National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2232

Indium DSC Calibration Standard
Temperature and Enthalpy of Fusion

Standard Reference Material (SRM) 2232 is primarily intended for use in the temperature and enthalpy of fusion calibrations of differential thermal analyzers (DTA), differential scanning calorimeters (DSC), and similar instruments. A unit of 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 156.5985 EC 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.

Certified Temperature of Fusion (156.5985 " 0.000 34) EC

Based on samples tested, the temperature range of melting of bulk material is not expected to exceed 0.0003 EC. Temperatures of melting curve plateaus for samples of this material are expected to differ by not more than 0.0001 EC from each other and by not more than 0.000 34 EC from the assigned temperature. The basis for assigning an expanded uncertainty (k = 2) of 0.000 34 EC to the temperature of fusion of SRM 2232 is described in [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) 0.046 EC.

Certified Enthalpy of Fusion (28.51 " 0.19) J·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 fourteen measurements of the enthalpy of fusion of indium. Component two was the standard deviation of the mean of fourteen 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, 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 J·g^{-1} to the enthalpy of fusion of SRM 2232 is described in [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.

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

Richard F. Kayser, Chief
Physical and Chemical Properties Division

Gregory J. Rosasco, Chief
Process Measurements Division

Gaithersburg, MD 20899
Certificate Issue Date: 1 March 1999

Thomas E. Gills, Chief
Standard Reference Materials Program

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.

NOTICE AND WARNINGS TO USERS

Source of Material: The indium metal1[1] (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.

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

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 [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 about 3 °C 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 03EC 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 about 1EC above the freezing point temperature to slowly melt the metal over a time 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 [5].

During the freezing and melting curve measurements, an environment of inert argon gas at 101 325 Pa pressure 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 ± 0.010) EC. 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 l 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.

[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.

Enthalpy of Fusion Measurements: Seven indium samples, 5 mg to 15 mg each, cut from a l 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.

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] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed. ISO, Geneva, Switzerland, (1993): see also Taylor, B.N. and Kuyatt, C.E., "Guidelines for Evaluating and Expressing Uncertainty of NIST Measurement Results," NIST Technical Note 1297, U.S. Government Printing Office, Washington, DC, (1994).

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

[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 Gakirolu, 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," Metrologia **33**, pp. 215-225, (1996).

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 (select "Certificates"), Fax: (301) 926-4751, e-mail srminfo@nist.gov, or via the Internet at [http://ts.nist.gov/srm.](http://ts.nist.gov/srm)

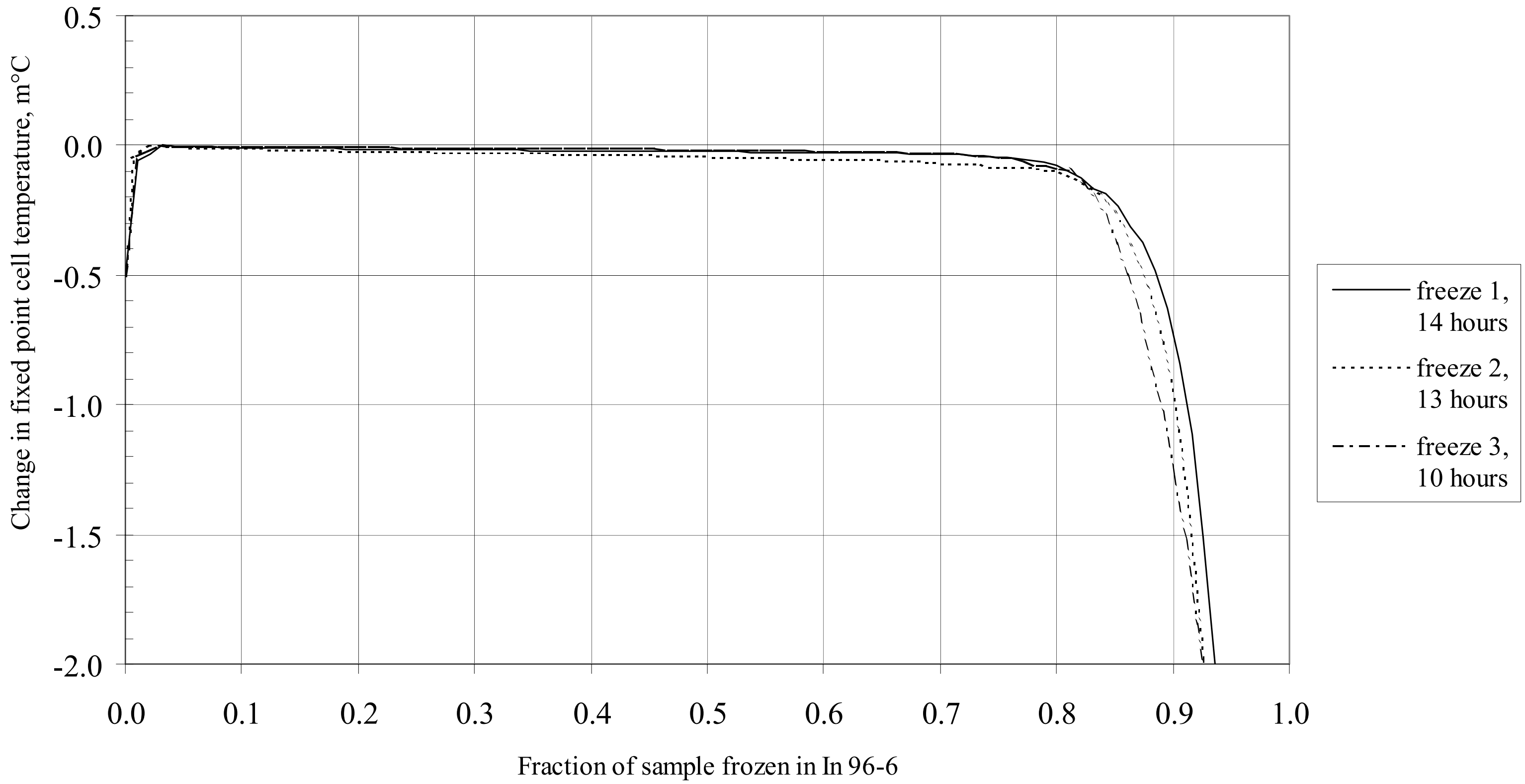

Figure 1. The freezing curves of SRM 2232 Indium DSC Calibration Standard using the "induced inner freeze" preparation technique.

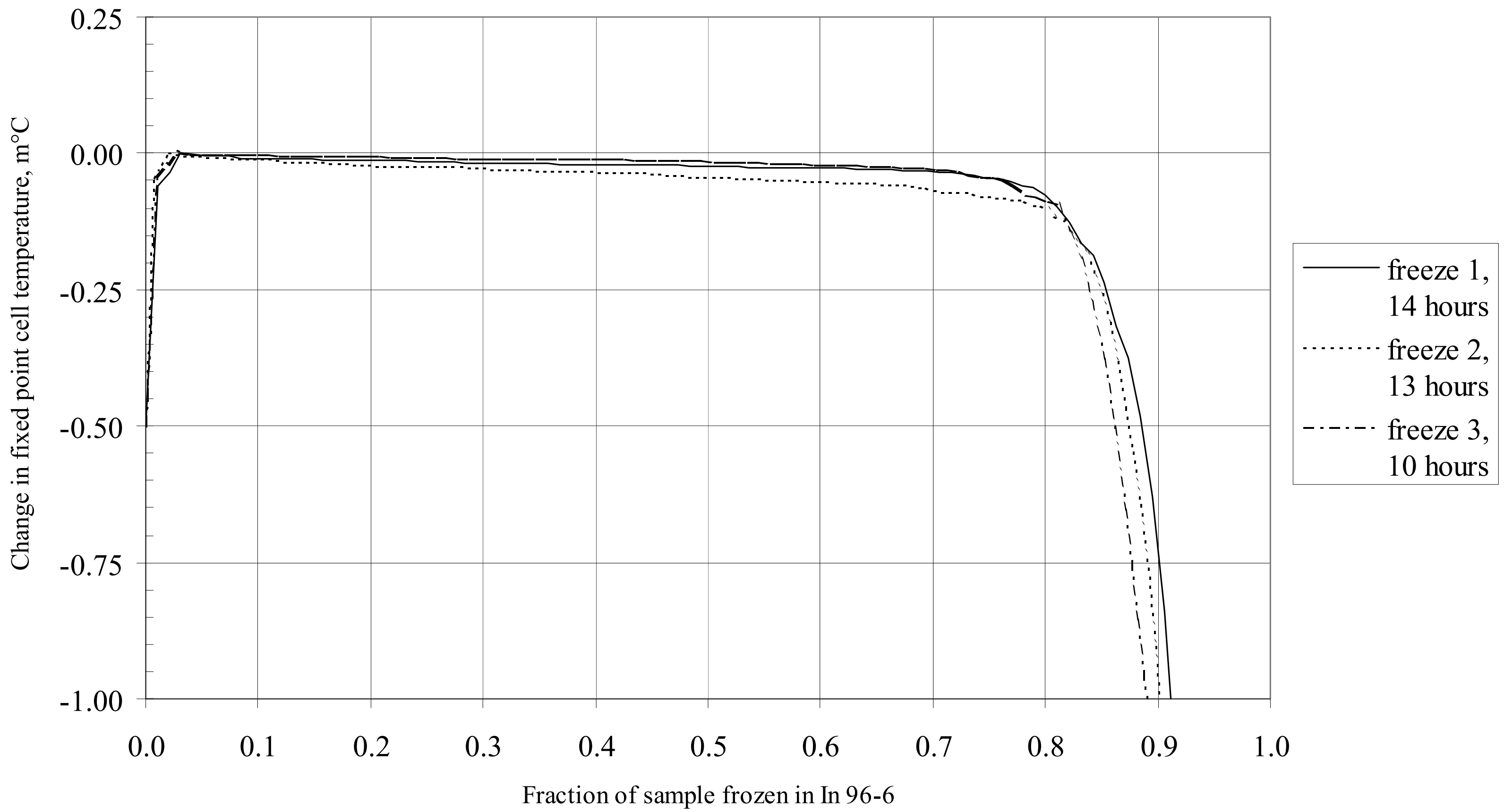

Figure 2. The freezing plateau regions of Figure 1 at greater resolution.

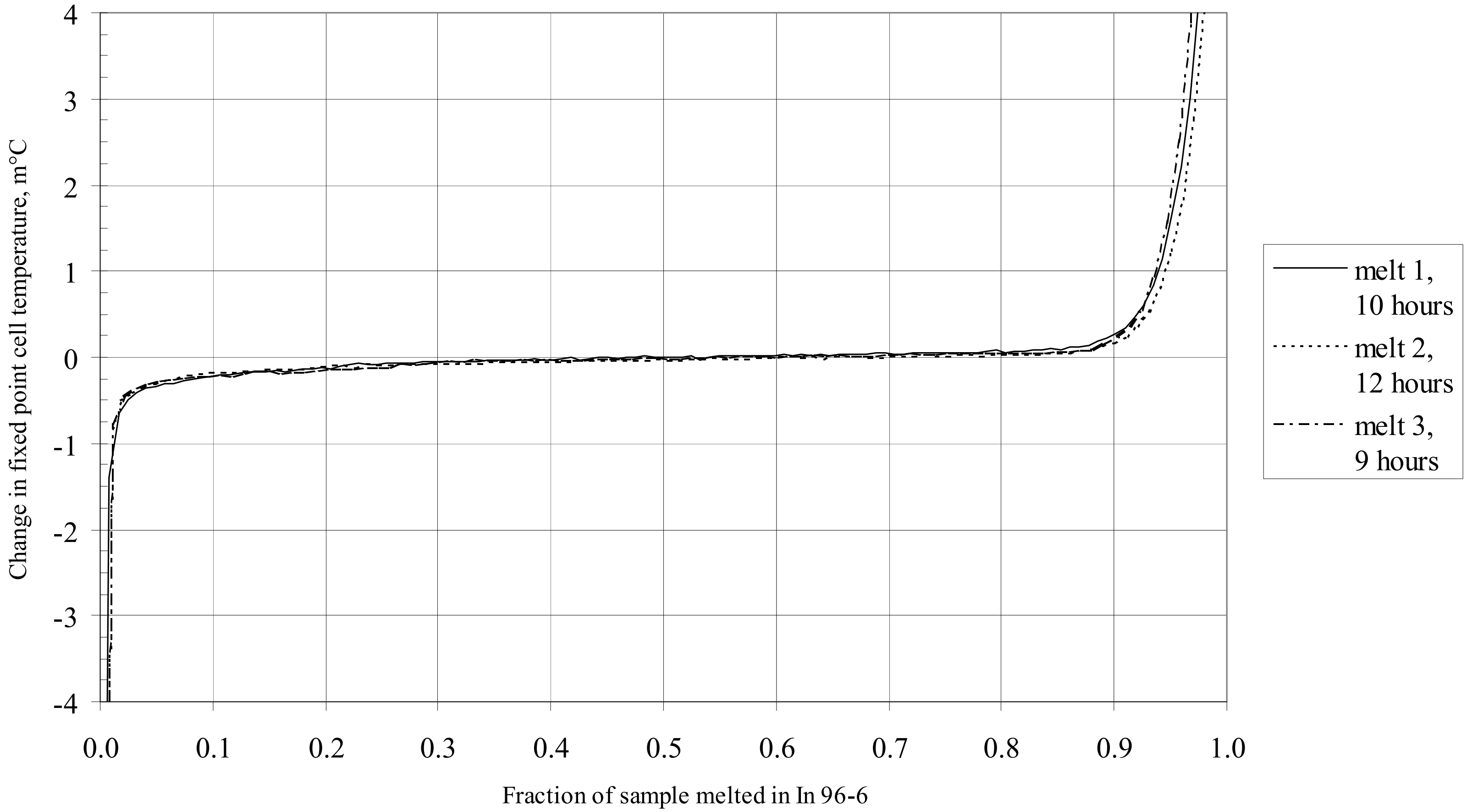

Figure 3. Three melting curves of SRM 2232 Indium DSC Calibration Standard following a slow freeze. Each melt followed the respective slow freeze of Figure 1.

.

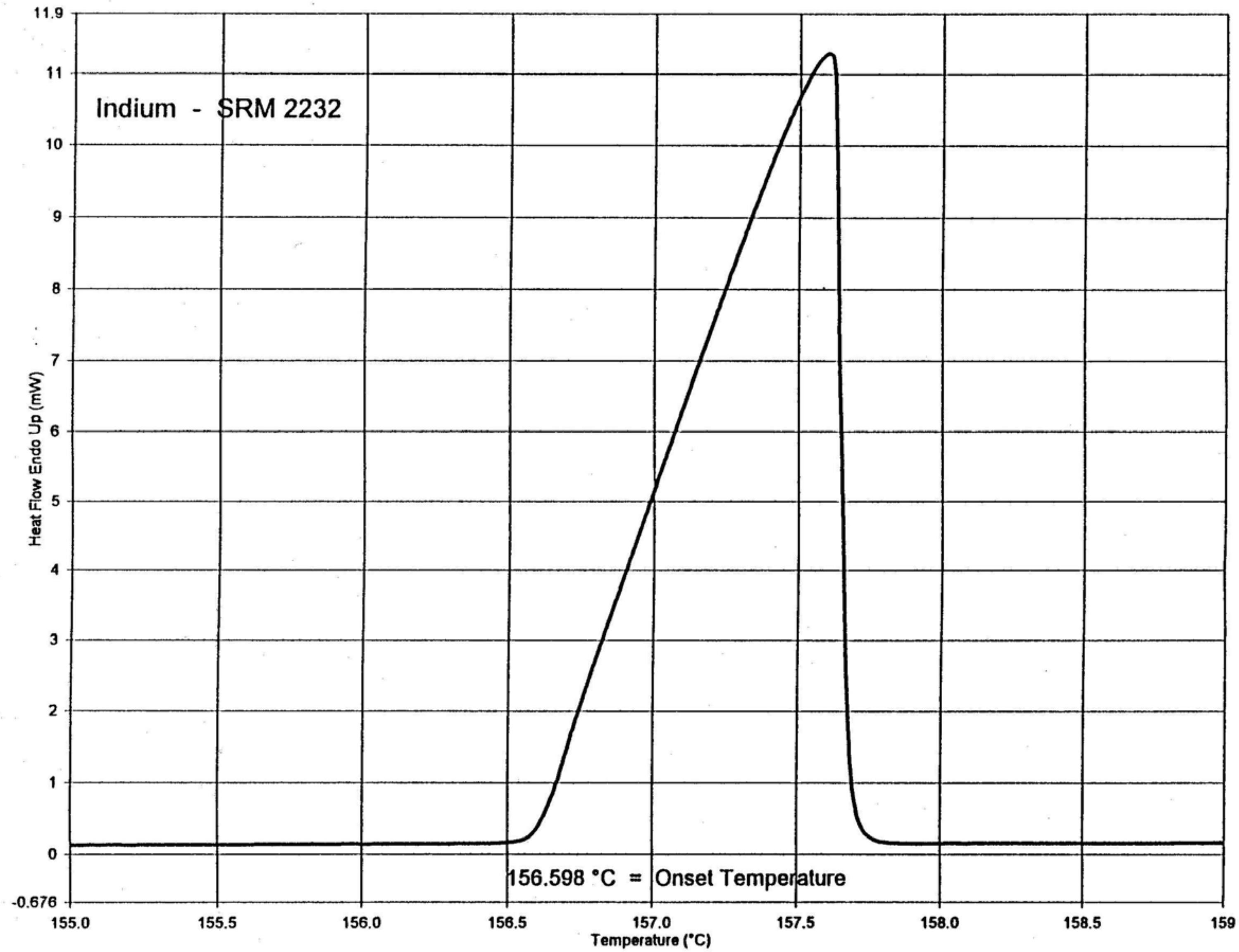

Figure 4. Typical DSC melting curve for SRM 2232 Indium DSC Calibration Standard.