



National Institute of Standards & Technology

Certificate

Standard Reference Material 1968

Gallium

Melting-Point Standard

Cell No. _____

$29.7646 \pm 0.0007^{\circ}\text{C}$

International Temperature Scale of 1990 [1]

This Standard Reference Material (SRM) is intended to be used to calibrate thermometers near 30°C . SRM 1968 consists of approximately 25 grams of high-purity gallium in a specially designed, epoxy-sealed cell. The melting-point temperature of this cell, as given above, was verified using a set of three stable thermistors that were regularly recalibrated against a Standard Platinum Resistance Thermometer. The Standard Platinum Resistance Thermometer was used in the determination of the gallium triple-point temperature at NIST [2]. The stated uncertainty, 0.0007°C , represents one-half of the total range of the temperatures observed in 152 determinations of the melting points of this and similar cells.

SRM 1968 can be used to calibrate all thermometers that have temperature sensing elements smaller than 3.5 mm in diameter. With care, such thermometers may be calibrated with an accuracy of 0.0007°C . The liquid-solid equilibrium temperature of pure gallium at a pressure of one standard atmosphere (101.325 kPa) is a defining fixed point of the ITS-90 and is assigned the value 29.7646°C [1]. This equilibrium is best realized by slowly melting the solid.

The high-purity gallium metal used in SRM 1968 was obtained from Eagle-Picher Industries, Inc., Quapaw, Oklahoma.

The technical measurements at NIST (formerly NBS) leading to certification were performed by D.D. Thornton and B.W. Mangum of the Heat Division.

This certificate is a revision of the certificate dated June 1977. The changes consist primarily of the conversion of temperature on the IPTS-68 to those of the ITS-90 by B.W. Mangum of the Process Measurements Division.

The technical and support aspects involved in the revision, update and issuance of this Standard Reference Material were coordinated through the Standard Reference Materials Program by J.C. Colbert. The original coordination of certification efforts was performed by R.W. Seward.

Gaithersburg, MD 20899

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(Revision of certificate dated June 1977)

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(over)

SRM 1968 was designed by D.D. Thornton of the NBS Heat Division [3]. The cell components were fabricated by the NBS Instrument Shops Division. The cell consists of a Teflon body, a nylon well, and a nylon cap stem, as shown in figure 1. Filling, assembling, and sealing of the cells were performed in a dry argon atmosphere.

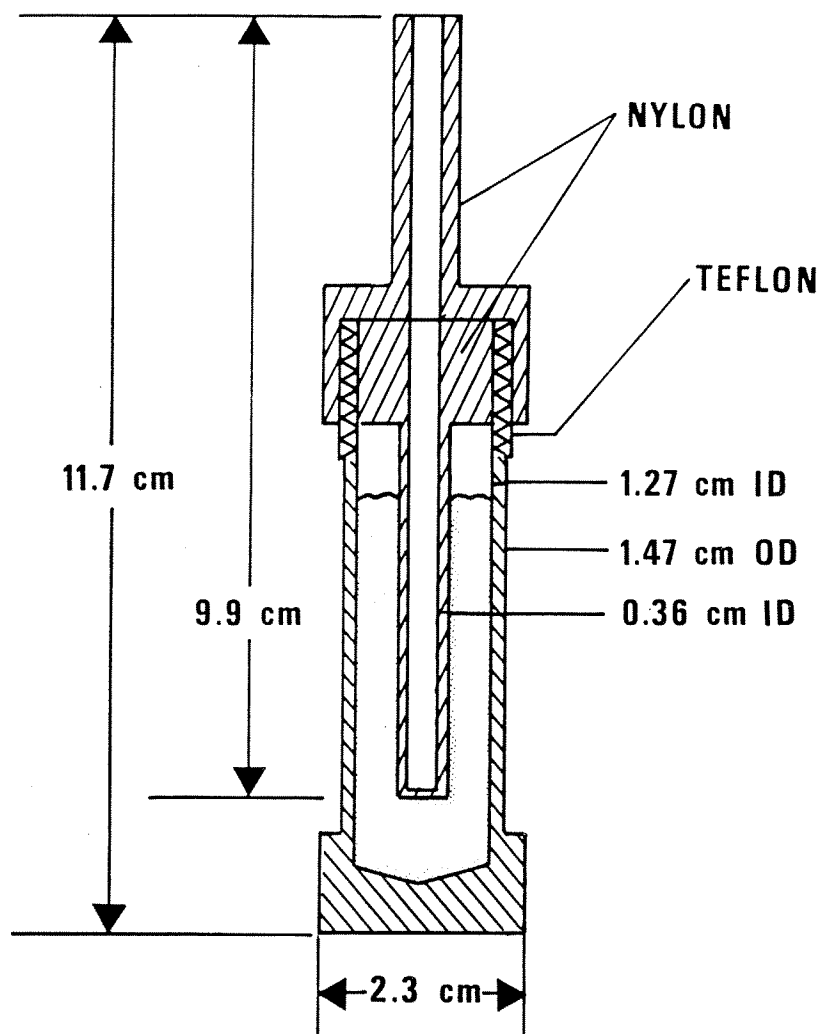


Figure 1. Cross-sectional drawing of SRM 1968, Gallium Melting-Point Standard

CAUTION: Because the nylon parts of the cell will absorb water, the cell should not be immersed in water for periods exceeding two days. Particular care should be exercised to prevent water entering the cap stem and well. When the cell is immersed in water, at least one centimeter of the cap stem should protrude from the surface.

NOTE: The gallium in SRM 1968 was frozen before leaving NIST. If the metal should melt completely, it should refreeze readily in an ambient of 20 °C. Should the metal supercool and remain liquid at room temperature, solidification can be induced by placing the cell in an ice bath or freezer for about two hours.

Instructions for Use of SRM 1968

SRM 1968 should be used as a calibrant in a temperature-regulated bath. To calibrate a thermometer with SRM 1968, the user should employ a light mineral oil for thermal contact in the well. Any water should be removed from the sensor before inserting it into the well. The temperature sensing element should be inserted into the well and the cell then inserted into the bath, which has been regulated to a temperature of 30.0 ± 0.1 °C. Within 30 to 60 minutes, the thermometer should indicate a steady reading, which corresponds to the gallium melting point (29.7646 °C). The duration of the melting plateau

depends on the bath temperature (figure 2). Thus, once melting has begun, the duration of the plateau can be prolonged by setting the bath to 29.80 ± 0.03 °C—just above the gallium melting point. The bath temperature can be checked by temporarily placing the thermometer sensor directly into the bath.

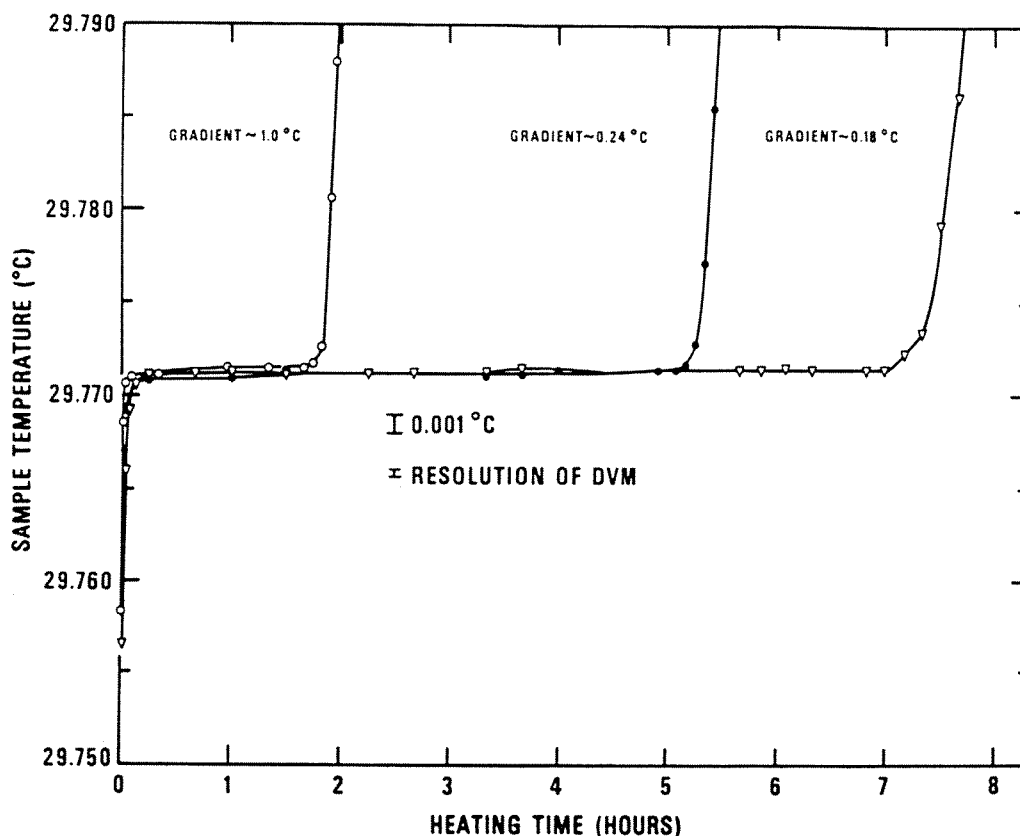


Figure 2. Melting curves of SRM 1968 (nominal 99.99999% pure gallium) for several different temperature gradients.

Accuracy of Calibration

The melting temperature of the gallium is very accurately known; however, several factors introduce uncertainty, and thus reduce the accuracy of the calibration. The most likely limiting factor in the accuracy of the calibration is the scale (or read-out) of the thermometer being calibrated. For example, a thermometer with a digital read-out to the nearest 0.01 °C will read all temperatures from 29.765 to 29.774 °C as 29.77 °C. For thermometers with resistive sensing elements (e.g., thermistors), another limiting factor is the self-heating caused by the current through the element. The size of the self-heating effect depends upon the local environment of the sensing element, and will introduce an uncertainty into the calibration. The instruction manual for the thermometer should be consulted, both in this regard and in reference to the resolution problem mentioned above.

The immersion effect is yet another potential source of calibration error. In the presence of a temperature difference (ΔT) between the bath and the sensor, heat will be conducted along the leads to or from the sensor and will produce an resulting immersion error (δT). When the thermometer is sufficiently immersed, most of the heat conducted along the leads is transferred through the insulation and the immersion error, ΔT , is insignificant. Because the error, δT , depends on ΔT , any significant immersion error can be minimized. The extent of such a problem can be determined when calibrating a thermometer with SRM 1968 in a bath at 30.0 °C. Set the bath temperature to 31.0 °C and observe the thermometer in the gallium cell for 15 minutes. When a new steady value is reached, the difference (δT) between this value and the value obtained with the bath at 30.0 °C is a measure of the immersion effect. For example, if the thermometer showed a change of 0.02 °C when the bath temperature was increased by 1 °C, then the immersion effect is 0.02 °C per degree Celsius. This example implies that the bath must be set no higher than about 29.9 °C if the immersion error is to be less than 0.003 °C.

REFERENCES

- [1] H. Preston-Thomas, "The International Temperature Scale of 1990 (ITS-90)," *Metrologia* **27**, 3-10 (1990).
- [2] Mangum, B.W., and Thornton, D.D., Determinations of the Triple-Point Temperature of Gallium, *Metrologia* **15**, 201-215 (1979).
- [3] Thornton, D.D., The Gallium Melting-Point Standards: A Determination of the Liquid-Solid Equilibrium Temperature of Pure Gallium on the International Practical Temperature Scale of 1968, *Clinical Chemistry* **23**, No. 4, 719-724 (April 1977).