NIST National Institute of Standards and Technology U.S. Department of Commerce

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Reference Material 8174
Gallium Melting-Point Reference (29.7646 °C)
(fixed point cell)

REFERENCE MATERIAL INFORMATION SHEET

Purpose: Reference Material (RM) 8174 is intended primarily for use as a reference point to enable laboratories to compare their present realization of a temperature scale at the gallium melting point with NIST's historical realizations of the International Practical Temperature Scale of 1968 (IPTS-68) [1] circa June 1977 and of the International Temperature Scale of 1990 (ITS-90) [2] circa March 11, 1991, where these dates correspond to the dates on the certificates initially provided with SRM 1968 and when updated for the transition from IPTS-68 to ITS-90.

Description: RM 8174 was previously offered as Standard Reference Material (SRM) 1968, with the initial certificate dated June 1977. The certificate was updated March 11, 1991 with the change from IPTS-68 to ITS-90 [3]. SRM 1968 no longer meets international quality standards (ISO 17034) for serving as a certified reference material [4,5]. The remaining inventory of SRM 1968 is thus being offered as RM 8174. A unit of RM 8174 consists of approximately 25 grams of high-purity gallium in a specially designed, epoxy-sealed cell.

Non-Certified Values: The melting-point temperature of this cell is 29.7646 °C relative to ITS-90 and was verified using a set of three stable thermistors that were regularly recalibrated against a Standard Platinum Resistance Thermometer (SPRT) used to determine the gallium triple-point temperature at NIST [6]. The stated uncertainty of 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. Note that 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 [2].

The melting-point temperature relative to the IPTS-68 is 29.7723 °C, with the same uncertainty of 0.0007 °C. The melting-point temperature of gallium was not assigned a fixed value in the establishment of IPTS-68. Instead, the RM IPTS-68 values are referenced to a melting-point value of 29.7723 °C ± 0.0004 °C determined relative to IPTS-68 [6,7].

The reported temperature values and their uncertainties are based on best practices at NIST when the certificate for SRM 1968 was initially created (June 1977) and later revised (March 11, 1991). The non-certified values of RM 8174 do not provide metrological traceability to the International System of Units (SI) or other higher-order reference system due to lack of backing by a standard quality system such as described in ISO 17034, *General requirements for the competence of reference material producers* [4].

Period of validity: The non-certified values are valid indefinitely within the measurement uncertainties specified, provided the RM is handled and stored in accordance with instructions given in this Reference Material Information Sheet (RMIS). This RMIS is nullified if the RM is stored or used improperly, damaged, contaminated, or otherwise modified.

Maintenance of Non-Certified Value: NIST will continue to monitor this material under the period of validity. NIST will notify the purchaser if substantive technical changes occur that affect the information in this information sheet. Registration (see attached sheet or register online) will facilitate notification.

Use: RM 8174 is intended for research use. The user of this material should be familiar with best practices in the operation and handling of metal freezing-point temperature standards and particularly gallium freezing point standards [8–10].

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Storage: The gallium cell should be carefully stored and handled, particularly given the age of the nylon components (well and stem cap). Gallium is a liquid at body temperature. Handling with gloves is thus recommended in case the cell leaks since gallium can be difficult to clean off skin and may damage jewelry.

Detailed Description of RM 8174: The cell consists of a Teflon body, a nylon well, and a nylon cap stem, as shown in Figure l. The initial filling, assembling, and sealing of the cells were performed in a dry argon atmosphere. The high-purity (nominal 99.99999 % pure) gallium metal used in this RM was obtained from Eagle-Picher Industries, Inc., Quapaw, Oklahoma sometime prior to 1977.

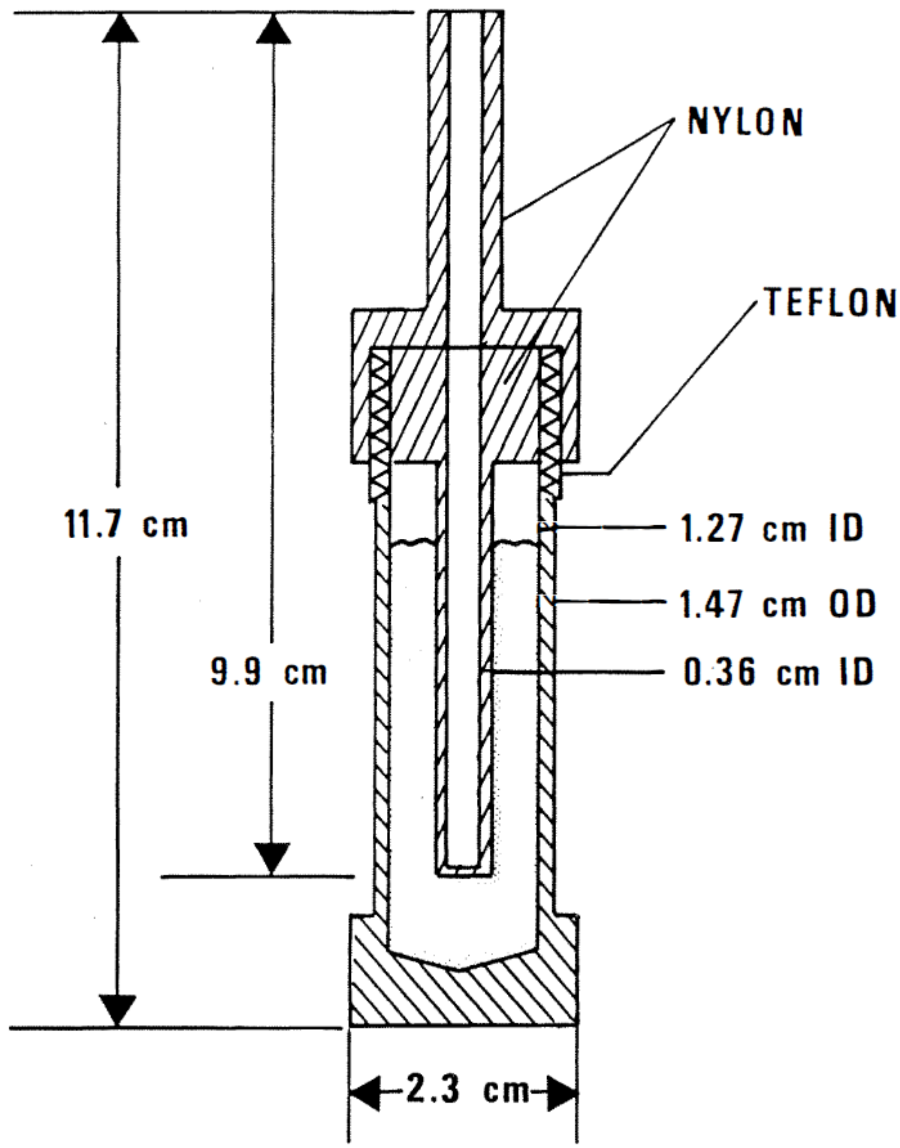

Figure 1. Cross-sectional drawing of RM 8174, Gallium Melting-Point Reference

CAUTION: Because the nylon parts of the cell will absorb water, the cell should not be immersed in water for periods exceeding two days. 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 RM 8174 was frozen before leaving NIST. If the metal should melt completely, it should refreeze readily in an ambient temperature 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 RM 8174: RM 8174 should be used as a reference in a temperature-regulated bath. To evaluate a thermometer with RM 8174, 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 °C ± 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 °C ± 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.

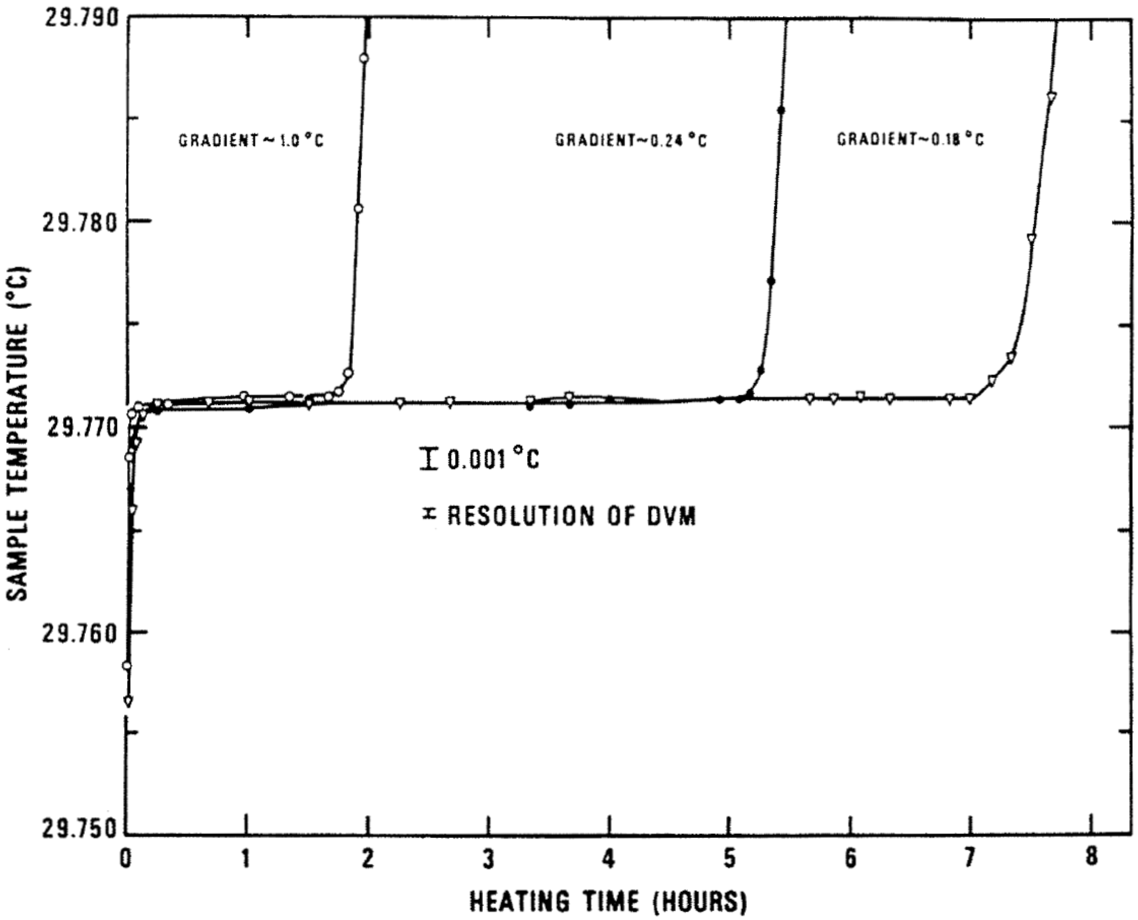

Figure 2. Melting curves of RM 8174 (nominal 99.99999 % pure gallium) for several different temperature gradients.

Discussion of the Temperature Uncertainty Relative to ITS-90: Under ITS-90, the melting temperature of the gallium is defined to be 29.7646 °C. Several factors introduce uncertainty in the use of RM 8174 as a temperature reference for ITS-90. The most likely limiting factor is the uncertainty in 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 29.755 to 29.764 °C as 29.76 °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, introducing an uncertainty into the measurement. 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 uncertainty. 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 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 by minimizing ΔT. The extent of such a problem can be determined when checking a thermometer with RM 8174 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

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Users of this RM should ensure that the Reference Material Information Sheet in their possession is current. This can be accomplished by contacting the Office of Reference Materials 100 Bureau Drive, Stop 2300, Gaithersburg, Maryland 20899-2300; telephone (301) 975-2200; e-mail srminfo@nist.gov; or via the Internet at https://www.nist.gov/srm.

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