National Institute of Standards & Technology

Certificate

Standard Reference Material® 1747

Tin Freezing-Point Cell

Serial No. Sn 95–2

Certified Freezing Point (231.928 ± 0.000 39) °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 value of 231.928 °C is the temperature assigned to the freezing-point of pure tin (ITS–90) [1]. The fixed-point temperature is realized as the plateau temperature of the freezing curve of the high purity tin when it is frozen slowly. SRM 1747 consists of approximately 1071 g of tin with impurities of 0.4 mg/kg contained in a high purity graphite crucible containing a high purity graphite re-entrant well. The design of the metal fixed-point cells and their assemblies have been described previously [2–5].

An expanded uncertainty (k = 2) [6,7] of 0.39 m°C is assigned to Sn 95-2. The Type A standard uncertainty component of 0.15 m°C is the standard deviation of $W(t_{90})$ values of repeated measurements of the laboratory standard cell with a check Standard Platinum Resistance Thermometer (SPRT) [8]. The Type B standard uncertainty components are obtained from the estimated uncertainty of 0.09 m °C in the freezing-point temperature of the laboratory standard calculated from the impurities listed in the metal assay [8], the temperature difference of 0.10 m°C between the SRM cell and the laboratory standard cell as determined from the intercomparison measurements and the uncertainty of 0.014 m°C in those intercomparison measurements [9].

Notice and Warnings to Users: This fixed-point cell is not of the sealed-cell type. Prior to use, the fixed-point cell should be evacuated and back-filled with an inert gas (Helium or Argon) at least three times. The gas should be controlled to a pressure slightly larger than atmospheric pressure to prevent contaminants from entering the cell.

Expiration of Certification: The certification of **SRM 1747** is valid, within the measurement uncertainty specified, **indefinitely**, provided the SRM is handled in accordance with instructions given in this certificate (see “Instructions for Use”). This certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

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

Statistical consultation was provided by W.F. Guthrie of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

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See Certificate Revision History on Last Page

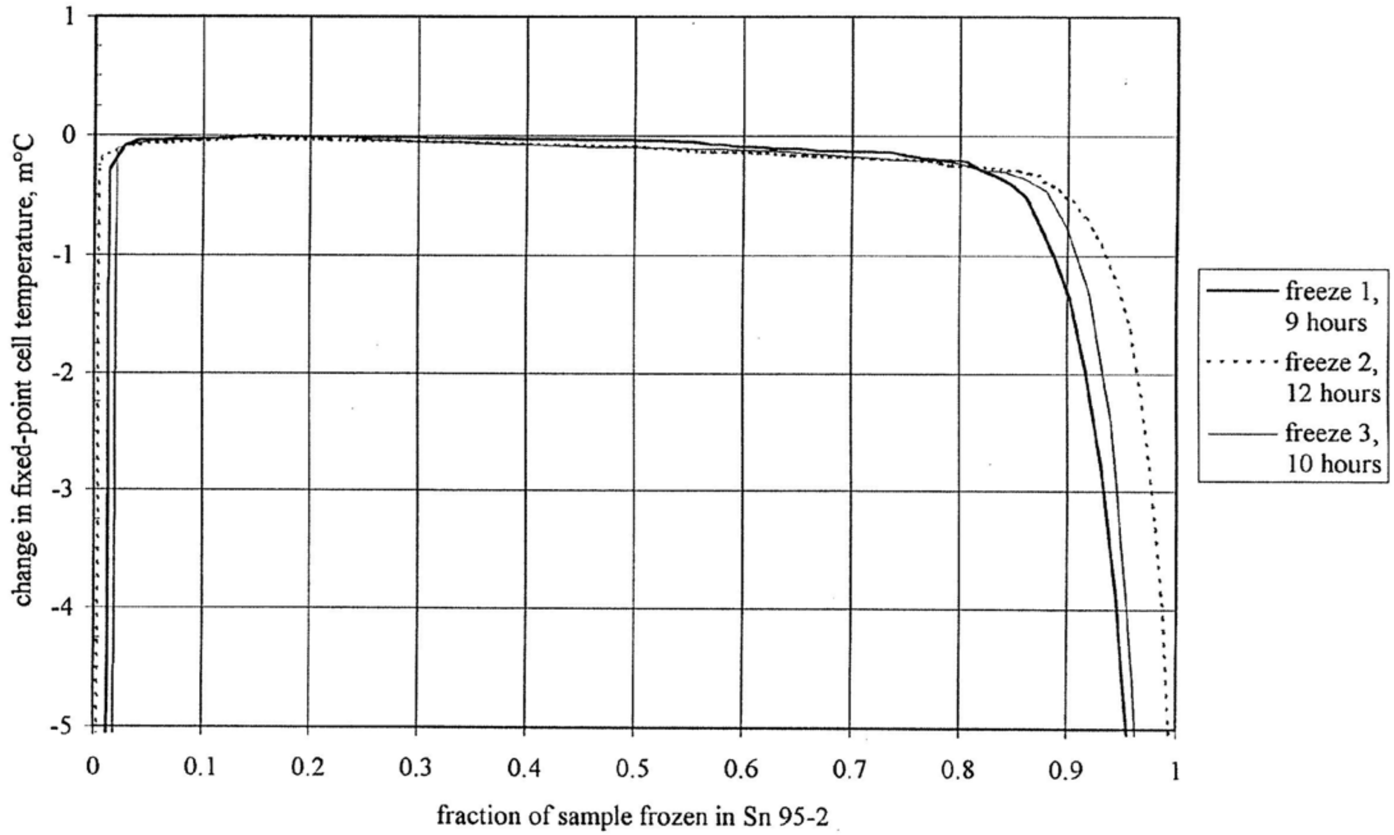

Figure 1. Three freezing curves for the SRM 1747 fixed-point cell Sn 95-2 using the "induced inner freeze" preparation technique.

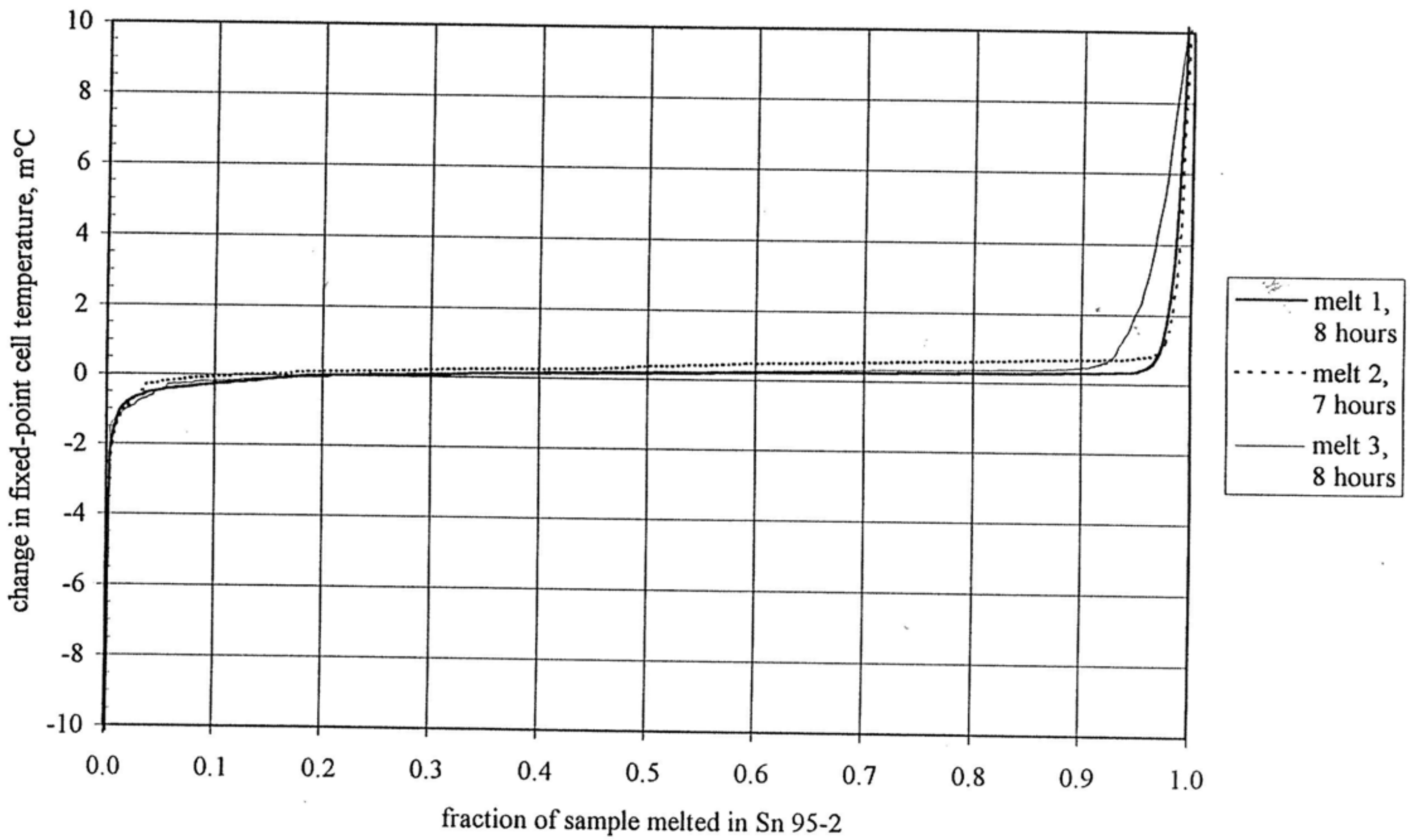

Figure 2. Three melting curves for the SRM 1747 fixed-point cell Sn 95-2 following a slow freeze. Each melt followed the respective slow freeze of Figure 1.

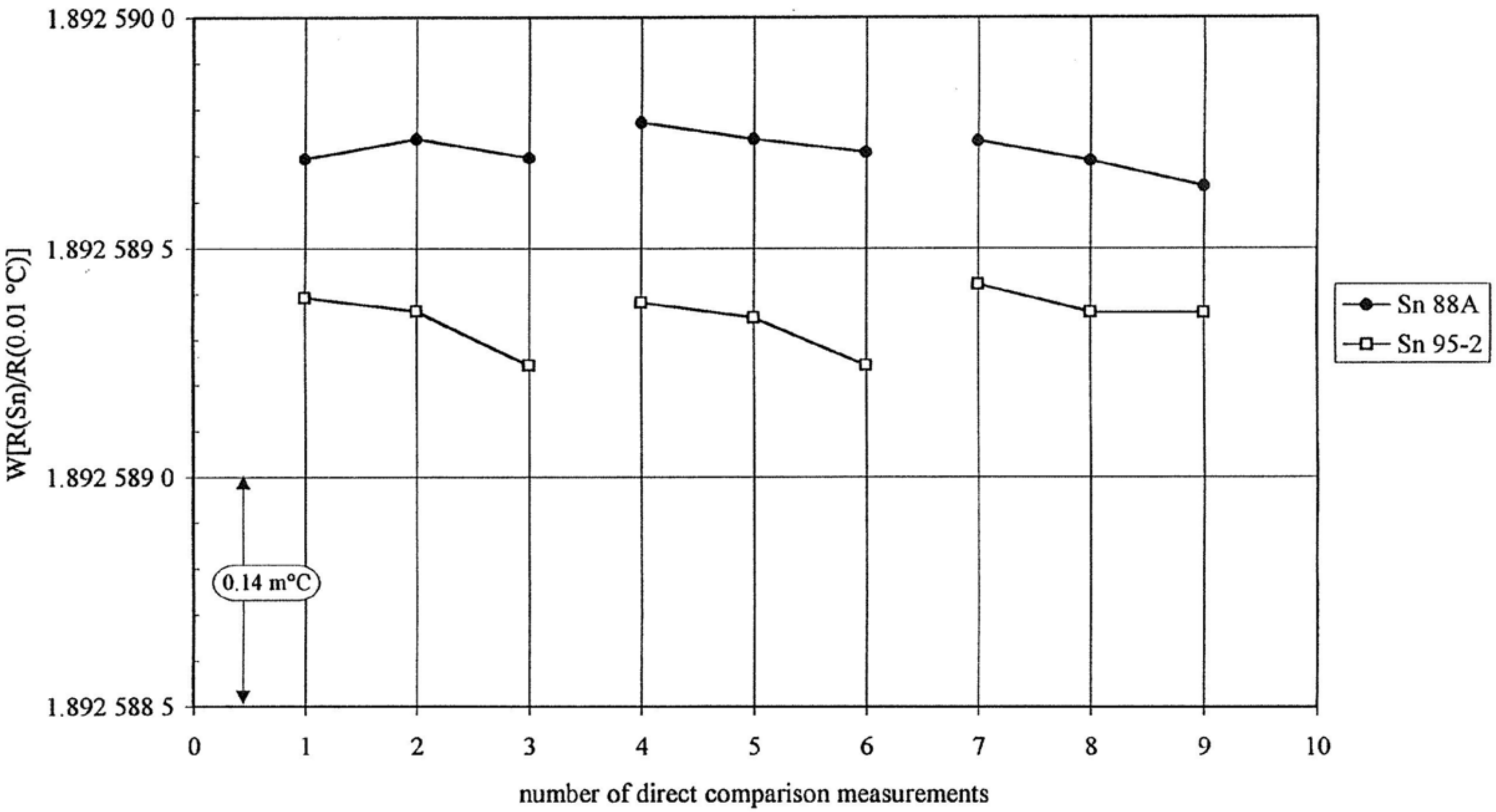

Figure 3. Direct freezing plateau comparison results of SRM 1747 freezing-point cell Sn 95-2 with Sn 88A (laboratory standard). Each set of symbols connected by lines are for the direct comparison measurements made during the simultaneous freezes.

INSTRUCTIONS FOR USE

In assigning a temperature value to fixed-point cells 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/dl = [2.2×10^{-3}] °C/m). The immersion depth of this cell is 18 cm for SPRTs. Using a NIST-manufactured three-zone furnace, the cell has sufficient immersion to track the hydrostatic-head effect over the lowest 10 cm of immersion. If the pressure (p) over the cell during the measurements is not controlled at 101 325 Pa (1 standard atmosphere) [10], a correction (dt/dp = ($3.3 \times 10^{-8)}$ °C/Pa) must be made for the difference in pressure.

Source of Material:[1] The tin metal (Lot M3821) for this SRM was obtained from Johnson Matthey Co., Spokane, WA 99216.

Certification Testing: The thermal tests for the certification of this SRM were performed in a manner similar to that described in reference 3. The cell contains approximately 1071 g of tin obtained from randomly-selected bottles of lot M3821.

The freezing points were prepared using the recommended "induced inner freeze" method [2]. With the metal completely melted, the furnace was set to control at 0.5 °C to 0.75 °C below the freezing-point temperature. The sample cooled and when the SPRT indicated that the cell temperature was within about 10 m °C of the freezing-point value, the cell and the SPRT were withdrawn from the furnace. The cell cooled rapidly and when the SPRT detected recalescence, the cell was replaced in the furnace. In order to freeze a thin mantle of solid around the thermometer well, the SPRT was withdrawn from the cell well, and two fused-silica glass rods, each initially at room temperature, were inserted successively in the well for about 3 min each. Then, the cool SPRT was re-inserted into the cell. Three freezing curves are shown in Figure 1 of this certificate (the region of supercooling and recalescence is not shown, as the curve begins after the reinsertion of the thermometer).

After the metal was slowly and completely frozen, the furnace was set at about 2 °C above the freezing-point temperature to slowly melt the metal over a time of approximately 8 h. Thermometer readings were recorded continuously until the melting was complete. Three melting curves obtained under such conditions are shown in Figure 2 of this certificate.

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 tin freezing-point cell of the Platinum Resistance Thermometer Calibration Laboratory, using a 25.5 Ω SPRT. Three pairs of measurements, with the SPRT being transferred directly from one cell to the other, were made on each of three separate freezes of the two fixed-point cells being intercompared. Although only the first of the three pairs of measurements on a given freeze of a sample was used to determine the temperature difference between the two cells, the other two measurements on the cells provided information on the progress of the freezes (see Figure 3). (Ideally, the difference between the measurements of each of the pairs would be identical.) Only the first 25 % of the freezing curves were used for the intercomparisons. To remove any bias in the measurements, the cell measured first in the sequence was changed from comparison to comparison. The method of direct comparison is described in detail in reference 9.

The electronic measurement equipment for the direct intercomparisons included an Automatic System Laboratories (ASL) F18 resistance-ratio bridge, operating at a frequency of 30 Hz, and a temperature-controlled Tinsley 5685A 100 Ω reference resistor. This reference resistor was maintained at a temperature of (25.00 ± 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 √2 mA with a

[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.

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.

REFERENCES

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[10] *Taylor, B.N.; Guide for the Use of the International System of Units* (SI); NIST Special Publication 811, 1995 ed. (April 1995).

Certificate Revision History: 15 January 2010 (This certificate has been revised to reflect editorial changes); 31 March 1997 (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) 975-2200; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.