National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2232

Indium DSC Calibration Standard

Temperature and Enthalpy of Fusion

This Standard Reference Material (SRM) is intended for use in the temperature and enthalpy of fusion calibrations of differential thermal analyzers (DTA), differential scanning calorimeters (DSC), and similar instruments. This SRM consists of a 1 g piece of indium metal sealed in an argon atmosphere in a mylar bag.

Certified Temperature of Fusion: The certified value of 156.598 5 ºC is the temperature assigned to the melting point of pure indium [1]. The melting point is realized as the plateau temperature of the melting curve of the slowly melted, high purity indium.

Temperature of Fusion: 156.598 5 ºC ± 0.000 34 ºC

Based on samples tested, the temperature range of melting of bulk material is not expected to exceed 0.000 3 ºC. Temperatures of melting curve plateaus for samples of this material are expected to differ by not more than 0.000 1 ºC from each other and by not more than 0.000 34 ºC from the assigned temperature. The basis for assigning an expanded uncertainty ($k = 2$) of 0.000 34 ºC to the temperature of fusion of SRM 2232 is described in reference 2.

Certified Enthalpy of Fusion: The certified enthalpy of fusion value at the DSC onset temperature was determined by performing duplicate measurements on seven SRM 2232 DSC samples. The DSC onset temperature has an expanded uncertainty ($k = 2$) of 0.046 ºC.

Enthalpy of Fusion: $28.51\ \mathrm{J{\cdot}g^{-1}} \pm 0.19\ \mathrm{J{\cdot}g^{-1}}$

The uncertainty was calculated as two times the square root of the sum of three components of variance. Component one was the standard deviation of the mean from 14 measurements of the enthalpy of fusion of indium. Component two was the standard deviation of the mean of 14 measurements of the enthalpy of fusion of SRM 2220 Tin that were used to calibrate the energy scale. Component three was the variance of the enthalpy certification measurements for SRM 2220 *Temperature and Enthalpy of Fusion – Tin* the calibration standard. The variance for SRM 2220 was calculated as the square of one-half the stated uncertainty on the certificate. The basis for assigning an expanded uncertainty ($k = 2$) of $0.19\ \mathrm{J{\cdot}g^{-1}}$ to the enthalpy of fusion of SRM 2232 is described in reference 3.

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. However, the certification is nullified if the SRM is damaged or contaminated.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

Temperature studies on fixed-point cells were performed by G.F. Strouse of the NIST Process Measurements Division. DSC studies were performed by D.R. Kirklin of the NIST Physical and Chemical Properties Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald of the NIST Measurement Services Division.

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See Certificate Revision History on Last Page

NOTICE AND WARNING TO USERS

Handling of SRM: Any handling procedures of this high purity material are subject to introduce contamination. Every possible effort should be made to maintain the purity of this SRM.

SOURCE AND ANALYSIS[1]

Source of Material: The indium metal (Lot S2739) for this SRM was obtained from Arconium (Providence, RI). The indium is of high purity, with the total of all elements that affect the melting point temperature being less than 0.1 mg/kg.

Temperature of Fusion Measurements: The thermal tests for the certification of this SRM were performed on a fixed-point cell prepared in a manner similar to that described in reference 4. The cell contains approximately 1190 g of indium obtained from the randomly-selected 1 g pieces of indium of lot S2739.

The freezing points were prepared using the recommended "induced inner freeze" method. With the metal completely melted, the furnace was set at approximately 3 ºC below the freezing point temperature. After supercooling and recalescence had been observed with a 25.5 Ω standard platinum resistance thermometer (SPRT) in the cell, the thermometer was removed and two fused silica glass rods, each initially at room temperature, were inserted successively into the well for about three minutes 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 0.000 03 ºC during the first 50 % of the duration of the freeze. A typical freezing curve obtained under such conditions is shown in Figure 1 (the region of supercooling and recalescence is not shown, as the curve begins 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 approximately 1 ºC above the freezing point temperature to slowly melt over a time period of approximately 10 hours. Thermometer readings were recorded continuously until the melting was complete. A typical melting curve obtained under such conditions is shown in Figure 3.

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 of the 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 environment of inert argon gas at 101 325 Pa was maintained in the cells.

The electronic measurement equipment included an ASL F18 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 25.00 ºC ± 0.010 ºC. Freezing curve and melting curve measurements were made with an excitation current of 1 mA. Direct comparison measurements were made using a 25.5 Ω SPRT at low exitation currents of 1 mA and $\sqrt{2}$ mA to allow for the 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.

Enthalpy of Fusion Measurements: Seven indium samples, 5 mg to 15 mg each, cut from a 1 g ingot of SRM 2232 and seven tin samples, 9 mg to 14 mg each, cut from a 6.25 cm^2 sheet of SRM 2220 were used in the DSC measurements, which were performed on a Perkin Elmer DSC 7 Differential Scanning Calorimeter. The temperatures of fusion for tin and indium were used to calibrate the temperature axis. The enthalpy of fusion of tin was used to calibrate the measured energy (measured area). After linearization of the temperature axis, the temperatures and enthalpies of fusion were measured for the set of seven SRM 2220 tin samples used in the calibration of the instrument. Duplicate measurements were made on seven SRM 2232 indium samples to determine the certified values and the observed variability due to the instrument and sample configuration. A typical DSC melting curve of the indium is shown in Figure 4.

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

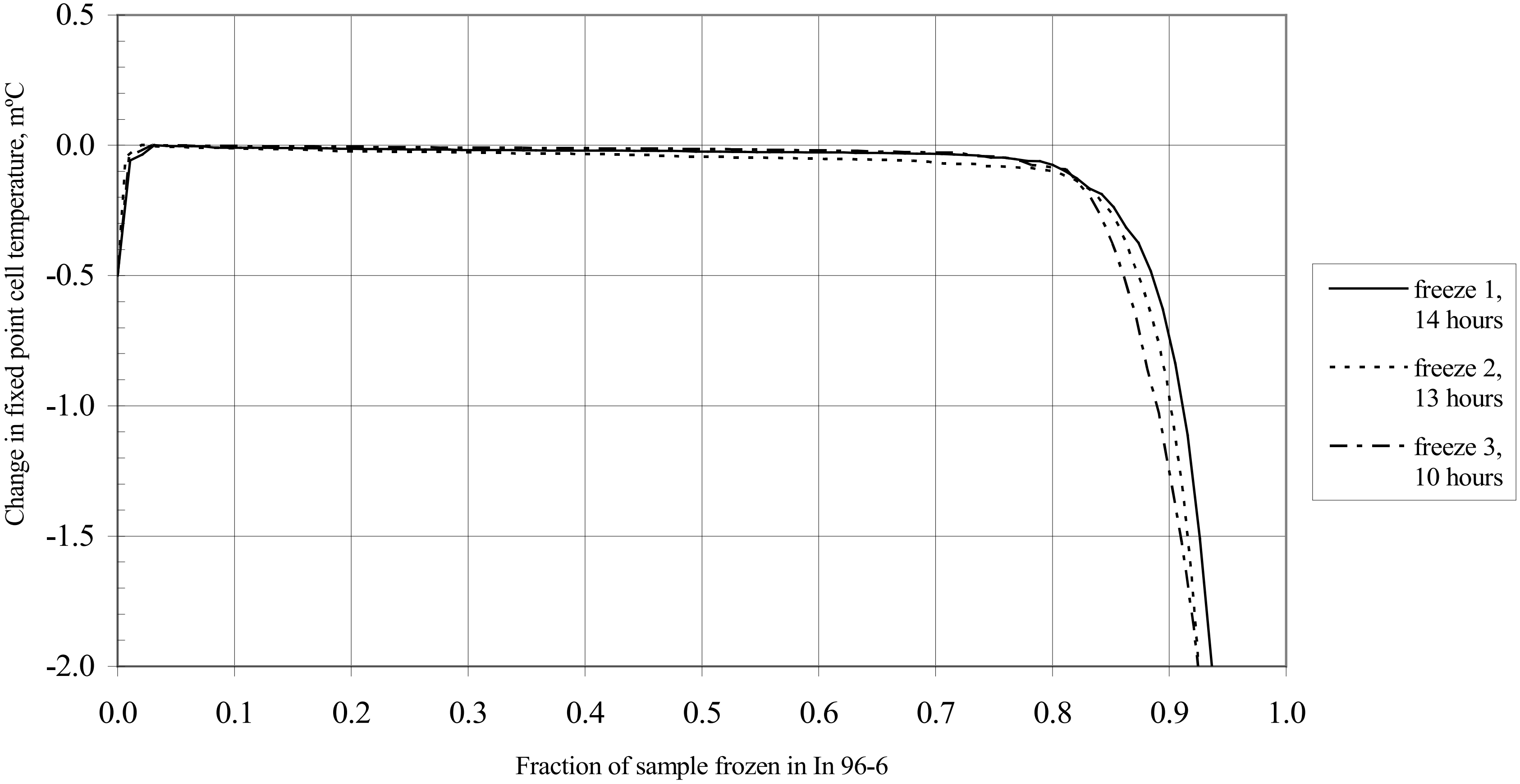

Figure 1. The freezing curves of SRM 2232 Indium DSC Calibration Standard using the "induced inner freeze" preparation technique.

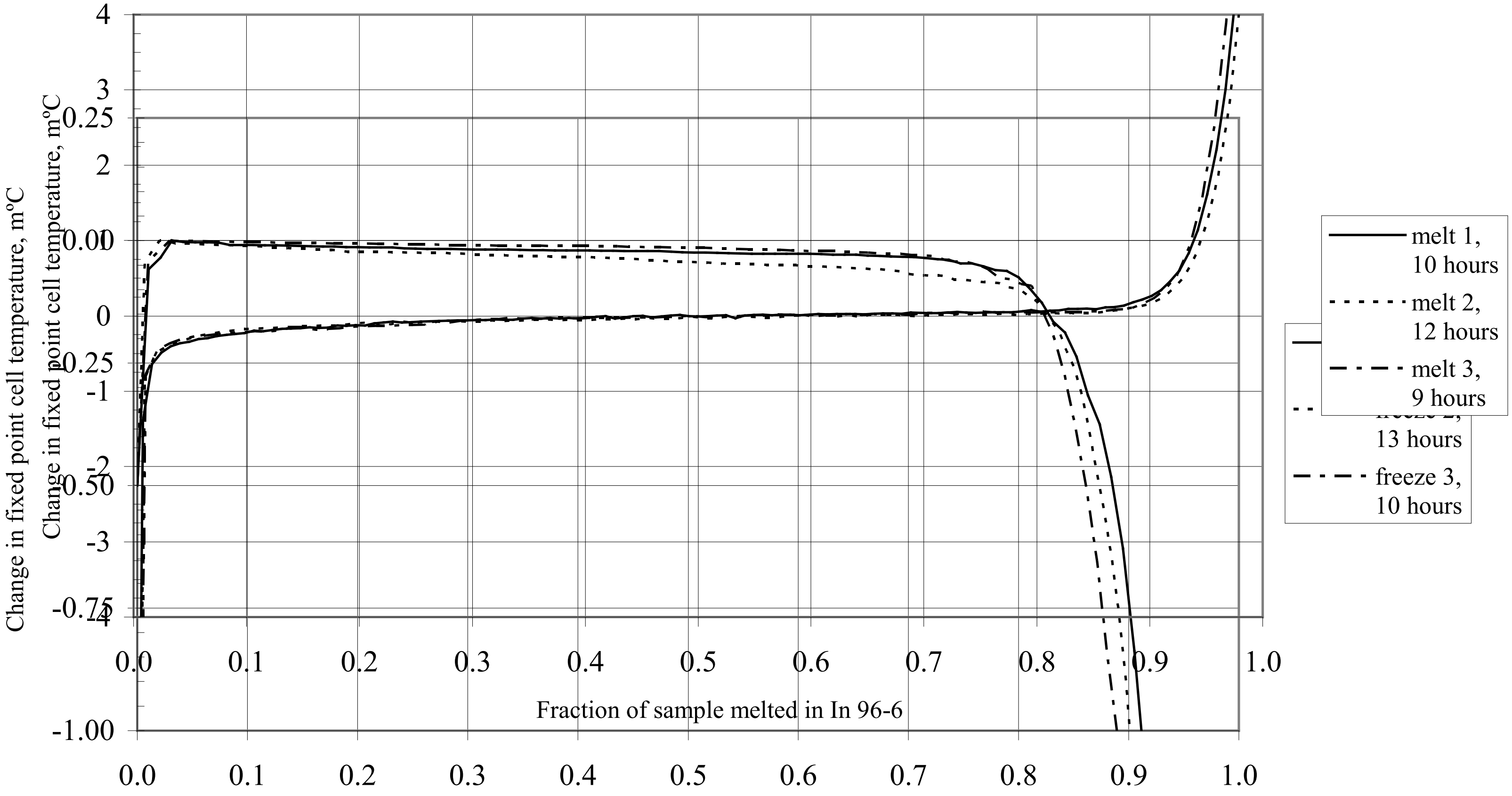

Figure 3. Three melting curves of SRM 2232 Indium DSC Calibration Standard In 96-6 following a slow freeze. Each melt followed the respective slow freeze of Figure 1.

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

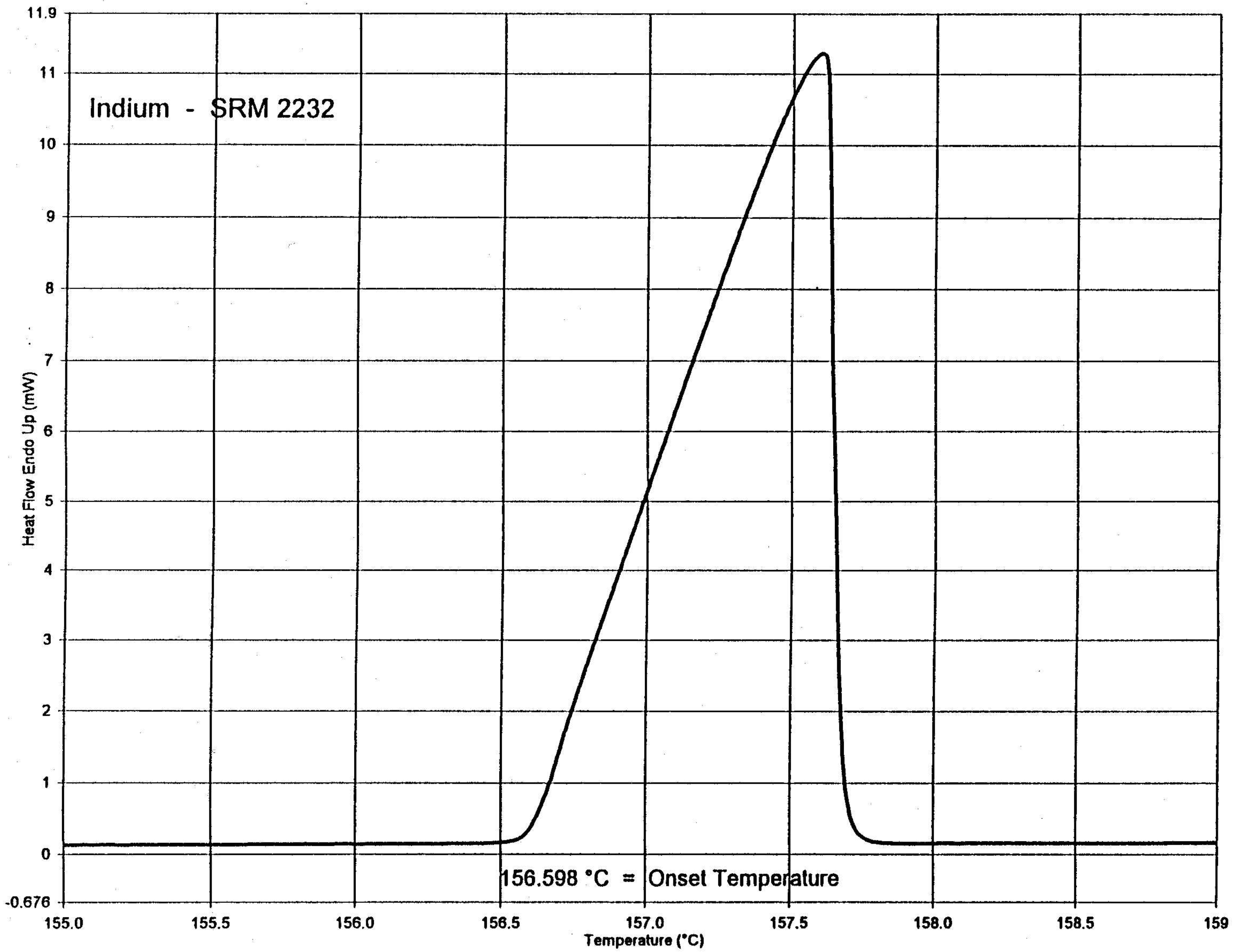

Figure 4. Typical DSC melting curve for SRM 2232 *Indium DSC Calibration Standard*.

REFERENCES

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[2] Strouse, G.F.; Tew, W.L.; *Assessment of Uncertainties of Calibration of Resistance Thermometers at the National Institute of Standards and Technology*; NISTIR 5319; National Institute of Standards and Technology, U.S. Department of Commerce: Gaithersburg, MD, p. 16 (1994).

[3] ISO; G*uide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed.; International Organization for Standardization: Geneva, Switzerland (1993); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297; U.S. Government Printing Office: Washington, DC (1994); available at http://physics.nist.gov/Pubs/.

[4] Furukawa, G.T; Riddle, J.L; Bigge, W.R.; Pfeiffer, E.R.; *Standard Reference Materials: Application of Some Metal SRMs as Thermometric Fixed Points*; NBS Special Publication 260–77, U.S. Government Printing Office: Washington, DC, p. 140 (1982).

[5] Mangum, B.W.; Pfeiffer, E.R.; Strouse, G.F.; (NIST); Valencia-Rodriguez, J.; (CENAM); Lin, J.H.; Yeh, T.I.; (CMS/ITRI); Marcarino, P.; Dematteis, R.; (IMGC); Liu, Y.; Zhao, Q.; (NIM); Ince, A.T.; Gakirolu, F.; (UME); Nubbemeyer, H.G.; Jung, H.J.; (PTB); *Intercomparisons of Some NIST Fixed-Point Cells with Similar Cells of Some Other Standards Laboratories*; Metrologia, Vol. 33, pp. 215–225 (1996).

Certificate Revision History: 21 July 2005 (This revision reflects editorial changes); 01 March 1999 (Original certificate date).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.