ice will form around the thermometer well. If the cell is then placed in an environment at 0.1°C, the triple point equilibrium can be maintained for several days. When the cell is to be used, the ice mantle is freed by inserting a warmed rod into the thermometer well, so that a thin film of liquid forms between the mantle of ice and the surface of the well.

The water triple point, as a calibration reference, is important to the secondary laboratory whether or not it is required by National law.

First, it is the denominator of the ratio W in which thermometer calibrations are reported:

$$W = R(\text{at temperature}) / R(\text{at water triple point})$$

and, as such, is necessary for the most precise use of the reference thermometer tables. Best practice is to measure the thermometer resistance at the water triple point immediately after each measurement of resistance at another temperature, and use these results in the W equation. Second, a continuing record of the resistance at the water triple point, maintained on a control chart, is an excellent quality control check on the stability of the reference thermometer.

The Isotech Oceanus dry-block bath is the ideal environment in which to perform the cooling and maintenance functions. A temperature range of -15° to $+75^{\circ}\text{C}$ is accomplished by Peltier solid-state modules, used for cooling and for heating. A microprocessor-based digital controller with a displayed resolution of 0.1°C , and with optional RS422 communications, establishes accurate and repeatable temperature levels.

The melting point of gallium provides a second absolute temperature reference at 29.7646°C , with an uncertainty of about $200 \mu^{\circ}$ [3]. The gallium metal in the cell is first assured to be in the solid state, by immersing the bottom of the cell in crushed ice to a depth of 4 to 5 cm. This limitation of depth assures that the gallium will be frozen from the bottom of the cell upward; a necessary precaution, since gallium expands as it freezes. The frozen state can be demonstrated by measuring the temperature in the thermometer well, which, when the gallium is solid, will be in the vicinity of ambient temperature and well below 29°C .

The frozen cell is then placed in the Oceanus block at a controller setting of 30.1°C . As the cell warms it will reach the temperature where the gallium begins to melt, at which point the liquid-solid equilibrium has been achieved. In the Oceanus block, the melting equilibrium can be maintained for a number of hours.

Full realization of the ITS-90 to higher temperatures requires, of course, additional prescribed fixed points; the freezing points of indium, tin, zinc, aluminum, and for the full platinum resistance thermometer scale, silver. However, because of the extreme certainty of the water triple point and the gallium melting point, used in Oceanus, it is possible to use only these two points in the interpolation equation, and extrapolate to 660°C with an uncertainty of less than 10 mK [4]!

The Oceanus dry well bath, combined with a water triple point cell or a gallium melt point cell, is the only apparatus necessary for the realization and maintenance, in the secondary laboratory, of two fundamental and defining fixed points of the ITS-90; absolute calibration standards. It is achieving wide use in this context, and has proven to be a versatile and economical apparatus.

Also, the Table of Footnote [4] below shows that, over much of the range of ITS-90, calibrations of thermometers within what may be industrially-acceptable limits of error can be obtained using only the water triple point and the gallium point.

[1] Remote heating of a number of buildings by steam from a central plant has been a commonplace in the commercial building areas of New York City for at least 50 years. I have also encountered this system in the newer cities of The Netherlands, that are built on land reclaimed from the Zuider Zee (HES).

[2] For a complete discussion of the water triple point, see *Isotech Journal of Thermometry*, Vol. 1 No. 1 pp 23-24 and Vol. 1 No. 2 pp 63-64.

[3] For a complete discussion of the gallium melting point, see *Isotech Journal of Thermometry*, Vol. 1 No. 1 p 24, and Vol. 1 No. 2 pp 59-60.

[4] In the Table which follows, Column 2 has been developed by calculating a table for an ideal thermometer, using the ratios which are the values of the reference function given on ITS-90 at the triple points of argon and mercury, the water triple point, and the freezing points of tin, zinc and aluminium, and employing the ITS-90 interpolation algorithm. Column 3 has been developed by using the ITS-90 algorithm given for the range 0°C to the melting point of gallium, and two fixed points only; the water triple point adjusted so that its resistance value is 120 μ K lower than nominal, and the gallium melting point adjusted so that its resistance value is 120 μ K higher than nominal, to represent readings in error by those amounts. Column 3 thus represents a very long extrapolation of the thermometer calibration in both directions, which includes the effects of these uncertainties of the two fixed points. Column 4 shows the differences between the extrapolated table of Column 3 and the ideal table of Column 2 in terms of ratio. Column 5 expresses these differences as Celsius degrees.

1	2	3	4	5
TEMP	ITS-90	WA-GA	WA-GA MINUS	WA-GA MINUS
DEG C	RATIO	RATIO	ITS-90 RAT	ITS-90 DEG C
-200	.169754	.175975	.006221	.68665
-160	.342638	.354401	.002763	.12435
-120	.511547	.512280	.000733	.04926
- 80	.676790	.676901	.000111	.00969
- 40	.938436	.839431	-.000005	-.00050
0	.999960	.999960	.000000	.00000
40	1.158530	1.158535	.000005	.00005
80	1.315171	1.315181	.000010	.00105
120	1.469898	1.469913	.000015	.00168
160	1.622724	1.622744	.000020	.00213
200	1.733663	1.773688	.000025	.00268
240	1.922729	1.922758	.000029	.00313
280	2.069933	2.069967	.000034	.00368
320	2.215285	2.215323	.000038	.00411
360	2.358789	2.358832	.000040	.00463
400	2.500441	2.500488	.000047	.00504
440	2.640233	2.640285	.000052	.00551
480	2.778149	2.778205	.000056	.00587
520	2.914168	2.914229	.000061	.00632
560	3.948268	3.048332	.000064	.00654
600	3.180425	3.180493	.000068	.00690
640	3.310618	3.310691	.000073	.00736
680	3.438842	3.438909	.000077	.00755

The ITS-90 calculations were done using the Isotech "Daedalus" MS-DOS program for interpolating ITS-90.

ISOTECH'S COMMENT ON OCEANUS, ETC.

by John P. Tavener

I should like to put the Oceanus dry-well bath into the context of the apparatus that Isotech manufactures for actuating the triple point of water and gallium cells.

All Laboratories involved in temperature measurement have, as their reference standards, Standard Platinum Resistance Thermometers (SPRTs), which are the prescribed interpolation standard of the International Temperature Scale of 1990 (ITS-90), and most maintain secondary platinum resistance thermometers or high-precision thermocouples as working standards. Any of these standards must be checked periodically to ensure that their characteristics have not changed through time, use or accident. This check is usually performed by measuring and logging the resistance (or emf) at the triple point of water, 0.01°C. For SPRTs, the ITS(90) Document stipulates also that the ratio of the resistance at the gallium melting point, 29.7646°C, to the resistance at the water triple point, W:

$$W = R(\text{gallium point}) / R(\text{water triple point})$$

must be equal to or greater than 1.11807; it is convenient, therefore, to include the gallium point in the periodic check, as well as to have it available for calibration purposes as described in the preceding paper by Peter Klasmeier.

We manufacture apparatus for realizing and maintaining the water triple point and the gallium melting point at three levels, for three distinct sets of users.

1: For Primary and National Laboratories, we offer two pieces of apparatus.

For the water triple point, our Water Triple Point Maintenance Bath, Model 18233, is a free-standing top-loading chest, that will hold up to four water triple point cells of the NPL design (Isotech Model 811) or of the NBS design. It is thermoelectrically cooled, and, with occasional check, will maintain the water triple point for many months without renewing the ice mantles. Its main advantages are its very considerable stability ($\pm 0.001\text{K}$) and the safeguards built in to protect the cells. For the Laboratory which uses the water triple point frequently, it is de-

lightful to have this most fundamental fixed point available at any moment, without further preparation.

For the gallium melt point, we offer our Gallium Cell, Model 17401, and Gallium Standard, Model 17402; an automated melt environment in which to cycle the cell. The melt plateau can be achieved within half an hour of turn-on (which can be done by a timer before the Laboratory day begins), the melt point kept available throughout the day, and the melt plateau recycled for the next day. The unit is benchtop, and also contains built-in protection for the cell.

2: Many smaller Laboratories require a check of the SPRT or working standards less often, say, once or twice a month. For them, simpler and more economical approaches may be better.

For these Laboratories, we have designed two pieces of apparatus:

(a) The Oceanus dry-block system, described in Peter Klasmeier's paper, which will house one large water triple point cell of the Isotech 811 or the NBS pattern, and (not simultaneously) 1 Isotech Model 17401 Gallium Cell. Oceanus will create the conditions necessary to realize and maintain the equilibrium condition in these cells for a period of a few hours or more. Both cells will accommodate SPRTs with adequate stem immersion depth. Oceanus requires a small flow of tap water for cooling.

(b) The Hyperion dry-block system has been designed to accommodate a class of smaller water triple point and gallium cells, the Isotech Slim Cells. Because of the reduced thermometer well depth of Slim Cells, SPRTs will show a noticeable (1 to 2 mK) stem conductance error, but Slim Cells are ideal for smaller and shorter thermometers; industrial PRTs, thermocouples, and thermistors. Again, the equilibrium condition can be realized and maintained in these cells for a period of a few hours or more. Hyperion is air-cooled and completely self contained.

Both Oceanus and Hyperion can be used with removable insert blocks which are intended for comparison calibrations.

COMPARISON CALIBRATIONS AT THE BOILING POINT OF NITROGEN (OR ARGON)

by Henry E. Sostmann

ABSTRACT

Although the International Temperature Scale of 1990 stipulates calibration of a Standard Platinum Resistance Thermometer at the triple point of argon ($-189.3442^{\circ}\text{C}$), the realization of this calibration point can be costly and complicated. An alternative, employed by many Laboratories including National Laboratories, is a comparison calibration in boiling nitrogen or argon. The rationale, method and probable uncertainty of such a calibration is discussed here.

COMPARISON VERSUS ABSOLUTE CALIBRATION AT THE COLD END OF THE LONG-STEM SPRT RANGE

Interpolation tables of resistance ratio versus temperature for long-stem Standard Platinum Resistance Thermometers (SPRTs) usually begin no colder than -200°C . The defining fixed point of the International Temperature Scale of 1990 (ITS-90) in this vicinity is the triple point of argon, $-189.3442^{\circ}\text{C}$.

In theory, the triple point of argon is a simple point to establish. It has been realized in sealed cells by Pavese, Bonnier, Furukawa and others, particularly for capsule thermometers. Sealed cells have the drawback that, at room temperature, they exist under relatively high pressure (e.g, 3000 psi), and must be regarded as pressure vessels in handling and for transportation. Also, the cryostat required is not simple, and the prevention of heat transfer via the cell mountings and the tubes of long-stem thermometers must receive special attention. In practice, the realization of the argon triple point can be costly and complicated.

Most laboratories will elect to calibrate at this end of the SPRT range by the simpler and less costly comparison method. In this method, a thermometer under test is compared to a thermometer of known calibration; usually a calibration obtained from a National Laboratory. Indeed, many National Laboratories will calibrate thermometers submitted to them by comparison with their own thermometers, realizing the argon triple point itself only infrequently, and only for the calibration of their own reference SPRTs.

This policy has been announced by the National Physical Laboratory of England, in its publication "Adoption of the ITS-90", as follows:

"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."

Other National Laboratories will follow similar practices.

THE EFFECT OF PRESSURE (E.G., LABORATORY ALTITUDE) ON THE NITROGEN AND ARGON BOILING POINTS

The normal boiling point of nitrogen is -195.794°C ; a mere 6.45 K below the argon triple point. The normal boiling point of argon is -185.88°C . However, since these are two-phase (liquid-vapor) equilibria rather than triple points, temperature will vary with pressure, for example as a function of laboratory altitude above standard sea level. Table 1 shows the variation of the nitrogen and argon boiling temperatures with altitude.

For this Table, the pressure-temperature relationship has been computed from the Antoine vapor pressure equation

$$t = (B/(A - \log p)) - C \quad \text{Eq. 1}$$

in which, for nitrogen, $A = 6.4946$, $B = 255.68$, $C = 266.558$, and for argon $A = 6.6165$, $B = 304.277$, $C = 267.328$. The temperature at which a pure liquid boils is that temperature at which the vapor pressure and the ambient pressure are equal.

THE METHODOLOGY OF COMPARISON CALIBRATION

Comparison calibration is done by placing two thermometers in an isothermal situation at an approximately known temperature. One of these thermometers (the standard) must have a known calibration (such as is obtained from a NAMAS or National Laboratory), and a table of calibration values which includes the range under consideration.

Method 1: After the two thermometers have reached a condition of thermal equilibrium with the bath, the resistance of the standard thermometer is measured, the resistance of the unknown is measured, and the resistance of the standard is measured again, to assure that no change in temperature has taken place (due, for example, to a possible change in ambient pressure). The temperature of the bath, t_b , is then

determined from the measured resistance and the calibration table for the standard, and the measured resistance of the unknown at the same temperature is said to be its resistance at t_b .

TABLE 1			
THE BOILING POINT OF NITROGEN AND ARGON AT VARIOUS LABORATORY ALTITUDES			
Feet of Altitude	Pressure mm Hg	Nitrogen boils °C	Argon boils °C
-1000	787	-195.50	-185.54
0	760	-195.80	-185.88
1000	733	-196.11	-186.22
2000	707	-196.41	-186.56
3000	681	-196.72	-186.90
4000	656	-197.02	-187.24
5000	632	-197.33	-187.58
6000	609	-197.63	-187.92
7000	586	-197.93	-188.26
8000	564	-198.24	-188.60
9000	543	-198.53	-188.94

(Pressure-Altitude data from "Pressure-Altitude Tables Based on the United States Standard Atmosphere", W. Brombacher, NACA Report No. 538, 1948)

Method 2: There is actually a simpler and more satisfactory way to perform this comparison, if there is available a resistance measuring bridge or instrument which has terminal positions provided to accommodate external connections for an unknown thermometer and also for a standard or reference resistor. (Examples of such bridges are the Guildline Model 9975 and the Automatic Systems Laboratories F-16, F-17, F-18, etc.) This method has the additional advantage that the measurements are simultaneous, so that any error due to change in temperature over a short interval of time is eliminated. Using such a bridge, the standard thermometer is connected in place of the standard resistor, and the unknown thermometer in place of the unknown. The bridge will then read the ratio of the unknown to the standard directly:

$$W = R_u / R_s \quad \text{Eq. 2}$$

In this method, we never obtain the temperature of the bath, but it is not necessary that we do so; we can assign a temperature arbitrarily. The temperature in a bath of boiling liquid nitrogen or argon

will be within a degree or so of the nominal boiling temperature at the laboratory altitude under any reasonable conditions of barometric pressure, etc.

Assume that the ratio $R_u / R_s = 1.000012$, as determined in a bath of boiling nitrogen, and the resistance of the standard R_s at -196°C is 4.77602 ohms, read from its calibration table. (The choice of -196°C for this calculation is almost arbitrary; however, we choose the nominal integral value which is closest to the circumstances, and at sea level this temperature would be -196°C for nitrogen, and -186° for argon. At 7,000 feet of altitude, it would be -198°C for nitrogen, and for argon at 7,000 feet it would be -188°C .) We may then say that the resistance R_u at -196°C is $1.000012 \times 4.77602 = 4.77608$ ohms; and this statement is valid within acceptable limits *even if the temperature at which the ratio is measured is actually -197° , or -195°C , or any temperature within a few degrees of -196° , and even if we do not exactly know what that temperature is.*

What makes this statement possible is that the correspondence between the slopes and curvature of the characteristic calibrations of thermometers made from high-purity platinum wire, and qualified as interpolation standards for ITS-90, is very close.

To confirm this, I did some calculations on the correspondence between 17 thermometers calibrated at various times. The thermometers represented a fair selection of Leeds and Northrup 8163 and 8167 thermometers, of various ages and provenance. The reference standard for each comparison was one of two Leeds and Northrup Model 8163 thermometers having long and honorable histories and current NIST calibrations.

Each of the 17 thermometers was calibrated relative to the standard thermometer at the boiling point of oxygen (it might equally have been done at the boiling point of nitrogen, or of argon) by using the standard thermometer as the standard resistor and the unknown thermometer as the unknown resistance of a Guildline Model 9975 Precision Current Comparator. In addition, each unknown thermometer was calibrated at two fixed points of the ITS-90, the mercury triple point and the triple point of water, and a table calculated from this data at intervals of 1°C over the range from -200° to -179°C , using the Isothermal Technology Daedalus MS-DOS interpolation program.

From the table of resistance ratio for each thermometer, for each 1°C interval between -200° and -179°C , I calculated the increment of resistance ratio, and then the difference in increment for the same tem-

perature interval for each of the thermometers. Table 2 provides a summary of the results. (The detail is available, and will be sent to any reader on request). From this information, it was possible to derive the mean and range of increment for the group at each temperature, and the standard deviations at each temperature in terms of increment and of temperature. I then estimated the maximum errors to be expected if a nominal comparison temperature of -196° or of -186° were actually different from these values by enough to cause an approximately 1mK error.

1 TEMP DEG C	2 MEAN INCR	3 RANGE INCR	4 2 STD DEV INCR	5 2 STD DEV DEG C	6 DIVERG DEG C
-200	.004304	.000003	.0000015	.0003665	
-199	.004310	.000004	.00000174	.0003773	.00115
-198	.004316	.000004	.00000172	.0003849	.00077
-197	.004322	.000003	.00000150	.0003896	.00039
-196	.004326	.000004	.00000177	.0003917	.00000
-195	.004333	.000003	.00000165	.0003915	.00038
-194	.004333	.000004	.00000177	.0003894	.00077
-193	.004336	.000003	.00000162	.0003856	.00115
-192	.004338	.000004	.00000193	.0003805	
-191	.004340	.000003	.00000137	.0003743	
-190	.004341	.000003	.00000150	.0003674	
-189	.004342	.000003	.00000140	.0003601	.00106
-188	.004342	.000003	.00000165	.0003526	.00071
-187	.004342	.000003	.00000158	.0003452	.00036
-186	.004342	.000003	.00000154	.0003384	.00000
-185	.004341	.000003	.00000165	.0003323	.00033
-184	.004340	.000002	.00000118	.0003273	.00065
-183	.004339	.000002	.00000139	.0003238	.00087
-182	.004338	.000003	.00000150	.0003219	.00130
-181	.004337	.000002	.00000112	.0003220	
-180	.004334	.000003	.00000160	.0003244	
-179	.004333	.000003	.00000140	.0003294	

KEY TO TABLE 2: Col 2 shows the mean increment of resistance ratio for temperatures at 1°C intervals; Col 3 the range for 17 thermometers of Col 2; Col 4 the 2σ standard deviation for 17 thermometers in resistance ratio; Col 5 the 2σ standard deviation in $^{\circ}\text{C}$. Col 6 shows the maximum 2σ deviation between a test thermometer and a standard thermometer at 1°C intervals when (line -196) comparator temperature is nominally -196°C and (line -186) comparator temperature is nominally -186°C . The data of

Columns 5 and 6 have been smoothed by the method of least squares. The results would be approximately the same when the difference is measured against some other reference; e.g., boiling nitrogen at -188°C at a station altitude of 6000 feet.

CONCLUSION AND QUALIFICATION

It is clear from Column 6 of Table 2 that errors on the order of 1 mK are the limit of error, because of differences in the response of thermometers, for cryostat temperatures which differ from the nominal temperatures by $\pm 3^{\circ}\text{C}$. The limits of error for nominal temperatures other than -196°C and -186°C can be estimated by using Column 5 of Table 2.

It should be noted that this study has been limited to standard platinum resistance thermometers of Meyers type, manufactured by Leeds and Northup or YSI as Types 8163 and 8167. It is not expected that other standard platinum resistance thermometers, meeting the criteria of ITS-90, would exhibit substantially different correspondence, and this paper will be amended to include such thermometers as the data becomes available. If there is any doubt, then Method 1 is the measurement technique of choice.

When comparison methods are used with other thermometers, such as industrial platinum resistance thermometers, thermocouples, etc, that may either have characteristic curves quite different from those of SPRTs, or that may be based on an entirely different transfer principle, Method 1 above is the measurement technique of choice. Each situation should be evaluated on its own.

AN APPARATUS FOR COMPARISON CALIBRATIONS AT THE BOILING POINT OF NITROGEN OR ARGON.

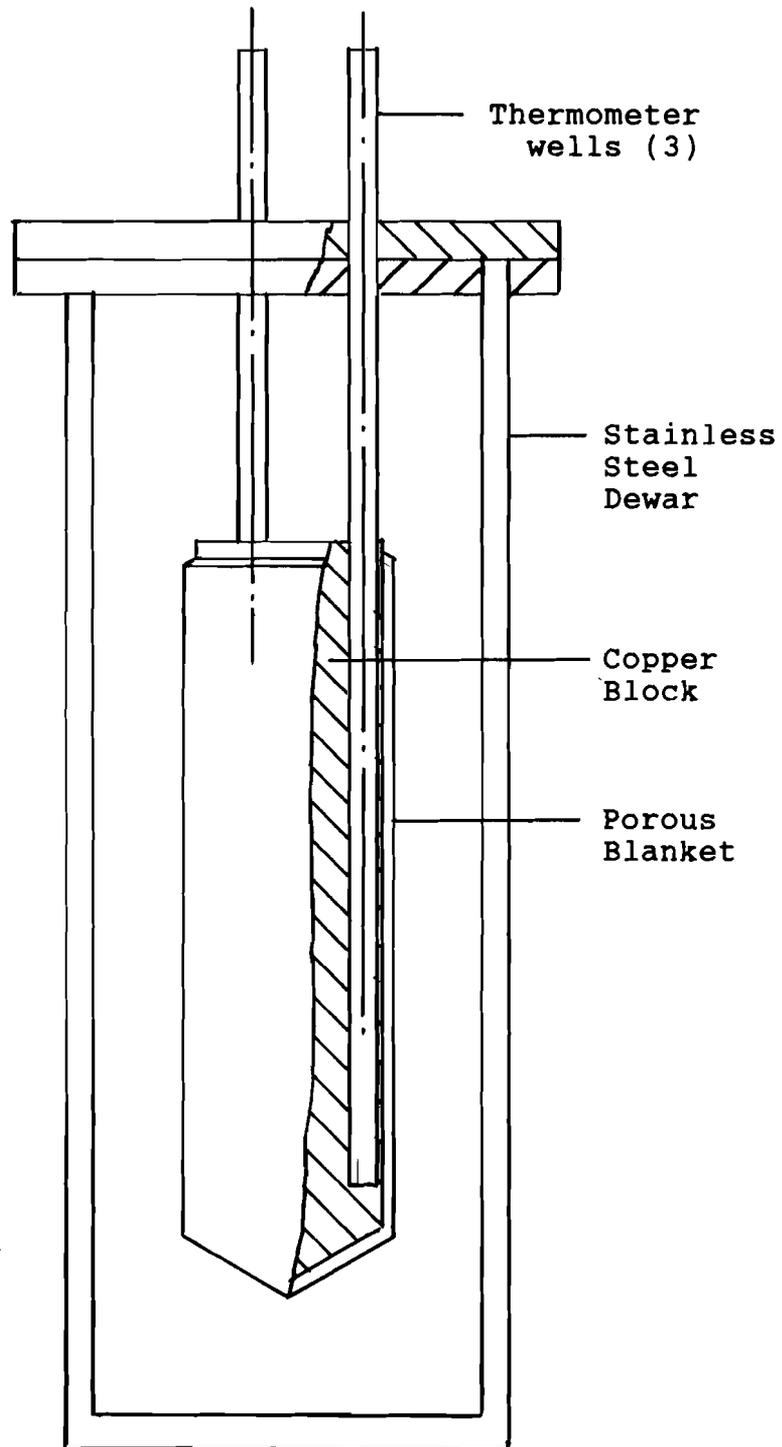
Fig. 1 shows a sectional drawing of an apparatus (Isothermal Technology Model ITL-18205) for comparison calibration of long-stem platinum resistance thermometers at the boiling point of nitrogen. It may be used also at the boiling point of argon, but most users will choose nitrogen on the basis of its ready availability and low cost.

(The user is cautioned NOT to use this apparatus with liquid oxygen, unless the porous blanket is removed and the system is properly cleaned for oxygen service. The danger of fire, explosion, etc., is otherwise high. Furthermore the operator must recognize and be trained to cope with the hazards of storing, transferring, and otherwise handling any cryogenic liquid, including liquid oxygen, argon, nitrogen, etc. as appropriate).

Referring to Fig. 1, B indicates one of three thermometer wells equally spaced on a diameter sufficient to accommodate SPRT heads. These wells project into a relatively massive block of tellurium copper, E, which equilibrates temperature between the wells. The block is surrounded by a porous blanket, D, which provides a very large number of points for nucleation of the cryogenic fluid. Not shown in the illustration are (a) the port, in the dewar cover, for filling with liquid cryogen (b) a float liquid level indicator (c) a vent to atmosphere, which if desired may be connected to a conduit for exhaust gas (d) an automatic and preset pressure relief valve and (e) a pressure gage with which the internal gas pressure may be monitored. Since the internal pressure should be atmospheric pressure, (d) and (e) are safety features, should the exhaust vent become plugged. (The manufacturer states that the dewar will withstand about 3.5 atmospheres of internal pressure. Isotech has not confirmed this statement by destructive testing and makes no warranty for it).

In use, two or three thermometers are placed in the wells B. One of these is an SPRT or other thermometer with a valid calibration print-out; the other one or two are thermometers to be calibrated. The thermometers may be sealed into the wells by means of Swagelock fittings with reusable Teflon ferrules, which allows the well space to be evacuated and filled with helium at the user's option. At low temperatures, helium has a much better thermal conductance than air (about 13 times higher) which improves the coupling of the thermometers to the block. Time to equilibrate, starting from a system at ambient temperature, is about 1/2 hour from the time that the liquid cryogen is introduced.

FIGURE 1



A CAUTION FROM PHILIP METZ

We asked our new associate, Phil Metz, Manager of Isothermal Service (USA), if he had any single trenchant bit of advice for users of SPRTs, and he gave us the following:

"I've been supervising calibrations of SPRTs at fixed points for over 10 years at YSI. In this time I've had many conversations with customers for calibrations, and I usually make a point of finding out how they are using their thermometers. I think it's to everyone's interest that an SPRT be used correctly, because there are some subtleties that will produce the wrong answer if they are not observed, and it is seldom obvious to the operator.

"I find that over the past few years, there has been a significant increase in gross error due to excessive measurement current through the thermometer.

"The problem is that many of the newer 7 and 8 digit digital multimeters look like an ideal choice for resistance thermometry, from the economics of purchase and also laboratory time. But when measuring a 25.5 Ω SPRT, most of them pass something like 10 mA through the thermometer coil.

"I've done a few experiments with retired SPRTs - ones I could risk - and a Guildline 9975, which allows a choice of measuring current. 10 mA heats an SPRT about 125 mK. Since it's an I^2R function, and 1 mA heats about 1 mK, that's about what one would expect. This large error can go unnoticed where there is no fundamental reference, such as a fixed point. There is also some reason to believe that 10 mA may cause permanent damage to a 25.5 Ω SPRT.

"Resistance measuring equipment intended for SPRTs generally uses 1 mA, or allows for several currents so that zero-power resistance can be calculated. The problem arises from general-purpose instrumentation, and particularly DMMs, where the current is not known and its effect not evaluated.

"This should seldom be a problem in a calibration laboratory, but is a matter well worth discussing with users of our calibrations"

NEWS OF A NEW METROLOGY SOCIETY

We have received news of the formation of the foundation of the Slovak Metrological Society at a Congress held in DT Zilina in October, 1990. The Society's purpose is to assist in the development of metrology in Slovakia; its directing and coordinating body is the Czechoslovak Institute of Metrology (CSMU, Ceskoslovensky Metrologicky Ustav, Bratislava,) which is responsible for the main focus of scientific metrology in Czechoslovakia. The Society will place emphasis on metrology as a tool for ensuring production and service quality, and on cooperation with such organizations as the West European Calibration Cooperation (WECC), CEN/CENELEC, and the activities of ISO and IEC. The complex issues of training metrologists in Czechoslovakia will also be addressed.

At the Foundation Congress, management and administrative details were approved, and a committee of 15 members chaired by Dr.-Ing. Marian Weidlich were elected.

We wish the SMS all good things, and understand its importance in the current transformation of Slovakia to a market economy and ultimate status in the community of Western Europe and the world. Anyone interested in membership, cooperation or sponsorship activities, is invited to write to Associate Professor Dr-Ing. Josef Mandák, Slovak Metrological Society, Tr. L. Novomeského IV/487, 842-55 Bratislava, Czechoslovakia.

(If I may add a personal note: I have been acquainted with the CSMU for perhaps 15 years, and have admired the dedication and qualification of its scientific personnel, and their ability to do world-class work with limited means. I cherish the recollection of the collaboration of Dr-Ing Milan Borovicka, Chief, Temperature and Radiation, and myself as we did our part toward the establishment of the gallium melting point as a defining point of ITS(90). I am proud to mention that I have been elected the first international member of SMS!- HES)

It seems appropriate here to continue our series of historic reprints with the paper Borovicka and I published in the Review of Scientific Instruments, Vol. 55 No. 10, October, 1984, on the melting and triple point temperatures of gallium. Please remember that the results are reported on IPTS(68); ITS(90) had not yet been formulated.

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The liquid–solid (melting) and liquid–solid–vapor (triple point) phase equilibrium temperatures of pure gallium have been realized and studied as proposed secondary reference points of the International Practical Temperature Scale. The melt temperature is found to be 29.771 94 °C (302.921 94 K) and the triple point temperature 29.773 88 °C (302.923 88 K) for gallium 99.999 99% pure. The technique and apparatus for these realizations, suitable for the calibration of high-precision thermometers, are described. These calibration points are finding important uses in medical, biological, ecological, and energy-source thermometry.

INTRODUCTION

The definition of temperature on the International Practical Temperature Scale (IPTS)¹ and the realization of temperatures at which to calibrate thermometers depend upon the establishment of the temperature values of thermometric fixed points. Fixed points are phase equilibria of pure materials, where temperature is invariant and is a natural constant of the material.

Defining fixed points of the IPTS are chosen to be as few in number as is consistent with determining the characteristic coefficients of interpolation instruments specified in the IPTS. Many fields of scientific investigation require thermometers which cannot be calibrated at defining fixed points—because greater accuracy is needed over a limited range, because the permissible range of the thermometer may not include the defining points, or because the mathematical transfer function of the thermometer may not be analytic or monotonic over the range of the defining fixed points. Examples are thermistors and liquid-in-glass thermometers.

These considerations have led to the study of additional equilibria, called secondary or reference fixed points. While the temperature values of the defining points are assigned and considered to be absolute, the temperatures of reference points are established by measurement.

There is particular reason to establish reference fixed points in the near-ambient region. These temperatures are important in medical, biological, environmental, energy-source, etc., research. The gallium points are of particular interest because they are close to 30 °C and only 7 °C away from human body temperature.

For example, the international standard reporting temperature for enzyme studies is 30 °C. Enzyme reaction rates may depend upon temperature as much as 10% per °C, but are typically carried out in spectrophotometer cuvettes which can accommodate only small thermistor thermometers. Immediate calibration of these thermometers at one of the gallium points effectively eliminates temperature uncer-

tainty as a potential source of error.

In the study of energy available from the temperature difference between warm surface sea water and cold deep-layer water, a thermometer of choice is the quartz-crystal thermometer. These thermometers are prone to drift, and it is convenient to reset them frequently at a gallium point.

The gallium points are a good reference for thermometers used to evaluate the heating of natural water by power-station effluent, and in the calibration of inertial guidance systems. The list could be extended indefinitely.

I. REQUIREMENTS FOR A REFERENCE POINT OF THE IPTS

To meet the requirements of the IPTS, any secondary or reference point must satisfy the following criteria²:

- (1) The equilibrium temperature must be highly reproducible. The material must be pure and physically stable. The material must be generally available in suitable form.
- (2) The attributed value of temperature must be measured on the IPTS. A number of determinations, using a number of samples of the material, is desirable.
- (3) A statement of experimental uncertainty must be given for the attributed value of temperature.

This report deals with research toward the precise determination of the melting point and triple point temperatures of pure gallium.

II. THE METAL

Gallium is an elemental metal of low vapor pressure and the longest liquid range of any metal (boiling point ca. 2400 °C). It is stable and does not oxidize readily. It is non-toxic. During solidification from the liquid phase it increases in volume by about 3.2%. When the temperature of the liquid metal is reduced, it can remain liquid as much as 70 °C below the freezing temperature (supercool).

III. THE CRUCIBLE

The crucible in which we contained our gallium is shown in Fig. 1. It is made of Teflon, which is flexible enough to accommodate the solidus expansion. Also, the interface free energies are such as to assist in nucleating the molten metal with only moderate supercool. The joints of the crucible are made to be pressure tight so that the crucible can be evacuated or filled with a protective atmosphere. A Teflon thermometer well is provided. The inside diameter of the well is 9 mm. The crucible was designed to contain 570 g of gallium.

Prior to filling, the crucible is thoroughly cleaned by degreasing with tetrachloride and then soaked in 1N HCl to remove metal ions. The cleaned parts are rinsed with copious distilled water and air or oven dried. During assembly any joints which do not have O rings are sealed with Cenasil silicone rubber. When the seals have set, the cell is evacuated repeatedly and washed with argon.

High-purity gallium is supplied in polyethylene squeeze bottles having long tapered tips. Two tubulations are provided in the head of the crucible. The bottle containing gallium, liquid above 30 °C, is connected to one of these tubulations and the gallium is forced into the cell by squeezing the bottle. The displaced argon escapes through the other tubulation. The cell is thus filled without compromising the purity of the gallium.

IV. THE THERMAL ENVIRONMENT

The crucible is designed for low heat transfer to the environment, so that the rate of melting is directly proportional to the flow of heat supplied from a unit surface to the gallium. The rate of advance of the liquid–solid interface can be controlled by varying the heat input, and is proportional to the temperature gradient of the solid.

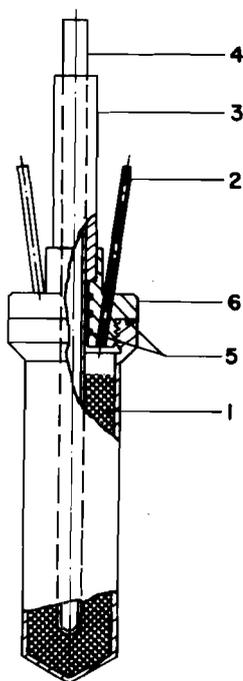


FIG. 1. Gallium crucible. (1) High-purity gallium, (2) evacuation tubes, (3) thermometer well protection tube, (4) thermometer well, (5) O-ring seals, (6) sealing head.

Toward this end, a heating jacket is provided, shown in Fig. 2. The heater winding of constantan wire is bifilar wound on the outer wall of the crucible. The resistance of the winding is 67.6 Ω . The input voltage is variable; with 5.8 V applied, the heat input is 497 mW. The latent heat of gallium is 79 342.6 J kg⁻¹, requiring 45 225.2 J to melt 570 g. The time required to melt the sample at this power level is about 25 h and the percent of metal melted is linear with time.

During heating, the space between the outside wall of the crucible and the inner wall of the jacket is evacuated to minimize heat transfer. For further control, the crucible in its jacket is immersed in a stirred 10-l water bath, whose temperature is controlled by a platinum resistance thermometer driving a thyristor controller. It is possible to adjust the bath within several tenths °C above or below the gallium melt temperature. Because this temperature is so close to ambient, control is facilitated by using a water cooling coil in the bath to establish a real differential against ambient. The standard resistor used as resistance reference for the measuring bridge is maintained in a thermostated oil bath. A schematic of the system, including means for evacuating and gas filling the crucible, is shown in Fig. 3.

V. MEASUREMENTS

Beginning in 1978, we have made a number of measurements on samples of gallium having purities of 99.9%, 99.999%, 99.9999%, and 99.999 99%. (Purity will be referred to in the following by the convention of the number of 9's; e.g., 5N = 99.999-wt.% pure.) Table I shows the analysis of the various gallium samples, furnished by SNP Ziar n. Hronom. All gallium was of Czechoslovak production.

In all cases measurements were made using Tinsley 25.5- Ω platinum resistance thermometers. Thermometer resistance was measured using a model 5650 Tinsley a-c bridge or an ASL model A7 automated a-c bridge. Measurements were made with thermometer currents of 1 and 2 mA, so that any thermometer self-heating could be eliminated by calculation to zero-power resistance. Alcohol was used in the well as a heat-transfer medium.

It is typical in fixed point thermometry to work with the metal freezing from the liquid–solid equilibrium. This is not

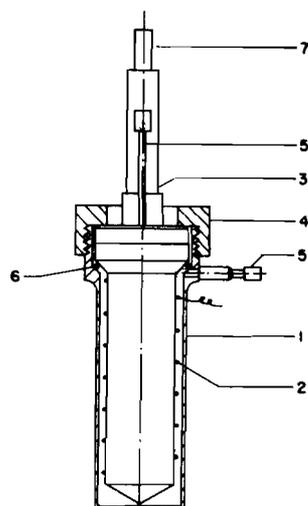


FIG. 2. Heating jacket with crucible installed. (1) Jacket, (2) heating winding, (3) gallium crucible, (4) sealing nit, (5) evacuation tubes, (6) O-ring seal, (7) thermometer well.

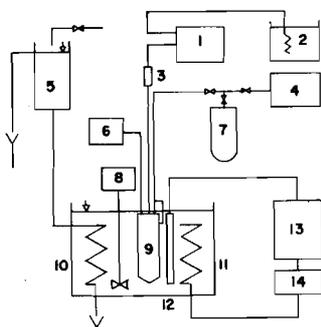


FIG. 3. Operating and measuring system. (1) ac bridge, (2) thermostated standard resistor, (3) platinum resistance thermometer, (4) vacuum pumps.

desirable with gallium, because the solidus expansion causes an increase in internal pressure and in turn causes one or more crystal phase changes, influencing the temperature by a few mK. Therefore, we used the phase equilibria moving toward the liquidus.

It is evident from phase diagrams that impurities affect the melting and triple point temperatures. The distribution of impurities within the frozen ingot is different for ingots which have been quickly or slowly frozen. When liquid metal is quickly frozen, impurities are distributed statistically and the sample is essentially homogeneous. When the liquid is slowly frozen, the systems with impurities, exothermal through solidification, are concentrated near the surface of the cooling bath. In general, the melting and triple points are depressed by increasing concentration of impurities, because of the higher fraction of systems which are endothermal through the phase change. The fraction of the sample containing the bulk of the impurities can be identified from the melting curve. Figure 4 shows the melting curves of 5N, 6N, and 7N gallium.

Impurities in gallium may form eutectic systems, alloys, or chemical compounds. These can produce exothermal or endothermal processes when gallium is melted, and the duration of the constant-temperature portion of the plateau is influenced. When systems which involve endothermal processes are melted, exothermal processes are still in

TABLE I. Analyses of gallium samples.

Sample purity (wt.%)	99.9 (3N)	99.999 (5N)	99.9999 (6N)	99.999 99 (7N)
Element found (ppm)				
Hg	188	0.54	0.05	...
Cu	2	0.36	0.03	...
Fe	3.3	1.18	0.10	0.10
Zn	13.9	0.86	0.05	...
Pb	1.3	0.18	0.05	...
Mg			0.02	0.006
Sn				...
Ca				0.010
Cr				...
Mn				...
Al				0.006
Si				0.007
S				0.005
K				0.010

* A blank space indicates no determination made. Ellipses indicate no trace found.

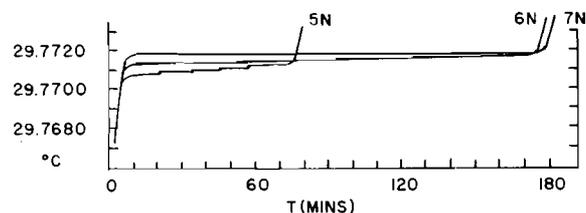


FIG. 4. Melting plateau characteristic of various-purity gallium.

the solid state, and the phase change is not influenced by these impurities. Our measurements show that, with 6N or purer gallium, we can reach the constant-temperature portion of the plateau when 40% of the sample is melted, and continue until 85% is melted. In Fig. 5, the solid line is the temperature seen by the thermometer in the crucible well. The dashed line represents the temperature of the melting front, registered by a small platinum thermometer mounted in the crucible outer wall. With less pure gallium than 6N the constant-temperature interval is shorter, and with 3N gallium there is no plateau. The influence of impurities in 7N gallium on the plateau is not detectable by contemporary instrumentation. We conclude that 7N gallium is adequate for the determination of the melting and triple point temperatures.

For measurements of the melting equilibrium temperature, the crucible containing slowly frozen 7N gallium, mounted in its jacket and immersed in the thermostated bath, is filled with argon at 1 standard atmosphere, and the metal temperature raised by the electrical heater at a rate of about 0.1 °C per minute. For measurements of the triple point temperature, the cell was evacuated below 1 Pa.

The actual effect of external pressure on the melt was found by measuring the temperature of the melt plateau at various pressures, and calculating to 1 Pa. It was found to be $-19.17 \text{ nK Pa}^{-1}$. This value agrees well with that calculated from the Clausius-Clapeyron equation:

$$\frac{dT}{dP} = \frac{(T)(\Delta V)}{l_{ga}} = -20.06 \text{ nK Pa}^{-1}, \quad (1)$$

where $T = 302.922 \text{ K}$, ΔV = the volume change of gallium at phase transition $= -5.2542 \times 10^{-6} \text{ per m}^3$, and $l_{ga} =$ the latent heat of gallium, $79 \text{ 342.6 J kg}^{-1}$.

The temperature of the melting or triple point equilibrium is the temperature of the physical conjunction between either the liquid and solid phases and the atmosphere, or the

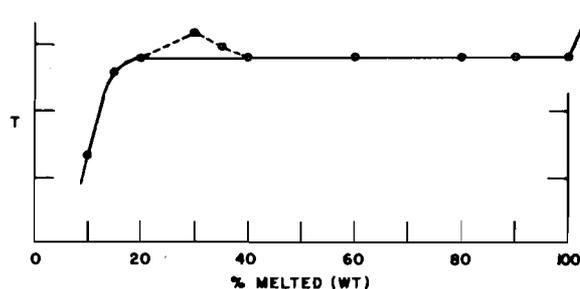


FIG. 5. Temperature vs % melted—6N or purer gallium.

TABLE II. Other determinations of the melting point of gallium.

Footnote	Institute	Year	Temp. (°C)	Uncertainty (°C)	Purity
^a	NBS	1976	29.772	0.002	7N
^b	ASMW	1976	29.769 7 ^c	0.000 6	5N5
^d	YSI	1977	29.770	0.002	6N
^e	NBS	1977	29.771 4	0.001 4	7N
^f	VNIIM	1982	29.770 4	0.000 4	5N5
^g	YSI	1983	29.771 51	0.000 12	6N +

^aD. Thornton and B. Mangum, Procès Verbaux du CCT, Doc. No. CCT/76-13, 1976.

^bS. Lau and P. Schlott, Procès Verbaux du CCT, Doc. No. CCT/76-23, 1976.

^cFreezing point.

^dH. E. Sostman, Rev. Sci. Instrum. **48**, 127 (1977).

^eD. Thornton, Clin. Chem. **23**, 719 (1977).

^fB. N. Oleinik, A. G. Ivanova, V. A. Zamkovets, and N. N. Ergardt, *Temperature, Its Measurement and Control in Science and Industry* (American Institute of Physics, New York, 1982), Vol. 5, pp. 317–320.

^gH. E. Sostman, Communication to the CCT, 1983. Results of measurements on 50 production gallium melting point cells.

liquid, solid, and vapor phases. This temperature is not measurable with real thermometers, which must be immersed below the surface and into the liquid and solid phases. At the thermometer location there is an equilibrium temperature given by the relationship

$$t_{68} = A + Bh, \quad (2)$$

where A is the measured temperature, B is the effect of the pressure of a 1-cm column of gallium = 1.165×10^{-5} K, and h is the height of the gallium column above the mean center of the thermometer element, in cm.

VI. RESULTS AND UNCERTAINTIES

Using the techniques described, the melting temperature of 7N gallium was determined to be 29.771 94 °C (302.921 94 K) and the triple point temperature,

TABLE III. Other determinations of the triple point of gallium.

Footnote	Institute	Year	Temp. (°C)	Uncertainty (°C)	Purity
^a	NBS	1982	29.773 98	0.000 16	6N +
^{b,c}	NPL	1982	29.773 70	0.000 55	6N
	INM	1982	29.773 65	0.000 6	6N
	IMGC	1982	29.773 84	0.000 55	7N

^aB. W. Mangum, *Temperature, its Measurement and Control in Science and Industry* (American Institute of Physics, New York, 1982), Vol. 5, pp. 299–309.

^bM. V. Chattle, R. L. Rusby, G. Bonnier, A. Moser, E. Renaot, P. Marcarino, G. Bongiovanni, and G. Frassinetti, *Temperature, its Measurement and Control in Science and Industry* (American Institute of Physics, New York, 1982), Vol. 5, pp. 311–6.

^cResults of each institute in a three-institute round robin. "Institute" refers to the source of the cells measured.

29.773 88 °C (302.923 88 K). It is our estimate that the differences between these determinations and the equivalent values for ideally pure gallium would be undetectable by contemporary instrumentation.

The thermometers used were calibrated against three fixed points of the IPTS, with the following probable limits of uncertainty: at the water triple point (0.01 °C), 0.2 mK; at the freezing point of tin, 1.0 mK; and at the freezing point of zinc, 2.0 mK. This provides an uncertainty at the gallium points not larger than 0.41 mK. The accuracy of the bridge is 1.10×10^{-7} . The reproducibility of measured values at a confidence level of 99% = 2σ , based on six samples and 40 measurements, provides an uncertainty not larger than 0.16 mK at the triple point and 0.03 mK at the melting point.

The values obtained agree well with other determinations of the melting and triple points. A summary of previous measurements is shown in Tables II and III.

¹H. Preston-Thomas, *Metrologia* **12**, 1 (1976).

²Procès Verbaux du CCT, 4^e Rapport du Groupe de Travail 2*, 1978.

ABOUT THE AUTHORS

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DAVID AYRES obtained his BSc (Honors) in Physics from the University of London in 1974, followed by research at Brighton Polytechnic in co-sputtered thin films of PTFE and carbon. He was employed by the Central Electricity Generating Board as First Engineer, and set up, there, a NAMAS Laboratory for temperature, pressure and voltage, using computer control techniques. In 1986 he joined Isotech as Deputy Head of Laboratory. His primary interest is in thermocouples, and he notes that he is partial to Type N.

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PHILIP METZ has recently joined Isotech to establish and manage its Western Hemisphere service organization, and it is expected that calibration services and selected manufacturing may be added in the future. Prior to this position, he was Chief Metrologist at YSI, and, until Henry Sostmann's retirement from YSI, his deputy in the YSI calibration station of the German Calibration Service (DKD). Phil comes to Isotech with a long background of experience in temperature measurement, calibration, the manufacture of SPRTs and precision thermistors, and was a chief member of the Staff during the years of development of the fundamental temperature calibration equipment now manufactured by Isotech.