National Institute of Standards & Technology

Certificate

Standard Reference Material 1746

Silver Freezing-Point Standard

961.78 °C

International Temperature Scale of 1990 (ITS-90)

This Standard Reference Material (SRM) is intended for use as one of the defining fixed points of the International Temperature Scale of 1990 (ITS-90). [1,2] The temperature value of 961.780 °C is assigned to the freezing point of pure silver. The fixed point is realized as the plateau temperature (or liquidus point) of the freezing curve of slowly frozen high-purity silver. The metal is in the form of millimeter-size "shot" and is provided in 300 g units in mylar envelopes in an atmosphere of argon.

Certified Freezing Point 961.780 ± 0.002 °C

This calibration temperature is on the International Temperature Scale of 1990 (ITS-90). Based on samples tested, the temperature range of melting of the bulk material is not expected to exceed 0.003 °C. Plateau temperatures of freezing curves for samples of this material are expected to differ by not more than 0.001 °C from each other and by not more than 0.002 °C from the assigned temperature.

The silver for this SRM is of high-purity with the total of all elements that affect the freezing-point temperature being less than one part per million.

Source of Material: The silver metal (lot M1282) for this SRM was obtained from Johnson Matthey Co., Spokane, WA.

Precautions for Handling: Any handling procedures on high-purity material are apt to introduce contamination. The "shot" form of this SRM minimizes 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 during handling. Also, a clean laboratory environment is essential.

The overall direction and coordination of the technical measurements leading to the certification of this SRM were performed under the direction of B.W. Mangum of the NIST Process Measurements Division.

Temperature studies on freezing-point cells prepared using metal from randomly-selected envelopes were performed by G.F. Strouse of the NIST Process Measurements Division.

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

Gaithersburg, MD 20899
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Thomas E. Gills, Acting Chief
Standard Reference Materials Program

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Certification: The thermal tests for the certification of this SRM were performed on three freezing-point cells prepared in a manner similar to that described in reference. [3] Each cell contained approximately 1400 g of silver obtained from randomly-selected envelopes of the lot. In assigning a temperature value to realizations of the silver 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 ($dT/d\ell = 5.4 \times 10^{-3}$ °C/m). Also, if the pressure (p) over the cell during the measurements is not controlled at 101325 Pa, a correction ($dT/dp = 6.0 \times 10^{-8}$ °C/Pa) must be made for the difference in pressure from this value.

The freezing-points were prepared using the recommended "induced inner-freeze" method. With the metal completely melted, the furnace was set at about 2 °C below the freezing-point temperature. After supercooling and recalescence had been observed with a high-temperature standard platinum resistance thermometer (HTSPRT) in the cell. The thermometer was removed and a silica-glass rod was twice successively inserted into and removed from the thermometer well at one min intervals to induce freezing of a mantle of 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 of the freezing curve was found to vary no more than 0.001 °C during the first 50 percent 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 2 °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. A typical melting curve obtained under such conditions is shown in Figure 3; some of the same data are plotted at greater resolution in Figure 4.

During the freezing and melting curve measurements, an inert environment of argon gas at 1 standard atmosphere (101325 Pa) pressure was maintained in the cells.

Following the freezing and melting curve measurements, the plateau temperature of the freezing curve of each test cell was compared directly with that of the standard silver freezing-point cell of the Platinum Resistance Thermometer Calibration Laboratory, using an HTSPRT.

REFERENCES

[1] H. Preston-Thomas, "The International Temperature Scale of 1990 (ITS-90)," Metrologia **27**, No. 1, pp. 3, (1990).

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

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

Figure 1. A typical freezing curve of SRM 1746 silver using the "induced inner freeze" preparation technique.

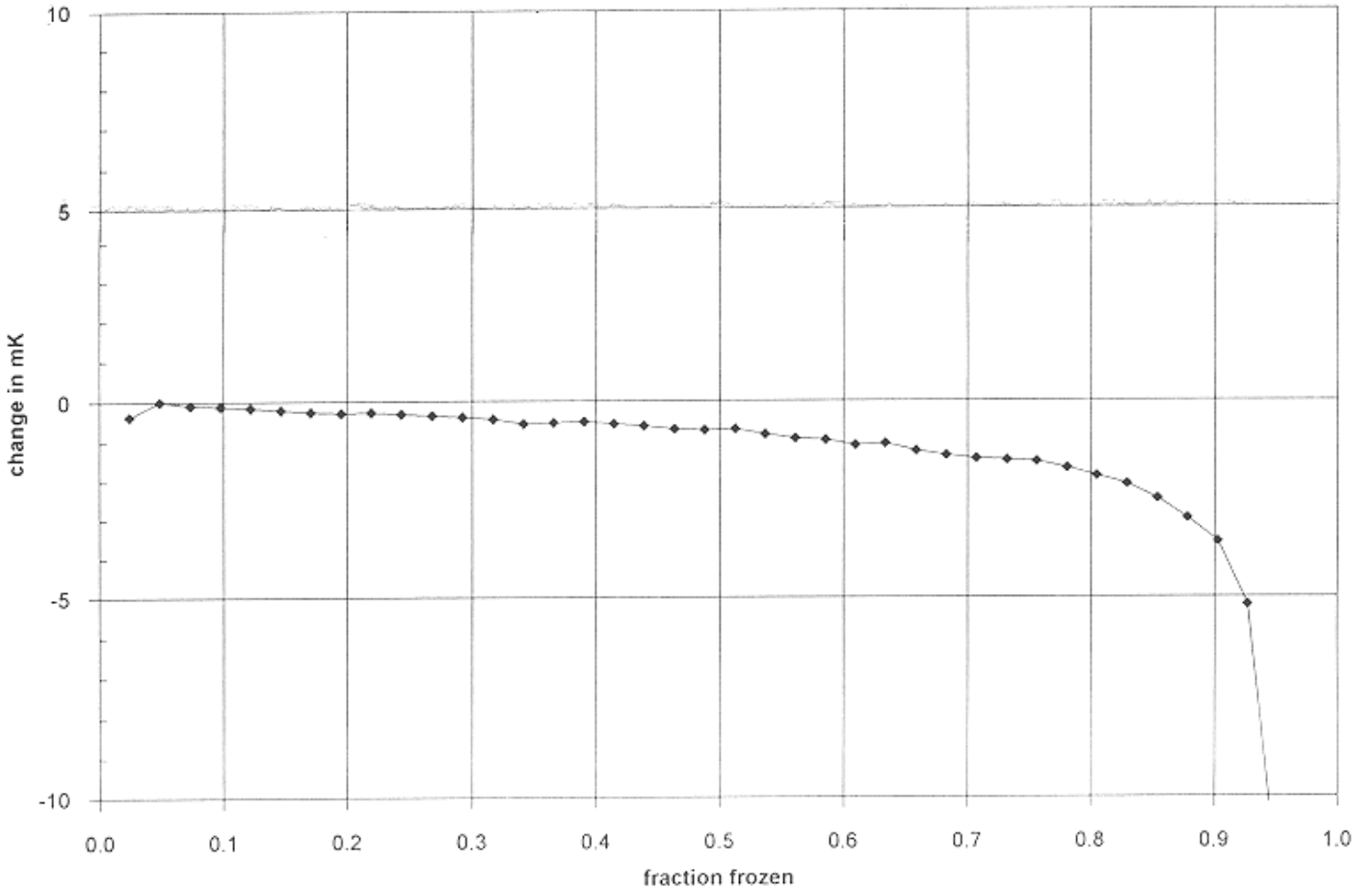

Figure 2. The freezing plateau region of Figure 1 at greater resolution.

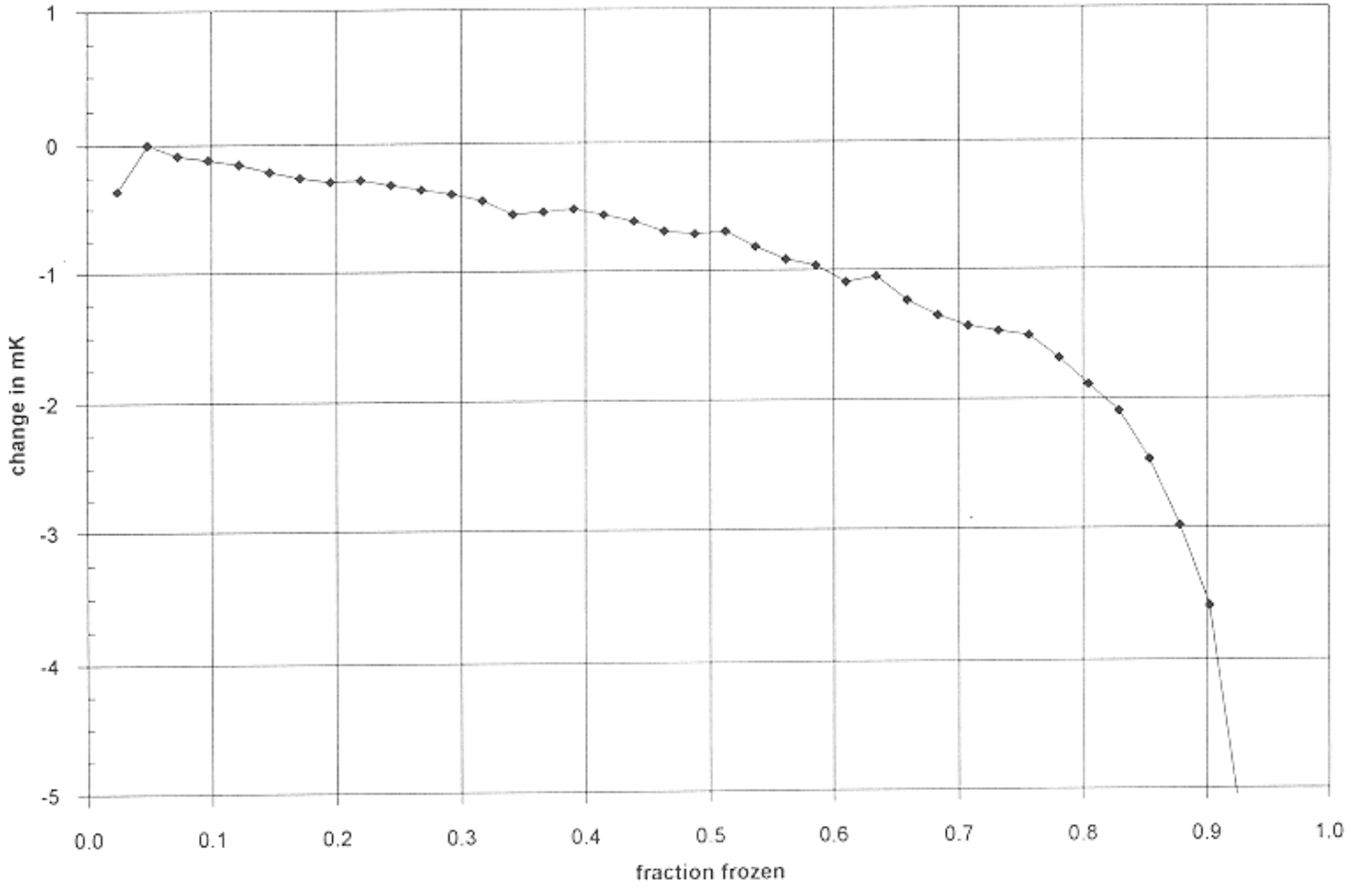

Figure 3. A typical melting curve of SRM 1746 silver following a slow freeze. This melt followed the slow freeze of Figure 1.

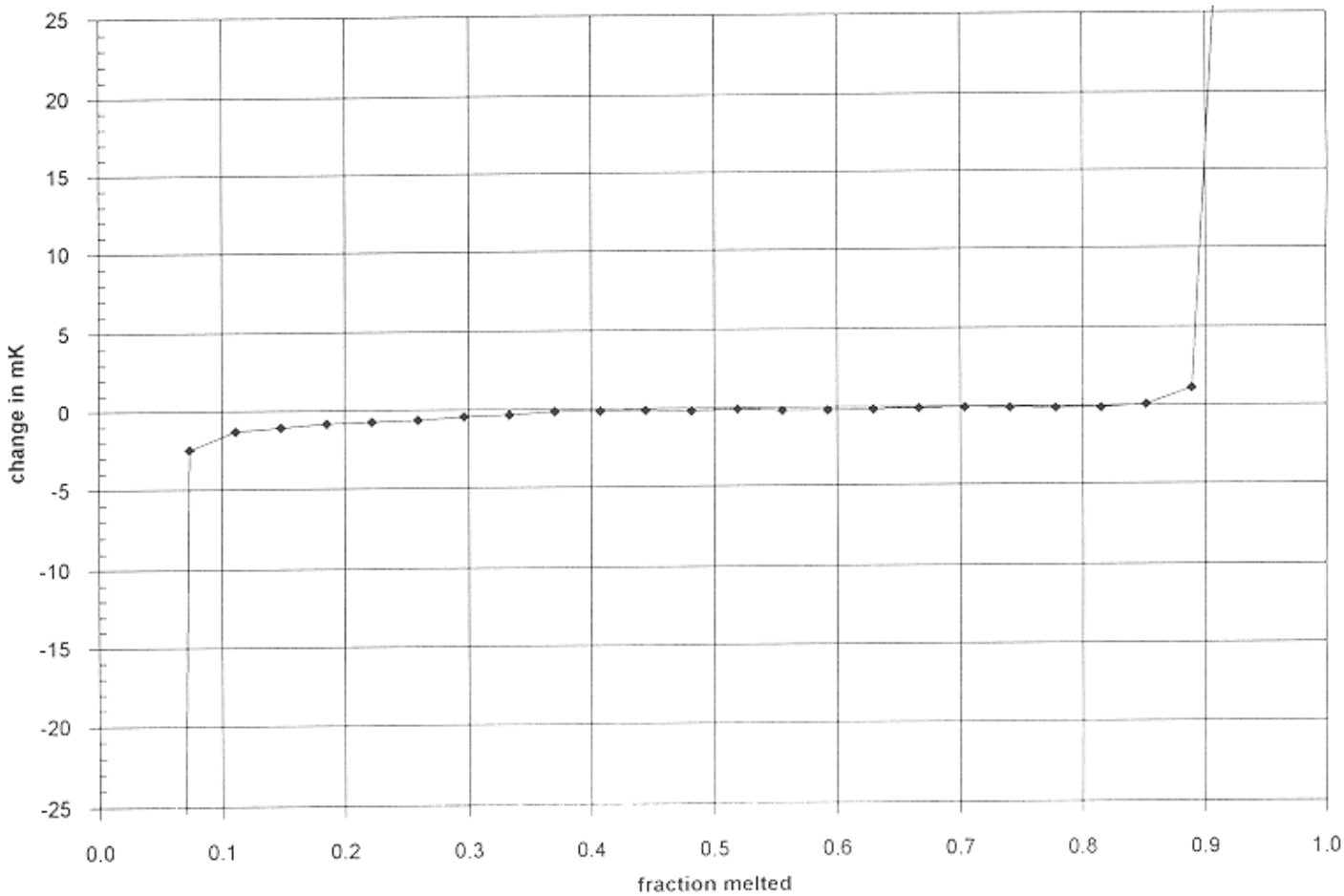

Figure 4. The melting plateau region of Figure 3 at greater resolution.