National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1745

Indium Freezing-Point Standard

Certified Freezing-Point Temperature: (156.5985 ± 0.00034) °C

International Temperature Scale of 1990 (ITS-90)

This Standard Reference Material (SRM) is intended primarily for use as one of the defining fixed points of the International Temperature Scale of 1990 (ITS-90) [1]. The certified value of 156.5985 °C is the ITS-90 temperature assigned to the fixed point of pure indium. The fixed point is realized as the plateau temperature (or liquidus point) of the freezing curve of the slowly frozen high purity indium. SRM 1745 consists of 200 g of indium in the form of 10 g ingots with each ingot sealed in an argon atmosphere in a Mylar[1] bag.

Based on samples tested, the temperature range of melting of bulk material is not expected to exceed 0.0003 °C. Temperatures of freezing curve plateaus (see Figure 2) for samples of this material are expected to differ by not more than ± 0.0001 °C from each other and by not more than ± 0.00034 °C from the ITS-90 assigned temperature.

An expanded uncertainty (k = 2) of 0.34 m °C is assigned to the freezing point-temperature of SRM 1745. The Type A standard uncertainty component of 0.16 m °C is the standard deviation of W (t_{90}) values of repeated measurements of the laboratory-standard indium cell with a check Standard Platinum Resistance Thermometer (SPRT) [2]. The Type B standard uncertainty components are obtained from: the estimated uncertainty of 0.03 m °C in the freezing-point temperature of the laboratory standard calculated from the impurities listed in the metal assay; the estimated uncertainty of 0.03 m °C in the freezing-point temperature of the SRM metal calculated from the impurities listed in the metal assay; the temperature difference between the SRM cell and the laboratory-standard indium cell as determined from direct comparison measurements; and the uncertainty in those direct comparison measurements.

The indium for this SRM is of high purity, with the total of all elements that affect the freezing-point temperature being less than 0.1 mg/kg.

Expiration of Certification: The certification of this SRM is valid indefinitely within the measurement uncertainties specified, provided the SRM is used in accordance with the Notice and Warnings to Users section of this certificate.

Temperature measurements of the fixed-point cells were performed by G.F. Strouse of the NIST Process Measurements Division.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by J.C. Colbert.

Gaithersburg, MD 20899
Certificate Issue Date: 26 January 1998

Thomas E. Gills, Chief
Standard Reference Materials Program

Source of Material: The indium metal (Lot S2739) for this SRM was obtained from Arconium Specialty Alloys, Providence, RI 02909.

Notice and Warning to Users: Because any handling of high purity material is apt to introduce contamination, this SRM is provided in 10 g ingot form in order to minimize the need for handling during freezing-point cell construction. Nevertheless, every possible effort should be made to maintain the purity of this SRM through the use of polyethylene gloves while handling. Also, a clean laboratory environment is essential.

Instructions for Use: In assigning a temperature value to realizations of the indium freezing point for calibration purposes, corrections must be applied for the average depth of immersion (ℓ) of the thermometer sensing element below the surface of the metal ($dt/d\ell = 3.3 \times 10^{-3}$ °C/m). Also, if the pressure (p) over the cell during the measurements is not controlled at 1.01325×10^2 kPa (1 standard atmosphere), a correction, $dt/dp = 4.9 \times 10^{-8}$ °C/Pa, must be made for the difference in pressure.

Certification Testing: The thermal tests for the certification of this SRM were performed on fixed-point cells prepared in a manner similar to that described in reference [3]. Each cell contains approximately 1190 g of indium obtained from randomly selected 10 g ingots of indium.

The freezing points were prepared using the recommended "induced inner freeze" method. With the metal completely melted, the furnace was set at about 3 °C below the freezing point temperature. After supercooling and recalescence had been observed with a 25.5 Ω SPRT in the cell, the thermometer was removed and two fused-silica glass rods, each initially at room temperature, were inserted successively in the well for about 3 min each to induce freezing of a thin mantle of solid metal around the well. The thermometer was then reinserted into the cell and the recording of readings was begun. After equilibrium was established, the temperature of the plateau on the freezing curve was found to vary no more than ± 0.03 m °C during the first 50 % of the duration of the freeze. Three freezing curves obtained under such conditions are shown in Figure 1 (the region of supercooling and recalescence is not shown, as the curves begin after the reinsertion of the thermometer); a sample of the data is plotted at greater resolution in Figure 2.

After the metal was slowly and completely frozen in the above manner, the furnace was set at about 1 °C above the freezing-point temperature to slowly melt the metal over a time of approximately 10 h. Thermometer readings were recorded continuously until the melting was complete. Three melting curves obtained under such conditions are shown in Figure 3; some of the same data are plotted at greater resolution in Figure 4.

Following the freezing and melting curve measurements, the plateau temperature of a freezing curve of the test cell was compared directly with that of the standard indium freezing-point cell maintained by the NIST Platinum Resistance Thermometer Calibration Laboratory, using a 25.5 Ω SPRT. The method of direct comparison is described in detail in reference [5].

During the freezing and melting curve measurements, an inert environment of argon gas at 1.01325×10^2 kPa (1 standard atmosphere) was maintained in the cells.

The electronic measurement equipment included an ASL F18[1] resistance ratio bridge, operating at a frequency of 30 Hz, and temperature controlled Tinsley® 5685A 100 Ω reference resistor. This reference resistor was maintained at a temperature of (25.000 ± 0.010) °C. Freezing curve and melting curve measurements were made with an excitation current of 1 mA. Direct comparison measurements of the thermometer resistance were conducted at two excitation currents, 1 mA and $\sqrt{2}$ mA with a 25.5 Ω SPRT, to allow analysis of the results at zero power dissipation. A computer controlled data acquisition system was used to acquire the ASL F18 bridge readings through the use of an IEEE-488 bus.

---

[1] Certain commercial materials and equipment are identified in order to adequately specify the experimental procedure. Such identification does not imply a recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment are necessarily the best available for this purpose.

REFERENCES

[1] Preston-Thomas, H., "The International Temperature Scale of 1990 (ITS-90)," Metrologia **27**, pp. 3-10 (1990), Metrologia **27**, p. 107, (1990).

[2] Strouse, G.F. and Tew, W.L., "Assessment of Uncertainties of Calibration of Resistance Thermometers at the National Institute of Standards and Technology," NISTIR 5319, 16 pages, (1994).

[3] Furukawa, G.T., Riddle, J.L., Bigge, W., and Pfeiffer, E.R., "Standard Reference Materials: Application of Some Metal SRMs as Thermometric Fixed Points," Nat. Bur. Stand. (U.S.), Spec. Publ. 260-77, 140 pages, (1982).

[4] Mangum, B.W. and Furukawa, G.T., "Guidelines for Realizing the International Temperature of 1990 (ITS-90)," NIST Tech. Note 1265, 190 pages, (1990).

[5] Mangum, B.W., Pfeiffer, E.R., and Strouse, G.F. (NIST); Valencia-Rodriguez, J. (CENAM); Lin, J.H.and Yeh, T.I. (CMS/ITRI); Marcarino, P. and Dematteis, R. (IMGC); Liu, Y. and Zhao, Q. (NIM); Ince, A.T. and Cakiroglu, F. (UME); Nubbemeyer, H.G. and Jung, H.J. (PTB), "Intercomparisons of Some NIST Fixed-Point Cells with Similar Cells of Some Other Standards Laboratories," to *Metrologia* 33, (1996).

It is the responsibility of users of this SRM to assure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: Phone (301) 975-6776 (select "Certificates"), Fax (301) 926-4751, e-mail srminfo@nist.gov, or via the Internet http://ts.nist.gov/srm.

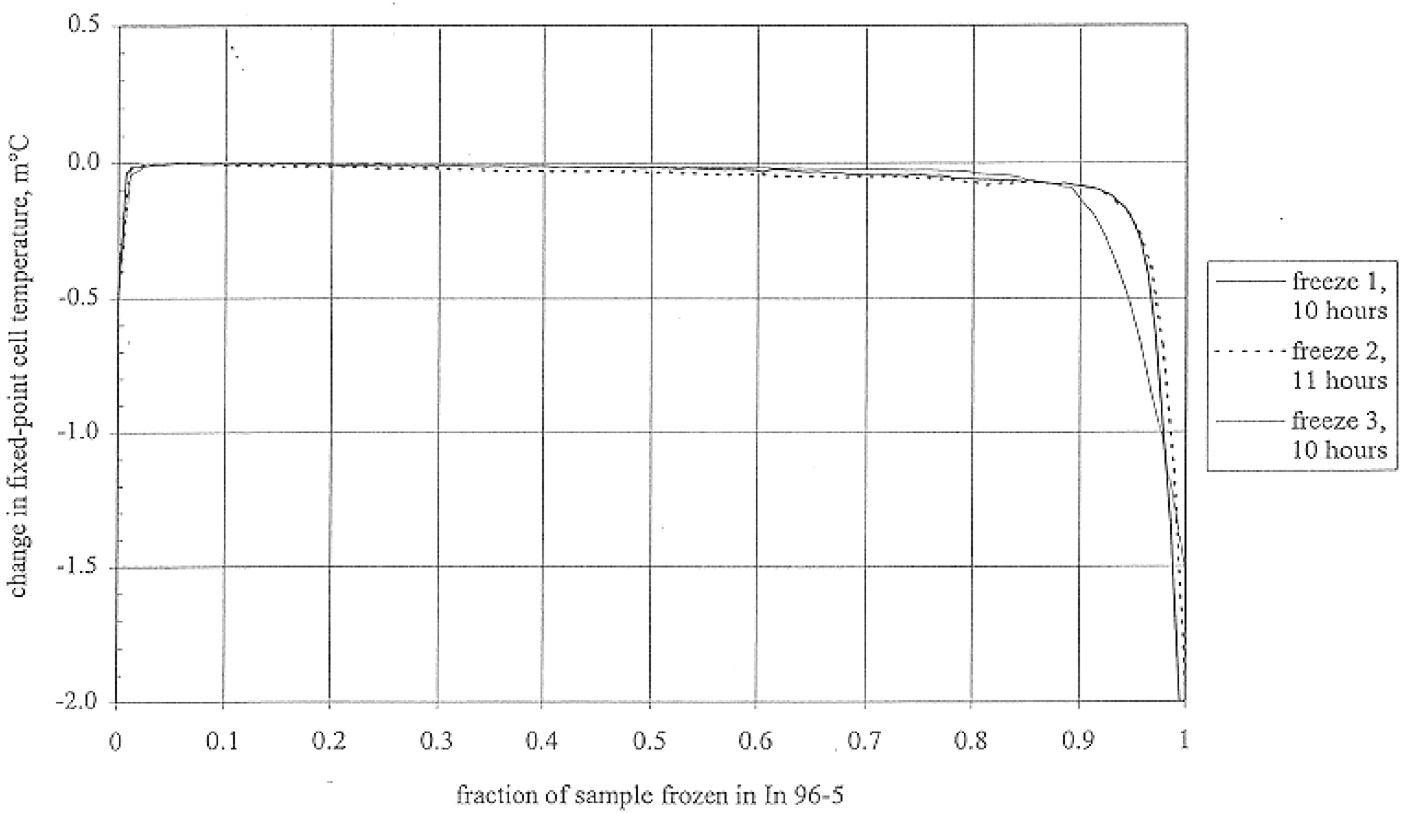

Figure 1. Three freezing curves of SRM 1745 indium using the "induced inner freeze" preparation technique.

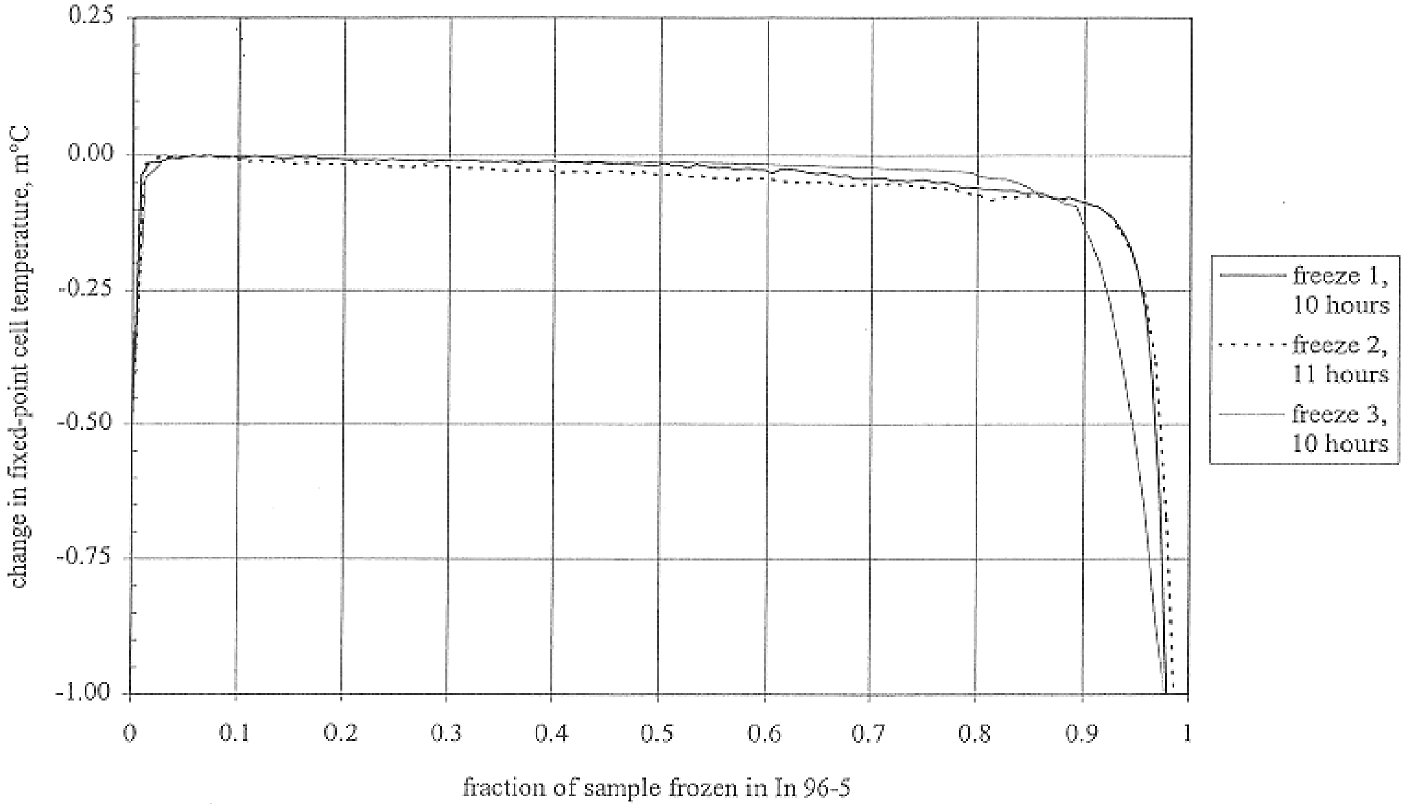

Figure 2. The freezing plateau regions of Figure 1 at greater resolution.

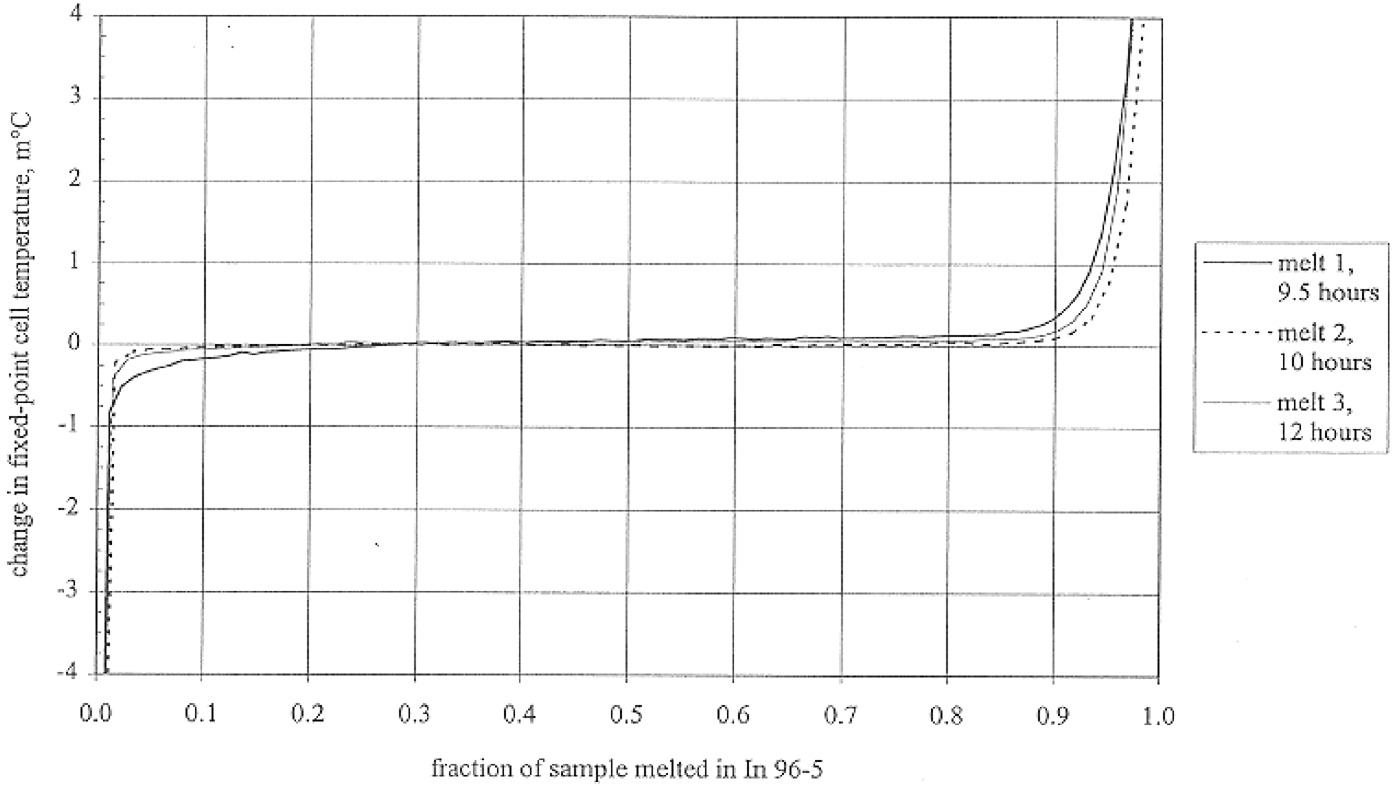

Figure 3. Three melting curves of SRM 1745 indium following a slow freeze. Each melt followed the respective slow freeze of Figure 1.

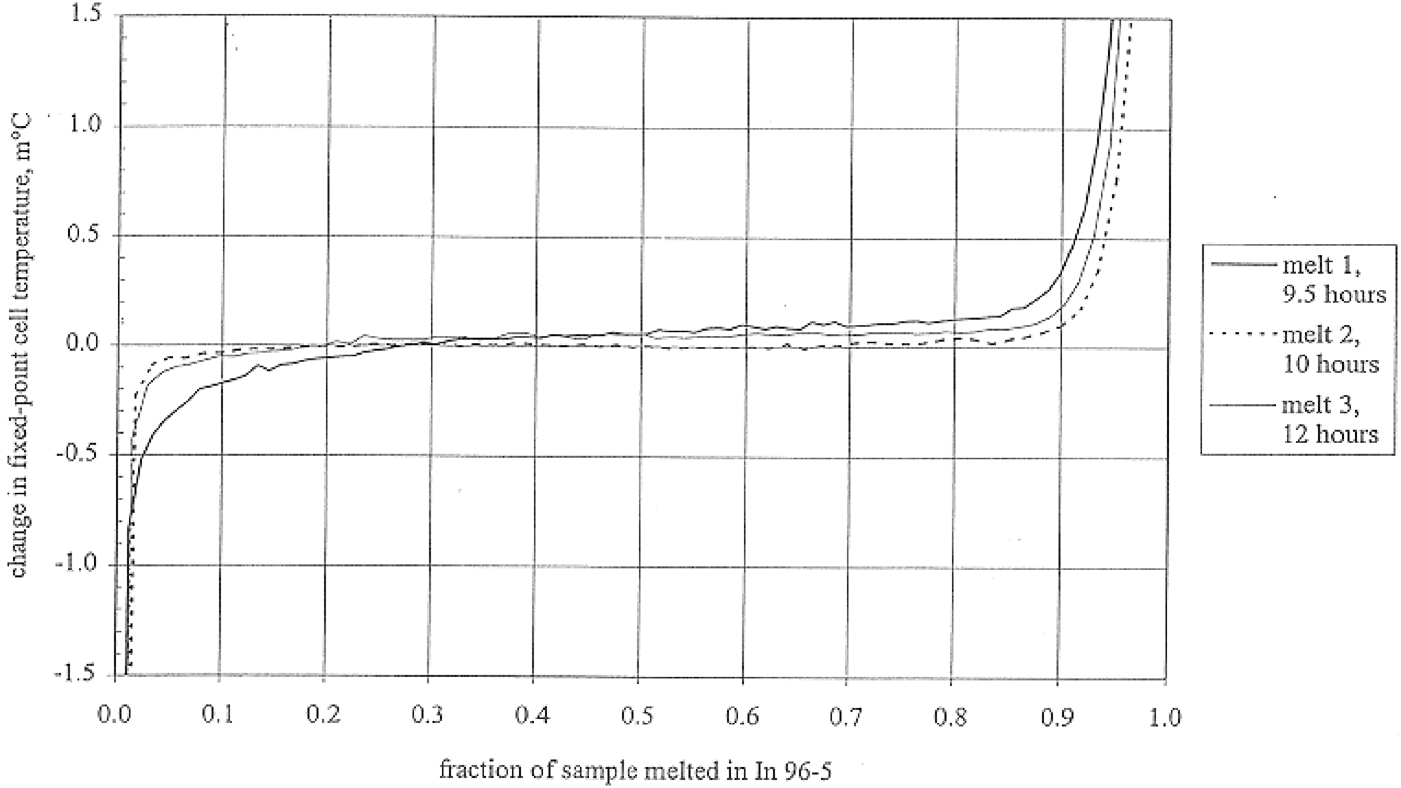

Figure 4. The melting plateau regions of Figure 3 at greater resolution.