

## FUNDAMENTALS OF THERMOMETRY PART VIII RADIATION THERMOMETRY AND CALIBRATION

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FIG. 1 - THE FIRST OPTICAL PYROMETER.  
(Please see Footnote 1)

The International Temperature Scale of 1990 (ITS-90) defines temperature in terms of constants of nature in which certain pure materials are in phase equilibrium (liquid-solid, liquid-vapor, solid-liquid-vapor), and specified interpolation instruments. The freezing points (or melting points of pure metals are examples of such phase equilibria, during which state changes and temperature remains invariant. Between 0.01°C (the triple point of water) and 961.78°C (the freezing point of silver) the specified interpolation device is a high-temperature platinum resistance thermometer (HTSPRT) calibrated at specified fixed points. Above 961.78°C the specified interpolation device is a radiation thermometer obeying Planck's Law, calibrated at one of the following freezing points: silver, gold or copper.

Planck's Law follows:

$$\begin{aligned} L_{\lambda}(T_{90}) / L_{\lambda}[T_{90}(X)] & \qquad \qquad \qquad \text{Eq. 1} \\ & = \exp [c_2 / \lambda T_{90}(X)]^{-1} / \exp [c_2 / \lambda T_{90}]^{-1} \end{aligned}$$

where  $L_\lambda(T_{90})$  and  $L_\lambda[T_{90}(X)]$  are the spectral concentrations of the radiance of a blackbody at wavelength  $\lambda$  in vacuum at  $T_{90}$  and  $T_{90}(X)$  respectively,  $c_2 = 0.014388$  m.K, and  $T_{90}(X)$  may be the freezing point of silver, gold or copper.

(Planck's Law derives from the earlier Wien Law for the distribution of energy in the emission spectrum of a blackbody. Planck's modification was simply to add the -1. However in attempting to explain why a much better agreement with experimental data was thus obtained, it was necessary for him to invent the quantum theory (that all electromagnetic waves can exist only as discrete packages or quanta) for which discovery he received the Nobel Prize in 1918).

In the design of the Scale, the silver point was chosen as the transition point between the HTSPRT and the radiation thermometer because  $961^\circ$  was the practical upper limit for the platinum thermometer, not because it was the lower limit for the radiation thermometer. Indeed, all surfaces at temperatures above zero absolute (0 K) emit and absorb some radiant energy. That this energy is not visible to us until high temperatures are reached is due to the limitation of the human eye to the electromagnetic spectrum between  $0.4 \mu\text{m}$  and  $0.7 \mu\text{m}$  (longer wavelengths go into the infra-red; shorter into the ultra-violet). As temperature increases, peak radiance shifts toward shorter wavelengths. At lower temperatures, the power emitted by the radiating blackbody is low; at room temperature is 470 W per square meter, at  $500^\circ\text{C}$  20 kW, at  $1000^\circ\text{C}$  150 kW, and at  $2500^\circ\text{C}$  3.4 MW (for reference, the full sun radiates 77 MW). Low power at low temperature limits the accuracy and repeatability of radiation thermometers; at high temperatures, other errors and uncertainties are important.

It is entirely realistic, and often useful, to employ radiation thermometers in the SPRT range as low as  $0^\circ\text{C}$ . An advantage, in some applications, is that radiation thermometers do not make physical contact with the device whose temperature is to be measured, and therefore do not perturb the measurement by adding heat to or subtracting heat from the source via a stem effect. Radiation methods are also useful where the object whose temperature is to be measured involves some hazard; for example, risk of explosion, nuclear reaction processes. Remote measurements are possible which could be made by no other means, for example, of the temperature of stars (although this latter requires highly specialized apparatus).

Cells for realizing the freezing points for the calibration of SPRTs between the triple point of water and the silver point are well understood, well characterized, and in common use. With appropriate equipment and techniques, the ITS-90 temperatures can be realized within fractions of a millikelvin ( $0.001^\circ\text{C}$ ) at the lower temperatures and several millikelvins at the higher temperatures.

Radiation thermometers, too, need to be calibrated, and the traditional deep-immersion cells for calibrating contact (immersion) thermometers can't be used. A radiation thermometer must be calibrated against a radiant source. It is worth noting that, unlike the contact thermometer where the thermometer (a coil of platinum wire, or a junction of dissimilar metals) is the sensor, the *sensor* in a radiation temperature measurement is the radiant source itself, where temperature is transduced into intensity of radiation. Indeed, radiation measurements are not analogs of thermodynamics but are thermodynamic directly.

Radiation thermometers are calibrated against radiating blackbodies. A blackbody is essentially a surface which emits and absorbs radiant energy identically. Obviously such a surface can be only approximated.

An ideal surface can be thought of as the interior of a hollow sphere, or another closed geometric shape, made of an opaque material. When the sphere is externally heated, radiant energy excited internally cannot escape. In this ideal condition it is impossible to observe the internal energy; such an ideal blackbody is an entirely closed universe, as alien and private as the interior of an unbroken egg. To make a useful approximation of a blackbody, a tiny aperture is cut, through which some optical instrument may observe the interior. If the design geometry is proper, the energy loss due to this compromise is *inconsiderable*; fractions of a percent.

All real sources reflect, transmit and absorb radiant energy. For an ideal source, the emissivity  $\varepsilon = 1$ . For a source which also transmits ( $\tau$ ) and absorbs ( $\alpha$ ) energy:

$$\varepsilon = 1 - \tau - \alpha \quad \text{Eq. 2}$$

The emissivity of real surfaces is usually far less than 1, and estimating the emissivity of the source is one of the real problems of radiation thermometry. Most present-day pyrometers have a dial or index which can be set to the estimated emissivity, to correct for its failure to be unity. The emissivity of some common surfaces is shown in the Table on the next page.

A blackbody is a primary calibration source if it surrounded by, so that its temperature is established by, an equilibrium constant-of-nature condition, such as the freezing equilibrium of a pure metal. A primary blackbody configuration [2] is shown in Fig. 2. The actual geometry can vary to the optimum for the specific temperature. Such sources were employed in establishing the equilibrium temperatures which are the basis of the high-temperature ITS-90, using as measuring instruments highly sophisticated pyrometers such as that of NPL, shown in Fig. 3.

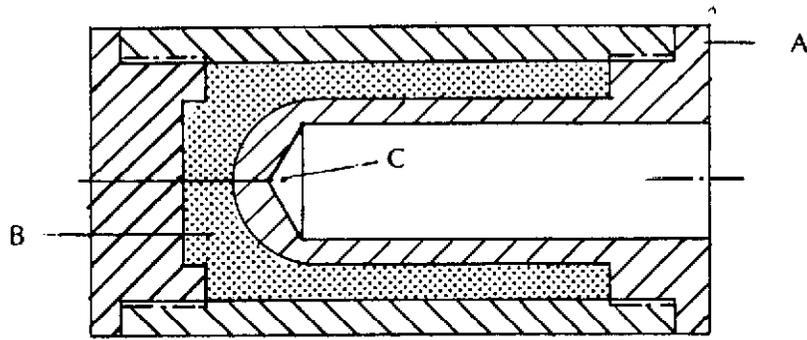


FIG. 2: A PRIMARY BLACKBODY CONFIGURATION: (A) high-density purified graphite, (B) pure metal (C) target upon which the pyrometer is sighted.

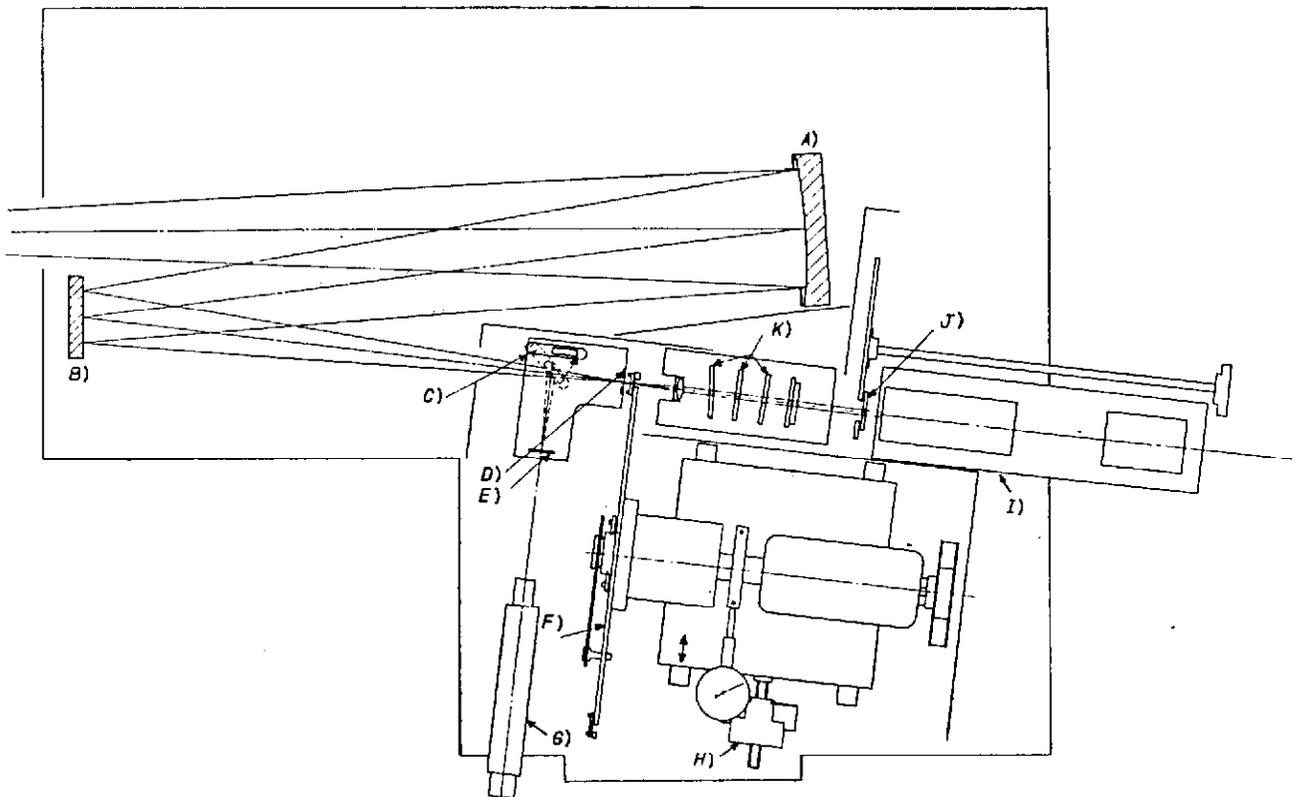


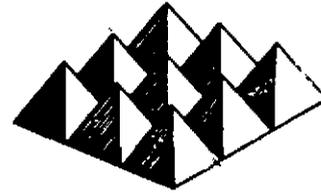
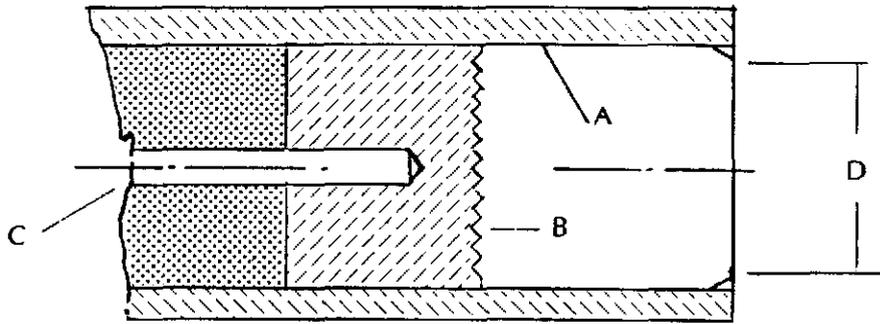
FIG. 3: THE NPL PHOTOELECTRIC PYROMETER: (A) off-axis ellipsoidal mirror, (B) plane mirror, (C) swinging mirror, (D) 0.75 mm diameter target aperture, (E) Sighting graticule, (F) sector disc, (G) Sighting telescope (H) Sector traverse mechanism (I) EMI Type 9558 photomultiplier and amplifier (J) Interference filter 662 nm, (K) Neutral density filters. (After Quinn, in Proceedings of the International School of Physics <Enrico Fermi>, Course LXVIII, *Metrology and Fundamental Constants*, North Holland Publ. Co. 1980).

MATERIAL	TEMPERATURE, K				
	300	500	1200	1300	1600
Stainless steel	.25	.55	.65		
Stainless steel polished	.16	.19			
Ordinary steel	.50				
Ordinary steel, oxidized	.80				
Rough cast iron	.90				
Liquid cast iron				.30	
Asbestos cement	.96				
Alumina, 10 $\mu\text{m}$ grain size			.30		.18
Alumina, 100 $\mu\text{m}$ grain size			.50		.40
Carborundum			.92		.82

Blackbody conditions can also be approximated by a plane surface, provided that the surface is properly configured [3]. For example, a flat graphite surface which is scored into numerous pyramidal indentations having the proper angles will approximate blackbody conditions because of the many surface-internal reflections which confine the radiant energy. Fig. 4 shows such a blackbody.

One novel (and patented) type of blackbody, which does not require either a sensor, [4] a controller or an equilibrium of a pure substance, is controlled by an integral gas-buffered heat pipe, which may be factory-set to a pressure to produce one specific temperature [Fig. 5].

Thermometers for reading temperatures as radiant energy are often instruments which make it possible to compare the brightness of the radiation source to some standard, the brightness of which may be continuously varied. The most common type of radiation thermometer contains an optical system which includes in the optical path a wire, ribbon or lamp filament heated by a variable current and superimposed upon the image of the radiating source. The radiating source is observed via the optical system while the lamp filament current, and hence its brightness, is varied until the brightnesses are equal, at which brightness the filament seems to disappear. The calibration of the instrument consists in establishing a relationship between temperature and the lamp excitation current. Such radiation instruments are said to be of the "extinction" type, because the appearance of the filament, as the brightnesses become equal, appears to be extinguished. Fig. 6 shows the arrangement of such an instrument, and Fig. 7 shows the commercial realization of it. Note, in particular, that a narrow band wave filter is located in the



DETAIL OF RADIATING SURFACE

FIG. 4: A RADIATING SOURCE WHOSE TEMPERATURE IS CONTROLLED BY A FURNACE TEMPERATURE CONTROLLER: (A) furnace wall, (B) radiating source, (C) control sensor pocket, (D) aperture

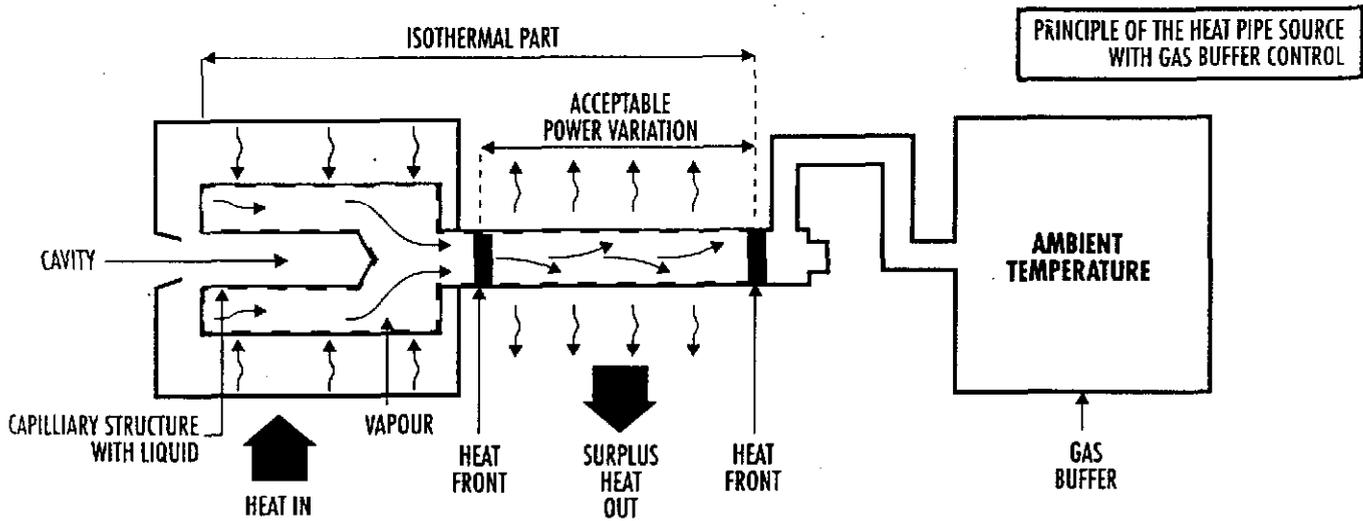


FIG. 5: A SINGLE-TEMPERATURE RADIATING SOURCE WHOSE TEMPERATURE IS CONTROLLED BY A GAS-BUFFERED HEAT PIPE

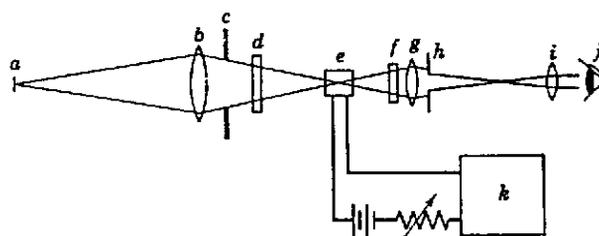


FIG. 6: OPTICAL PATH OF A COMMERCIAL PYROMETER. (A) source, (E) objective lens, (C) objective aperture, (D) absorption filter for temperatures above 1300°C, (E) pyrometer lamp, (F) red filter, (G) microscope objective lens, (H) microscope aperture stop, (I) microscope ocular, (J) eye, (K) instrument for measuring lamp current. (After NBS Monograph 41).

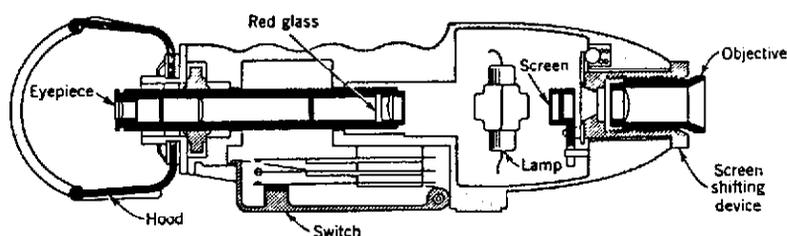


FIG 7: REALIZATION OF A COMMERCIAL PYROMETER: The arrangement of pyrometer elements by Leeds and Northrup. This pyrometer does not include emissivity compensation; most modern pyrometers do.

optical path. Simple radiation thermometers of this pattern, ("pyrometers"), may depend upon the observer to note the point at which the image of the reference filament brightness is equal to the brightness of the source, and disappears. Obviously, this won't work at lower temperatures where the radiance is at a wavelength too long for the eye to see. More sophisticated "automatic" pyrometers use a photodetector to detect the brightness null, or differential circuits involving splitters, or calibrated linear photodiodes.

How shall we calibrate, or assure the calibration of, our optical radiation thermometers? For those radiation sources that are not heated by a thermometric metal in liquid-solid equilibrium, but whose temperature is measured by an integral or inserted sensor, (a tiny thermocouple, or a very small industrial platinum resistance sensor), it is necessary simply to assure the calibration of the contact sensor. This can be done in conventional ways; by comparing the sensor, in an isothermal bath, to a superior thermometer, or by calibrating the sensor against a primary thermometric standard. The radiating source is then brought to a measured steady-state temperature, the pyrometer sighted upon it, and a measurement made. The process is repeated at several temperatures within the range of interest.

Blackbodies that are heated by a pure metal in phase equilibrium (a fixed-point blackbody) are used to calibrate radiation thermometers in a similar fashion. However it is sometimes demanded to check the validity of the fixed-point source itself.

"Calibration" of fixed point blackbodies is a misnomer, since the freezing temperature of a pure metal is fundamental, and is superior to any instrument which can be used to measure it. "Verification" of radiation cells could be made, at substantial expense, by direct comparison with cells maintained at a National Laboratory, such as NIST or NPL, using primary standard radiation thermometers, or transferred from tungsten strip lamps which themselves have received a primary calibration. (A tungsten strip lamp is a special lamp containing a strip of tungsten sealed into a vacuum or a gas, and a transparent window through which a pyrometer may see the strip. Primary radiation thermometry is used to establish the lamp current vs. temperature characteristic of the lamp strip, which then is a secondary calibration source. Such calibrators drift with time, and may require primary recalibration after 100 hours at temperature or so).

Isothermal Technology Ltd. has had many years of experience in the production of metal freeze point cells for the calibration of immersion thermometers, and has recently applied this experience to equivalent cells for calibrating radiation thermometers. These cells, intended for use in Isotech dedicated furnaces, were developed with the cooperation of England's National Physical Laboratory (NPL), which established the validity of their equilibria. Metal phase-equilibrium fixed point cells for use with radiation thermometers have a quite different geometry from cells intended for the immersion of contact thermometers, and so cannot be verified by SPRTs. However pure metals melt and freeze at temperatures which are physical constants and independent of the geometry of the containment. Exhaustive measurements with fine-wire thermocouples have found that the Isotech cells designed for radiation thermometry have virtually unmeasurable equilibrium temperature differences from cells intended for contact thermometers, and reproduce

the ITS-90 temperatures.

The equilibrium temperature of a fixed-point source depends crucially upon the purity of the metal. Isotech uses metals whose impurity fraction is less than 1 ppm. The containments for both immersion and radiation thermometer cells are dense and highly purified graphite. No other elements are introduced.

The most practical, appropriate and sufficient guarantee of the accuracy of the temperature produced at the freeze plateau is the purity of the metal in the cell. Isotech guarantees and certifies this purity on its certificates, and estimates that the temperature realized is within 0.1°C of the ITS-90 value. (In fact, this is the only certification made, and is the most reliable, since it does not depend upon a temperature measurement). Verification of a cell means, essentially, proving that its purity has not been impaired by contamination.

The technique for verifying the present purity of the metal in a blackbody cell is similar to that for an immersion thermometer in a cell intended for immersion instruments.

Assume, or establish by measurement, that the cell is at a temperature low enough so that the metal is completely in the solid phase. Set the furnace controller a few degrees (10°, perhaps; it is not critical) above the expected melt temperature [5]. Melt the metal completely. To assure that the metal is molten, monitor the emitted radiation with the pyrometer to observe (a) the temperature rise to the melt plateau; (b) the melt plateau; a period of time during which there is essentially no observable change in the indicated temperature (During this interval, the energy which the furnace is pumping into the cell is spent in changing the phase rather than raising the temperature; this is the *latent heat of fusion*). (c) the temperature rise above the melt plateau of the molten metal. Hold the cell at the molten temperature for some long period of time; ten to twelve hours is a good number. The purpose of this dwell is allow any contaminants or impurities to be well distributed through the melt. Then turn the furnace off, and freeze the sample as quickly as possible.

Set the furnace controller a few degrees above the anticipated melting temperature of the metal (as it was determined in the last paragraph), monitoring the temperature, as it rises, with the optical thermometer. At the beginning of the phase change - when the metal begins to melt - the melt arrest is observed. When the metal is completely molten, the temperature will resume its rise to the controller setpoint. The rise to the melt arrest, the melt arrest plateau and the rise from the plateau should all be recorded on a strip-chart recorder if the pyrometer has connections for one, or, failing that, by hand, and then conveyed to a graphical plot.

Freezing analysis can also provide useful information. To freeze the metal, first assure that it is completely molten, by ascertaining that the metal temperature has shown a rise above the melt plateau. Then reset the furnace controller to about  $1^{\circ}\text{C}$  below the melt plateau. Allow the furnace temperature to drift downward, monitoring it all the while, until the downward drift stops. Some metals exhibit supercool; that is, the metal remains liquid at a temperature below the temperature at which it would melt if it were in the solid state. Other metals exhibit very little supercool. If the metal does not supercool the temperature will stabilize on the freeze plateau; if it does supercool, the temperature will rise to the freeze plateau. Continue to record the temperature until the freeze plateau terminates and the temperature drops to the controller setting, and plot this temperature profile as well.

Figure 8(a) shows the characteristic graphs of the melting behavior of metals 6N+ (better than 99.9999%) pure, 5N+ (better than 99.999%) pure and 5N (99.999%) pure. If the metal is much less than 5N pure, there will be very little recognizable plateau. Fig. 8(b) shows typical freezing behavior for metals of several purities. Note in particular the slopes of the temperature drops after the freeze plateau has been finished.

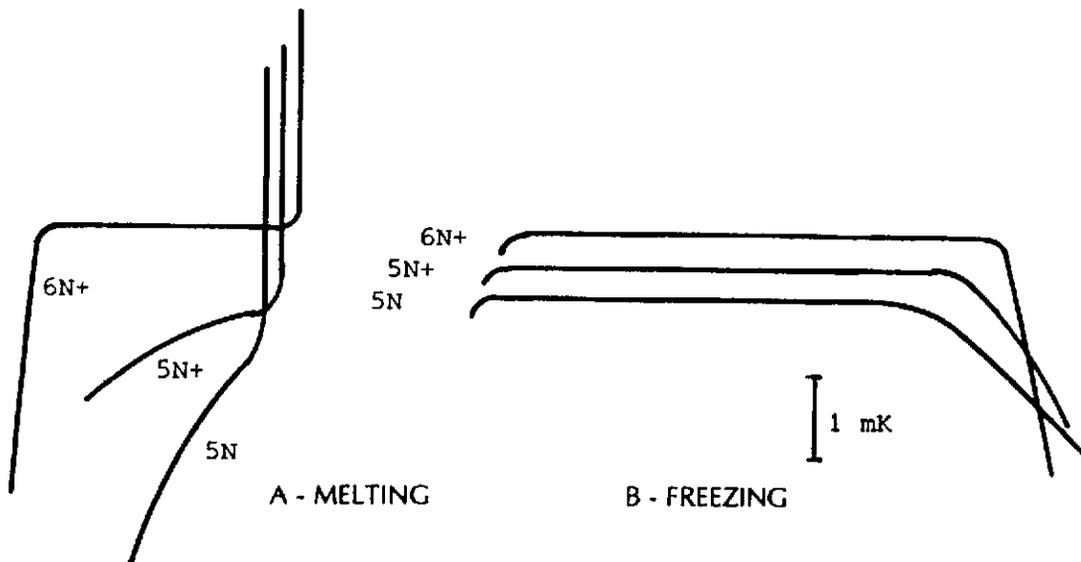


FIG 8: (A) typical melting curves of metals of three purities (B) typical freezing curves

## FOOTNOTES

[1] The first optical pyrometer. Objects which radiate within the visible portion of the spectrum glow whiter and brighter as temperature increases. Experienced workers in high temperature industries can often estimate temperature by eye to  $\pm 50^\circ$ . This cartoon is from *Heat and Temperature Measurement*, Robert L. Weber, © 1950, Prentice-Hall, Inc.

°C	COLOR
530	Dark blood red
570	Dark red
635	Dark cherry red
675	Medium cherry red
750	Cherry full red
850	Light, bright cherry red
900	Salmon, orange
950	Light salmon, orange
1000	Yellow
1180	Light yellow
1250	White

This Table supplied by The Vanadium Corporation.

[2] e.g., Isotech Model 970, 976, 982

[3] e.g., Isotech Model 976 with Isotech "Hockey Puck" cells containing either Gallium (29.76°C), Indium (156.60°C), Tin (231.93°C), Zinc (419.53°C), or Isotech Model 970 with Isotech Model GC cells containing either Indium, Tin, Zinc, Aluminum (662.32°C) or Silver (961.8°C)

[4] Isotech-Phillips Fixed Point Radiation Source, single temperature gas-buffered heat pipe; one factory-set temperature between 500° and 1000°C.

[5] Closer control can be obtained if the furnace controllers are occasionally calibrated. This can be done with little effort by using the metal fixed points themselves as reference.

(a) Set the furnace controller a few degrees below where your data indicates the melt temperature should lie.

(b) Allow the system to come to thermal equilibrium at that setting. Monitor the temperature to look for any thermal cycling. Note the controller setting and the mean indicated temperature.

(c) Reset the controller a few degrees higher than the expected melt temperature. Watch for the melt arrest. Observe the melt plateau temperature as indicated by the instrument.

(d) At the end of the melt arrest, allow the temperature to rise until it stabilizes. When it is in equilibrium, look for any thermal cycling. Note the controller setting and the mean indicated temperature.

(f) Using this notation:

$C_1, t_1$ , the controller setting and temperature indication below the melt plateau

$C_2, t_2$ , the controller setting and temperature indication above the melt plateau

$t_3$ , the temperature indicated by your instrument for the melt plateau.

The controller setting corresponding to the furnace temperature at the melt plateau is then calculated:

$$CS_{(mp)} = C_1 + [(C_2 - C_1) / (t_2 - t_1)] (t_3 - t_1) \quad \text{Eq.3}$$

For subsequent melts or freezes, set the controller slightly above  $CS_{(mp)}$ , (to melt) or below  $CS_{(mp)}$  (to freeze) or at a setting which assures that the peak furnace temperature when cycling is more than  $t_3$  (to melt) or less than  $t_3$  (to freeze).

## REFERENCES

(The following books in our library have been found particularly useful)

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*and for a view of the cutting edge and the development of ITS-90*

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