National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 1744

Aluminum Freezing-Point Standard

660.323 °C
International Temperature Scale of 1990 (ITS-90)

The certified value of 660.323 °C is the temperature assigned to the freezing-point of pure aluminum as one of the defining fixed points of the International Temperature Scale of 1990 (ITS-90) [1]. The fixed point is realized as the plateau temperature (or liquidus point) of the freezing curve of slowly-frozen high-purity aluminum. The metal is in the form of millimeter-size "shot" and is packaged in 200 g units in plastic bottles in the presence of argon.

Certified Freezing-Point Temperature: 660.323 ± 0.001 °C

Based on samples tested, the temperature range of melting of the bulk material is not expected to exceed 0.0015 °C. Temperatures of freezing curve plateau (see Figure 2) for samples of this material are expected to differ by not more than 0.0005 °C from each other and by not more than 0.001 °C from the assigned temperature. The expanded uncertainty (k=2), gives a 95.45% level of confidence for the fixed point measurements and is described in detail in reference 2.

The aluminum for this Standard Reference Material (SRM) is of high-purity, with the total of all elements that affect the freezing-point temperature being less than one mg/kg (part per million).

Source of Material: The aluminum metal (Lot M2075) for this SRM was obtained from Johnson Matthey Co., Spokane, WA 99216.

Notice and Warnings to Users: Any handling procedure for high-purity material is 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.

In assigning a temperature value to realizations of the aluminum 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 ($dt/d\ell = 1.6 \times 10^{-3}$ °C/m). Also, if the pressure (p) over the cell during the measurements is not controlled at 1 standard atmosphere (101325 Pa), a correction ($dt/dp = 7.0 \times 10^{-8}$ °C/Pa) must be made for the difference in pressure.

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

The technical and 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
November 4, 1994

Thomas E. Gills, Chief
Standard Reference Materials Program

(over)

Certification Testing: 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 listed below. Each cell contained approximately 356 g of aluminum obtained from randomly-selected bottles of the lot.

The freezing-point temperature was determined using the recommended "induced inner freeze" method [4]. With the metal completely melted, the furnace temperature was set at about 2 °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 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, after equilibrium was obtained, recording of readings was begun. After equilibrium was established, the measured temperatures of the plateaux of the nine freezing curves of the three samples decreased by no more than 0.0007 °C during the first 50 percent of the freezing curves. 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); some of the same data are plotted at greater resolution in Figure 2.

After the metal was slowly and completely frozen in the above manner, the furnace temperature 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 environment of argon gas at one standard atmosphere (101325 Pa) pressure was maintained in the cells.

Following the freezing and melting curve measurements, the plateau temperature of a freezing curve of each test cell was compared directly with that of the standard aluminum freezing-point cell of the Platinum Resistance Thermometer Calibration Laboratory, using a 25.5 Ω SPRT.

REFERENCES

[1] Preston-Thomas, H., "The International Temperature Scale of 1990 (ITS-90)," Metrologia **27**, 3-10 (1990), Metrologia **27**, 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.R., and Pfeiffer, E.R., "Standard Reference Materials: Application of Some Metal SRM's as Thermometric Fixed Points," Natl. 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)," Natl. Inst. Stand. Tech. Tech. Note 1265, 190 pages, 1990.

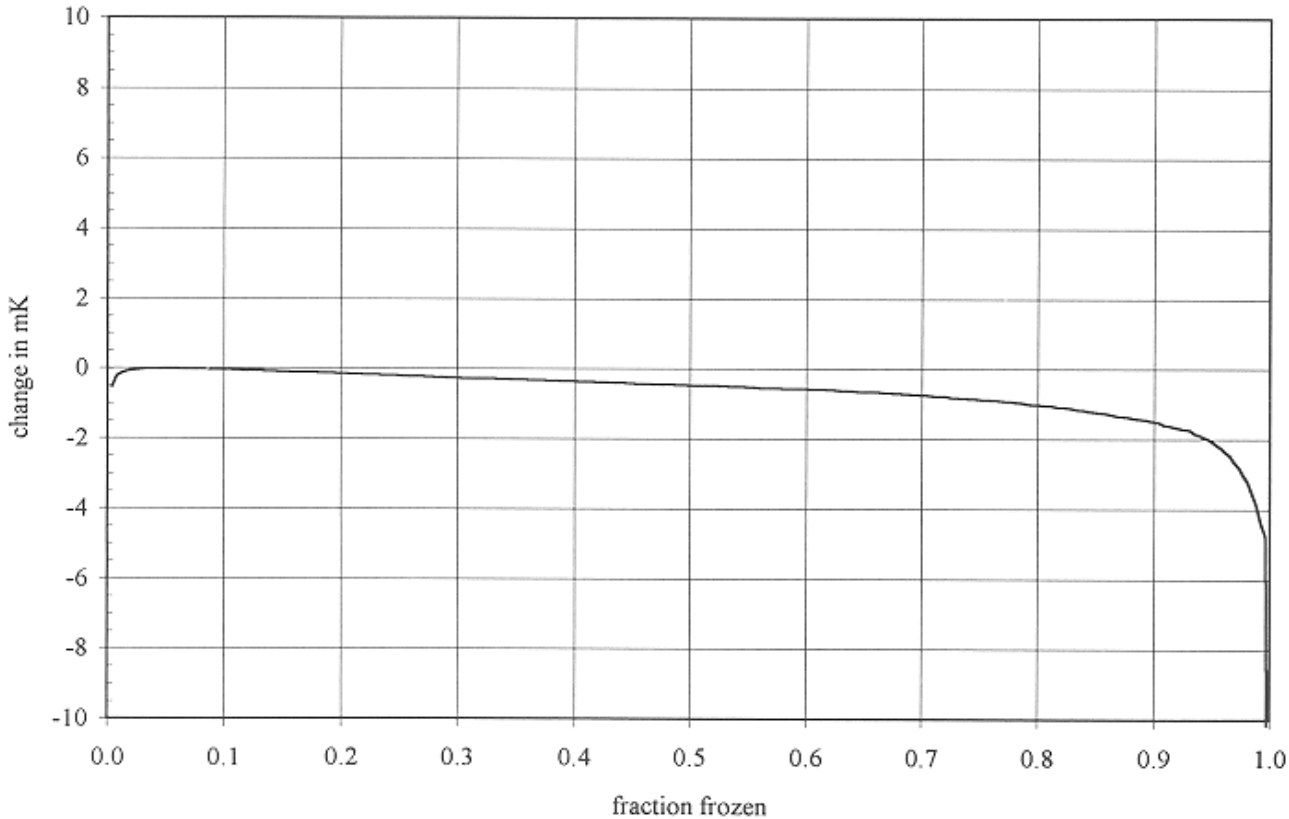

Figure 1. A freezing curve of SRM 1744 aluminum using the "induced-inner-freeze" preparation technique.

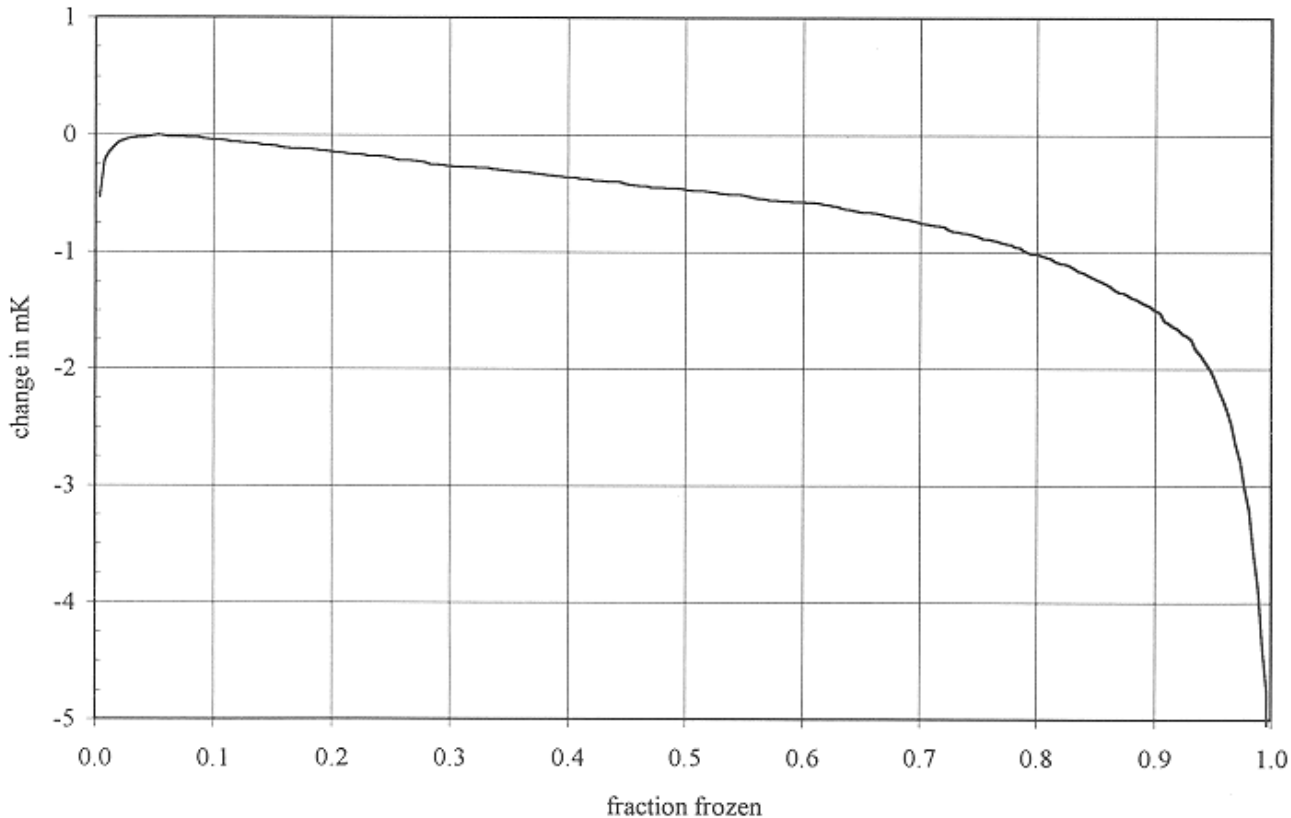

Figure 2. The freezing plateau region of Figure 1 at greater resolution.

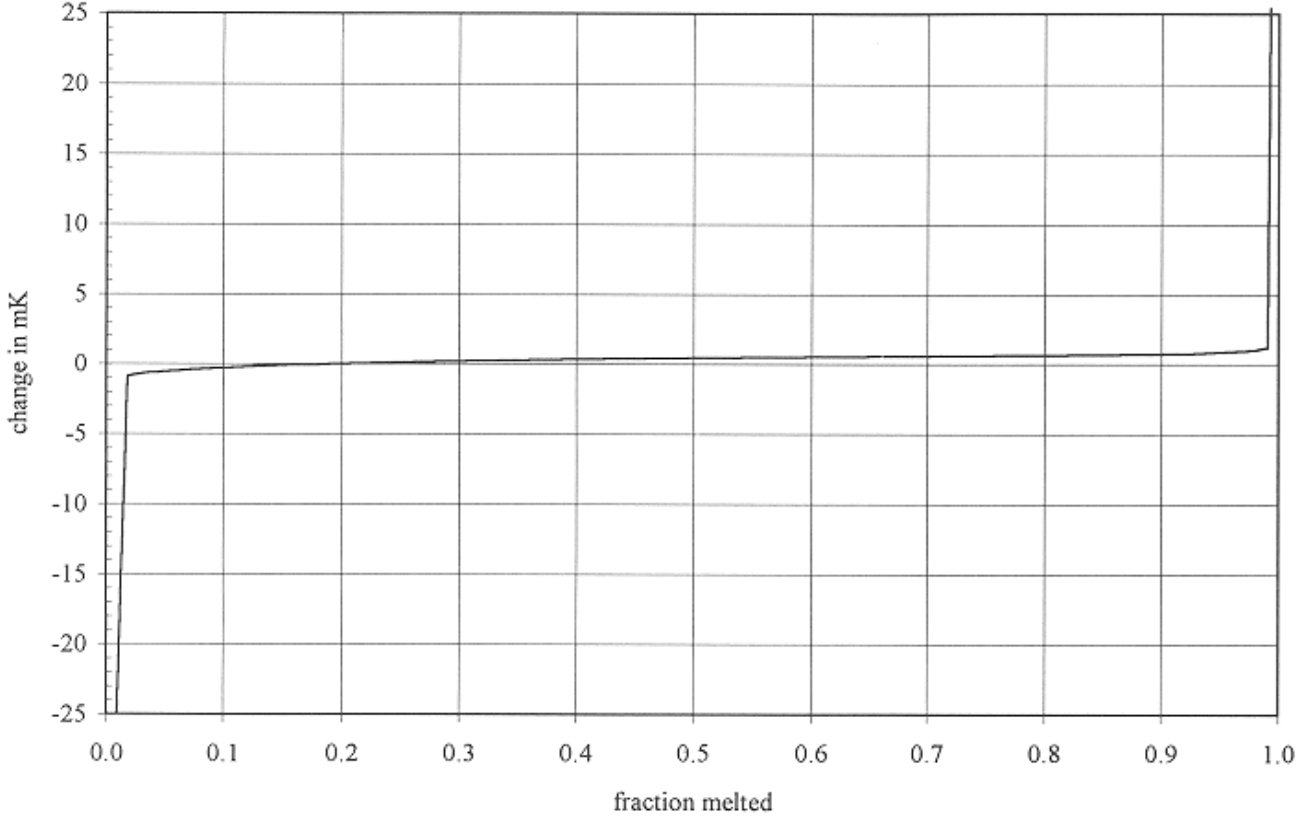

Figure 3. A melting curve of SRM 1744 aluminum following a slow freeze. This melt followed the slow freeze of Figure 1.

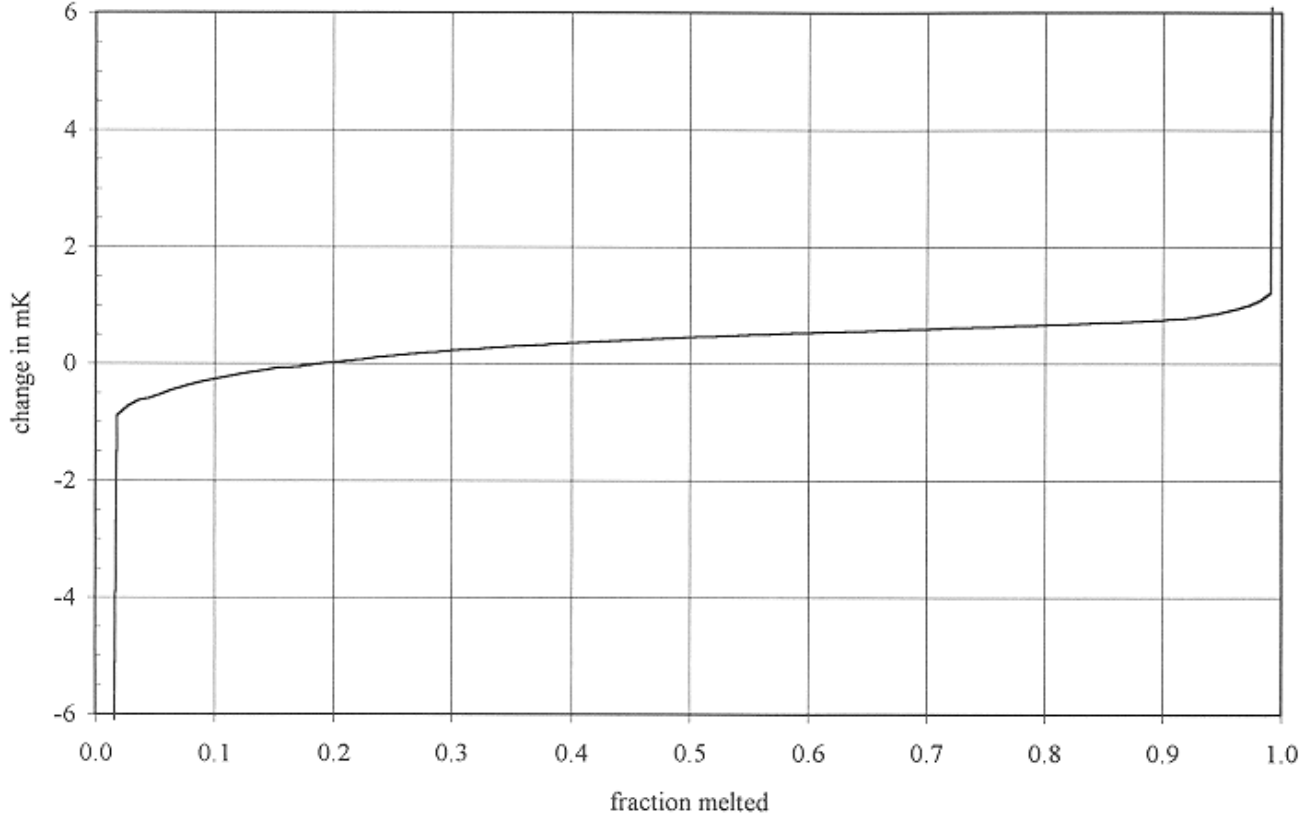

Figure 4. The melting plateau region of Figure 3 at greater resolution.