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U.S. DEPARTMENT OF COMMERCE/Technology Administration
National Institute of Standards and Technology

Standard Reference Materials:

Standard Reference Material 1744: Aluminum Freezing-Point Standard

Gregory F. Strouse

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[2]Some elements at Boulder, CO 80303.

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APPENDIX

Standard Reference Material 1744: Aluminum Freezing-Point Standard

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Abstract

The freezing point of aluminum (660.323 °C) is a defining fixed point of the International Temperature Scale of 1990 (ITS-90). Realization of this freezing point is performed using a fixed-point cell containing high-purity (≥99.9999% pure) aluminum. The aluminum constituting Standard Reference Material 1744 (SRM 1744) has been evaluated, and certified as suitable for use in the realization of the freezing-point temperature of aluminum for the ITS-90. Based on results obtained with three fixed-point cells containing random samples of SRM 1744, the plateau temperature of a freezing curve of the SRM 1744 aluminum is expected to differ by not more than 1 m°C from the ITS-90 assigned temperature. In this document, the methods used for the construction of the aluminum freezing-point cells and for the evaluation of SRM 1744 are described.

Disclaimer

Certain commercial equipment, instruments or materials are identified in this paper in order to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by NIST, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Acknowledgment

The author wishes to thank the Standard Reference Materials Program for their support in the development of this new SRM and B. W. Mangum and G.T. Furukawa for their helpful discussions.

1. Introduction

One of the freezing points required to realize the International Temperature Scale of 1990 (ITS-90) from 420 °C to 962 °C is that of aluminum (660.323 °C) [1]. This freezing point is realized by using a thermometric fixed-point cell containing high-purity (≥99.9999% pure) aluminum metal. Such a fixed-point cell is used for the ITS-90 calibration of standard platinum resistance thermometers (SPRTs) from –189 °C to 660.323 °C and for high-temperature SPRTs (HTSPRTS) from 0 °C to 961.78 °C. To provide evaluated and certified material for this purpose, we have developed a Standard Reference Material (SRM), the Aluminum Freezing-Point Standard (SRM 1744).

The certification of SRM 1744 was performed by evaluating three fixed-point cells (designated Al 94-1, Al 94-2 and Al 94-3) containing random samples of the high-purity (≥99.9999% pure) metal constituting SRM 1744. The certification included the evaluation of freezing and melting curves and the direct comparison with the laboratory aluminum freezing-point standard (Al 78-1) in the NIST Platinum Resistance Thermometry (PRT) Laboratory. Using the three fixed-point cells, these methods of evaluation were used to confirm the purity and the freezing-point temperature of SRM 1744 relative to the aluminum (99.9999% pure) used in Al 78-1. The construction of the aluminum fixed-point cells used to certify SRM 1744 are described and the results from the certification of SRM 1744 are given.

2. Procedure

2.1 SRM 1744 Sample

The high-purity (≥99.9999% pure) aluminum metal used for SRM 1744 was purchased by the NIST Standard Reference Materials Program from Johnson Matthey Company of Spokane, Washington. A 20-kg lot (M2075) of high-purity aluminum metal, in millimeter size button "shot" form (nominally 0.7 cm o.d. by 0.3 cm thick) constitutes SRM 1744. Johnson Matthey Company placed the randomized metal in plastic bottles sealed in an argon atmosphere, each containing 200 g of aluminum. The purification of SRM 1744 was performed by the Johnson Matthey Company. The emission spectrographic assay provided by Johnson Matthey Company with the aluminum shows the total impurity level to be 0.4 µg/g (0.4 parts per million), resulting from 0.3 µg/g of Ca and 0.1 µg/g of Si.

2.2 Aluminum Fixed-Point Cell

Three thermometric fixed-point cells (Al 94-1, Al 94-2 and Al 94-3) were constructed to certify the aluminum metal for use as an ITS-90 freezing-point standard. Each cell contained 356 g of the high-purity metal from randomly selected bottles of lot M2075. As shown in figure 1 (schematic not drawn to scale), the aluminum (J) was contained within a high-purity graphite crucible (K) with a

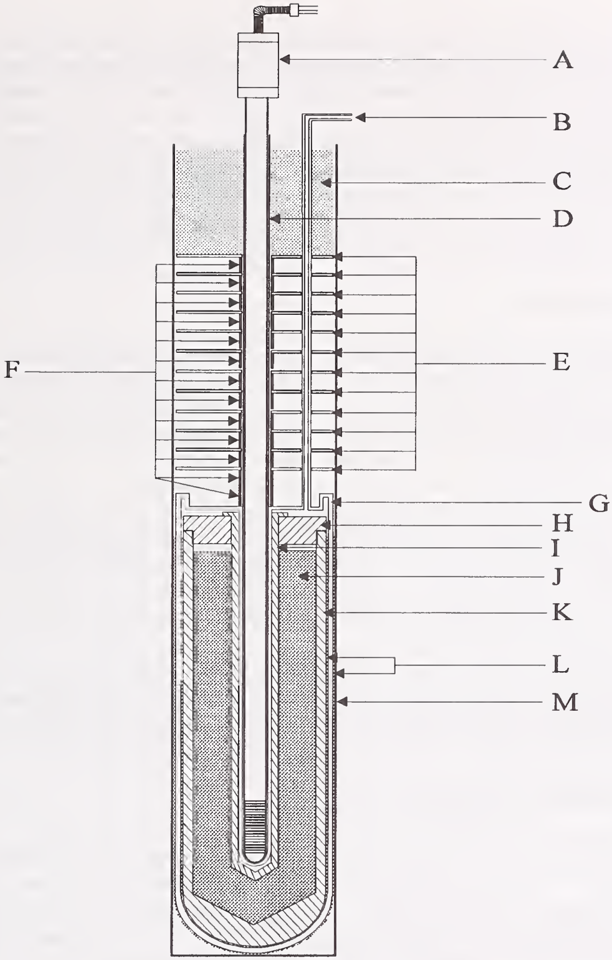

Figure 1. A schematic (not to scale) of an aluminum fixed-point cell assembly showing: (A) a 25.5 Ω SPRT; (B) a matte-finished silica-glass pumping tube; (C) thermal insulation (washed Fiberfrax); (D) a matte-finished, silica-glass thermometer guide tube; (E) twelve Inconel radiation shields; (F) thirteen silica-glass spacers; (G) a silica-glass envelope with a matte-finished, silica-glass re-entrant well; (H) a graphite cap for the graphite crucible; (I) a graphite re-entrant well; (J) aluminum metal SRM 1744; (K) a graphite crucible; (L) silica-glass tape for cushioning; and (M) an Inconel protecting tube.

high-purity graphite cap (H) and a high-purity graphite re-entrant well (I). To prevent possible breakage of the cell, the graphite re-entrant well can float freely up and down (about 0.3 cm) to accommodate shrinking of the metal during freezing and some of the buoyant force of the metal experienced during melting of the metal. The graphite assembly was placed inside a silica-glass envelope (G), with a silica-glass re-entrant well (G) inserted into the graphite well. Axially located in the annular space between the graphite crucible and the silica-glass envelope was a piece of silica-glass tape (L) (46 cm long, 2.5 cm wide and 0.03 cm thick) for cushioning the graphite assembly. The silica-glass re-entrant well had a matte finish to prevent "light piping." The silica-glass envelope also had an attached silica-glass pumping tube (B) (0.6 cm o.d.) that allowed the cell to be evacuated and filled with purified argon during use. This silica-glass pumping tube had also a matte finish to prevent light piping.

The fixed-point cell was inserted into an Inconel protecting tube (M) that was 61 cm long by 5 cm o.d., with a 0.08 cm wall thickness (see fig. 1). Axially located in the annular space between the silica-glass envelope and the Inconel protecting tube and between the silica-glass envelope and the graphite crucible was a piece of silica-glass tape (each 46 cm long, 2.5 cm wide and 0.03 cm thick) for cushioning the fixed-point cell. Above the silica-glass envelope, there were 12 Inconel radiation shields (E) (4.8 cm o.d. by 0.08 cm thick) separated by 1 cm long silica-glass spacers (F), a matte-finished, silica-glass thermometer guide tube (D) and the matte-finished, silica-glass pumping tube. The first radiation shield was 2 cm above the top of the silica-glass envelope. The space remaining above the top radiation shield was filled with Fiberfrax insulation (C). Radiation shields were used to create radiation traps that temper the thermometer and help provide adequate immersion. The thermometer guide tube extended about 0.5 cm above the top of the protecting tube.

The pumping tube extended about 2.5 cm above the Inconel protecting tube and then extended about 5 cm at a right angle for connection to a vacuum/gas-handling system. This fixed-point cell assembly was placed inside a sodium-filled heat-pipe furnace for evaluation of the aluminum. A description of the sodium-filled heat-pipe furnace may be found in Refs. [2] and [3].

2.3 <u>Assembling of the Fixed-Point Cell</u>

Any handling procedure of high-purity material is apt to introduce contamination. The "shot" form is convenient for handling and filling during freezing-point cell construction, while a solid cylinder sample may require cutting and cleaning. By using one-time use polyethylene gloves, every possible effort was made to maintain the purity of the aluminum and other fixed-point cell components that come in contact with the aluminum. Also, a clean laboratory environment was maintained.

Prior to filling the graphite crucible with the aluminum metal "shot," the graphite crucible assembly (crucible, cap and re-entrant well) was placed in a silica-glass furnace tube and "baked-out" at 975 °C under vacuum for 4 hours. This "bake-out" of the graphite was a final purification to remove hydrocarbons and other contaminants that might have been present from the fabrication process. Additionally, the silica-glass tape used for cushioning the graphite crucible in the silica-glass envelope was inserted into the furnace tube and "baked-out" at 975 °C at a partial pressure (34 kPa) of oxygen for 4 hours to remove any organic binders that may have been used during the manufacturing of the tape. The vacuum system used during the fabrication of the aluminum freezing-point cells is described in Ref. [4].

This silica-glass furnace tube used in the assembly process is a 4.8 cm o.d. test tube with a silica-glass pumping tube placed near the top of the furnace tube to allow for evacuation and filling with purified argon during use. The open end of the furnace tube is sealed with either a solid silicon-rubber stopper or a stopper with a vacuum seal that allows a silica-glass push rod to be used for pushing the graphite well into place.

After the graphite assembly was heated and cooled to ambient under vacuum, the furnace tube was purged with purified argon and the graphite assembly removed and placed inside a clean polyethylene bag for storage. To remove any contaminants from the "bake-out," the silica-glass furnace tube was (1) cleaned in hot, soapy water; (2) rinsed with copious amounts of water; (3) the inside soaked in 20% nitric acid-80% distilled water (volume) for 1 hour; (4) rinsed with copious amounts of distilled water and then (5) dried. After each use ("bake-out" or "fill"), the silica-glass furnace tube was cleaned.

High-purity argon gas is necessary for the during both the construction and use of the aluminum fixed-point cell. The gas purification system for argon described in Ref. [4] was designed to remove any hydrocarbons, oxygen and water that would contaminate and react with the molten aluminum.

In order to introduce 356 g of aluminum into the crucible and insert the graphite re-entrant well, two fillings were required. The aluminum "shot" was poured directly into the graphite crucible. Approximately, 290 g of aluminum "shot" could be poured into the graphite crucible for the first "fill." The graphite crucible with the graphite cap and the first filling were placed into the cleaned furnace tube and the furnace tube was placed in the furnace. The system was evacuated for 1 hour and then back-filled with purified argon to a pressure of 34 kPa. This process of pumping and flushing the system was carried out three times with the system finally under vacuum. Upon the final evacuation, the furnace was turned on and the temperature was brought to 670 °C to melt the aluminum sample. After about 2 hours the sample was completely melted, and the furnace was allowed to cool to ambient under vacuum.

For the second "fill," purified argon was introduced, the graphite crucible was removed from the furnace tube, and the remaining metal was added (about 66 g) to metal in the graphite crucible from the first "fill." The graphite re-entrant well was inserted in the graphite crucible as far as possible through the hole in the graphite cap and the assembly was placed in the newly cleaned furnace tube. A silica-glass push rod extended from the bottom of the graphite re-entrant well through a vacuum seal at the top of the furnace tube and for a sufficient distance above the seal at the top of the furnace tube to allow the graphite well to be pushed into place when the aluminum was molten. Finally, the furnace tube was placed in the furnace and pumped and flushed with purified argon using the method described above. When the sample was melted, the graphite re-entrant well was slowly inserted into the molten aluminum by pushing down the silica-glass push rod. When the graphite well was fully inserted, the furnace was turned off and the system allowed to cool to ambient under a partial pressure (34 kPa) of purified argon. The introduction of argon during the cooling of the graphite crucible assembly was used to prevent breakage of the graphite crucible. For unknown reasons, cooling the completed graphite assembly under a vacuum may cause the graphite crucible to split. The graphite crucible assembly was ready to be placed into its silica-glass envelope and fixed-point cell assembly that was described earlier.

3. Experimental Evaluation and Analysis of Results

3.1 Measurement Procedure

The first step in evaluating the fixed-point cells was to obtain three freezing and three melting curves for each of the specimens in the cells, using a 25.5 Ω standard platinum resistance thermometer (SPRT). A commercially-available 30 Hz ac resistance ratio bridge with an ac/dc 100 Ω reference resistor was used to measure the SPRT. A description of the measurement system used in the PRT Laboratory may be found in Ref [3].

In the realization of the freezing point, the recommended "induced inner freeze" method [5, 6] was used. With the metal completely melted, the furnace was set at about 2 °C below the freezing-point temperature. After supercooling and recalescence were observed with a 25.5 Ω SPRT in the fixed-point cell, the furnace temperature was set to about 1 °C below the freezing-point temperature for the duration of the freeze. Next, the thermometer was removed and a silica-glass rod was twice successively inserted into and removed from the thermometer well at 1 minute intervals to induce freezing of a mantle of metal around the graphite re-entrant well. Finally, the thermometer was then reinserted into the cell and, after equilibrium was obtained, the recording of readings was started, using an excitation current of 1 mA.

3.2 Analysis of Freezing Curves

Figures 2 to 4 show the freezing curves for each of the three fixed-point cells (the region of supercooling and recalescence is not shown, as the curves begin after the reinsertion of the thermometer). Using an excitation current of 1 mA, thermometer readings were recorded continuously until the freezing was complete. The average length of a freeze for the three cells was 16 hours. The calculated temperature depression when 50% of the metal was frozen and the shapes from the freezing-curve plateaus were used to estimate and confirm the overall purity of the sample in the cell.

In most cases, including the two known impurities in the present high-purity samples will cause a depression in the temperature of a freezing point. Using Raoult's Law of dilute solutions, calculations may be made to estimate the expected temperature depression when 50% of the metal has frozen during a freezing curve realization [7]. Using the known type and amount of impurities given in the emission spectrographic assay, the molar fraction of impurities was calculated. The total impurity level (2.98 X 10^{-7} mol) and Raoult's Law of dilute solutions give a calculated depression in temperature estimated to be 0.2 m°C. Another method of estimating the expected temperature depression when 50% of the metal has frozen during a freezing curve realization, was made using binary phase diagrams [8]. The binary phase diagrams for Al-Ca and Al-Si give a calculated depression in temperature estimated to be 0.3 m°C. As determined from the freezing curve plateaus in figures 2 to 4, the average estimated temperature depression from the extrapolated 0% frozen metal (time of recalescence) to the 50% frozen metal of the flat section of the plateaus were 0.6 m°C, 0.5 m°C and 0.6 m°C for Al 94-1, Al 94-2 and Al 94-3, respectively, which are several times larger than the calculated values. Differences between the calculated and the experimentally derived temperature depressions may indicate either an uncertainty in using Raoult's Law of dilute solutions, in using binary phase diagrams, the sample contained additional impurities not accounted for in the emission spectrographic assay or perhaps additional impurities were added to the metal during the construction of the fixed-point cells. In estimating the expected temperature depression, Raoult's Law of dilute solutions and binary phase diagrams are intended to provide a guideline and should not be strictly applied. Additionally, there is an uncertainty in the extrapolation method chosen to experimentally derive the temperature depression.

Comparisons of the average temperature depressions when 50% of the metal of the three fixed-point cells was frozen were made with Al 78-1. The average temperature depression from the extrapolated 0% frozen metal (time of recalescence) to the 50% frozen metal of the flat section of the plateaus was 0.7 m°C for Al 78-1. The smaller temperature depressions for the three cells relative to the laboratory standard indicate that the SRM metal is of slightly higher purity than that of the reference cell.

3.3 Analysis of Melting Curves

After the aluminum was slowly and completely frozen in the above manner, the furnace temperature was set at about 1 °C above the freezing-point temperature to slowly melt the metal over a time of approximately 10 hours. Using an excitation current of 1 mA, thermometer readings were recorded continuously until the melting was complete. Figures 5 to 7 show the melting curves for each of the three fixed-point cells. From the graphs, the average temperature range of the melting curves were 1.4 m°C, 1.0 m°C and 1.2 m°C for Al 94-1, Al 94-2 and Al 94-3, respectively.

During the melting of the aluminum, two liquid-solid interfaces are formed. One liquid-solid interface is next to the inner wall of the graphite crucible and the second liquid-solid interface is near the graphite re-entrant well. This second liquid-solid interface is formed where the lower-purity metal solidified at the end of the previous slow freeze. The lower-purity metal has a slightly lower freezing and melting temperature causing this second liquid-solid interface to form during the melt. The height of the freezing range and the melting range are about the same which is expected if an inner melt is formed during the melt [9].

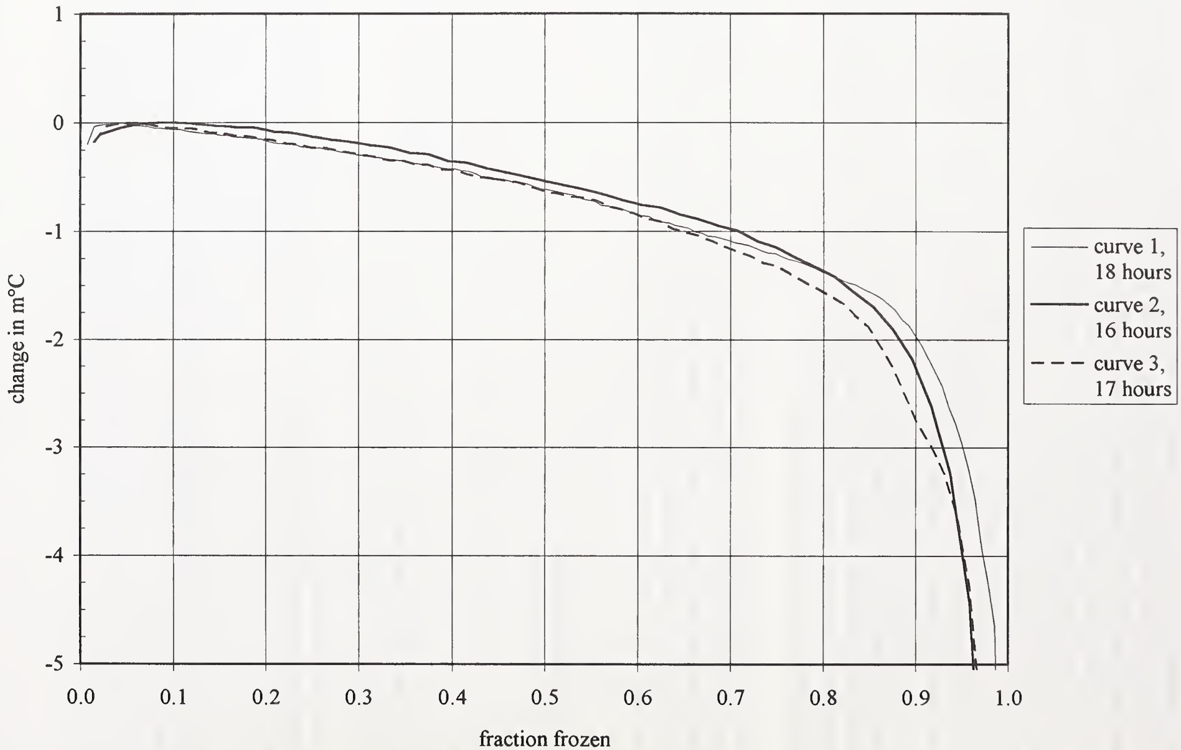

Figure 2. Three freezing curves for the aluminum fixed-point cell Al 94-1 using the "induced inner freeze" preparation technique.

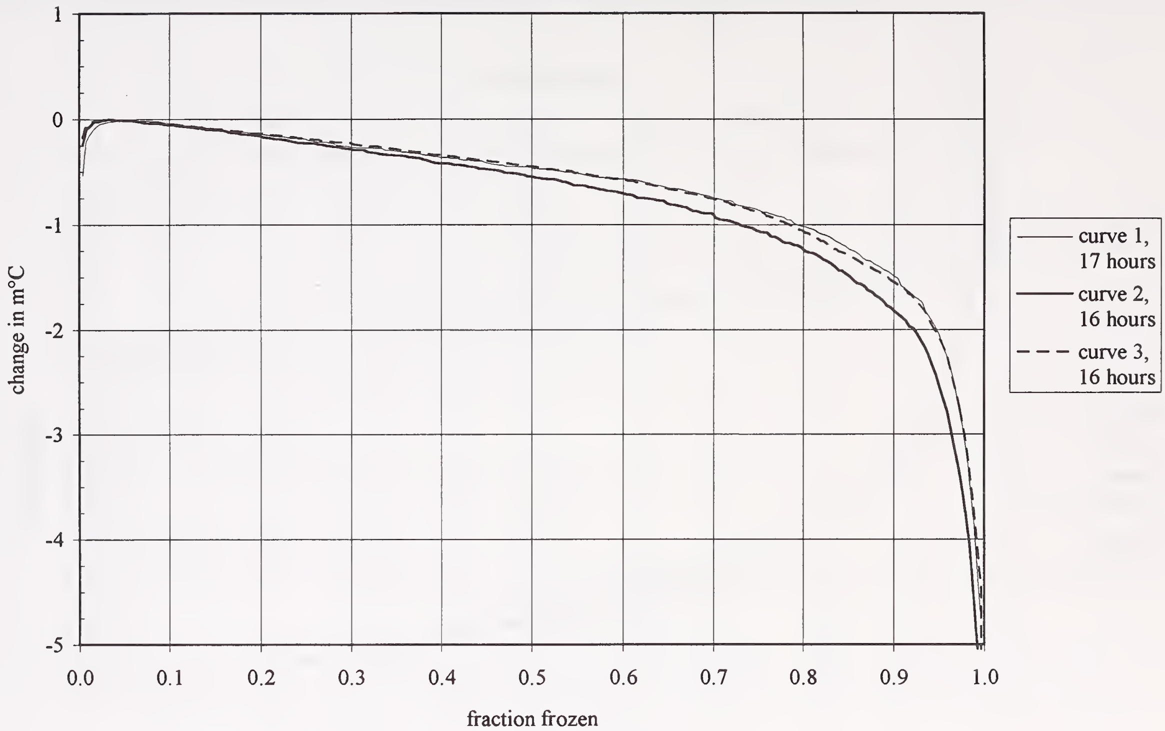

Figure 3. Three freezing curves for the aluminum fixed-point cell Al 94-2 using the "induced inner freeze" preparation technique.

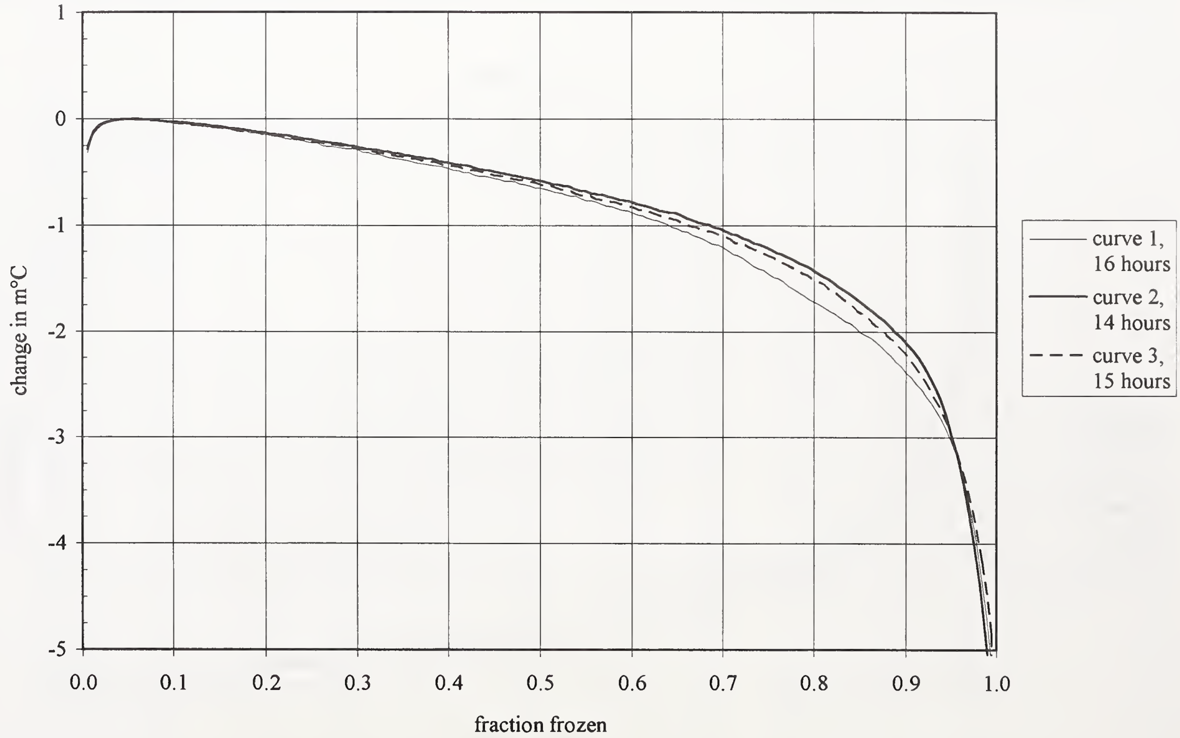

Figure 4. Three freezing curves for the aluminum fixed-point cell Al 94-3 using the "induced inner freeze" preparation technique.

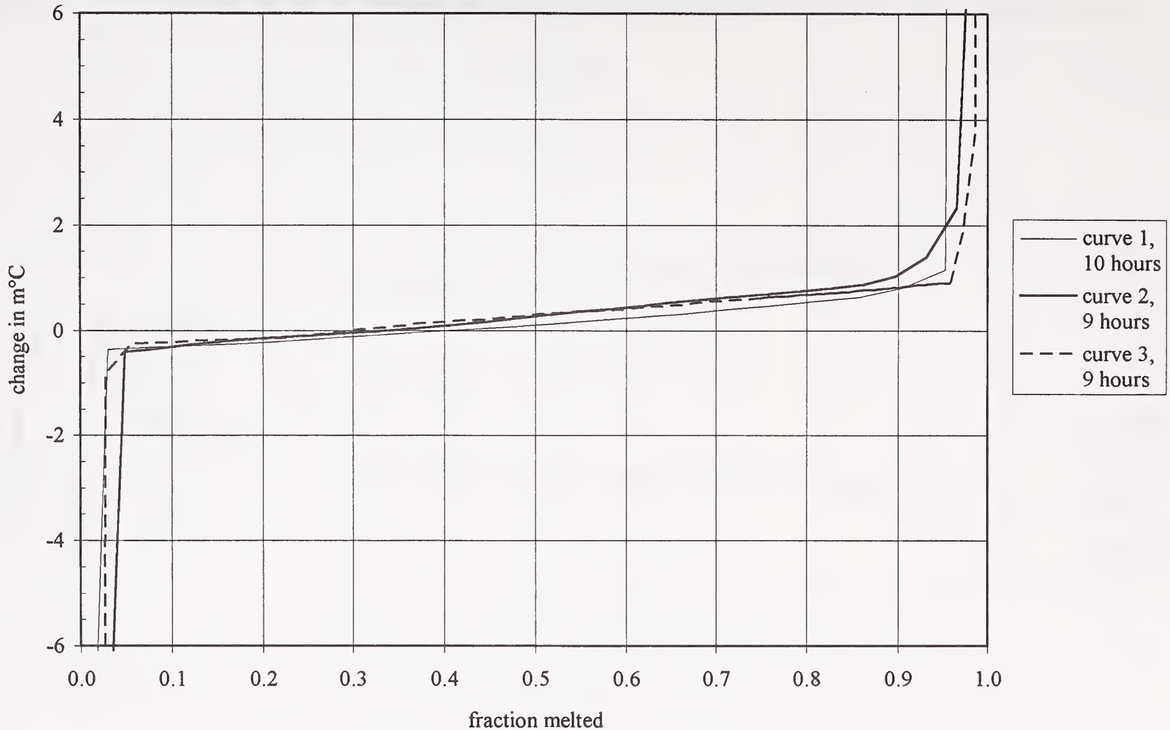

Figure 5. Three melting curves for the aluminum fixed-point cell Al 94-1 following a slow freeze. Each melt followed the respective slow freeze of Figure 2.

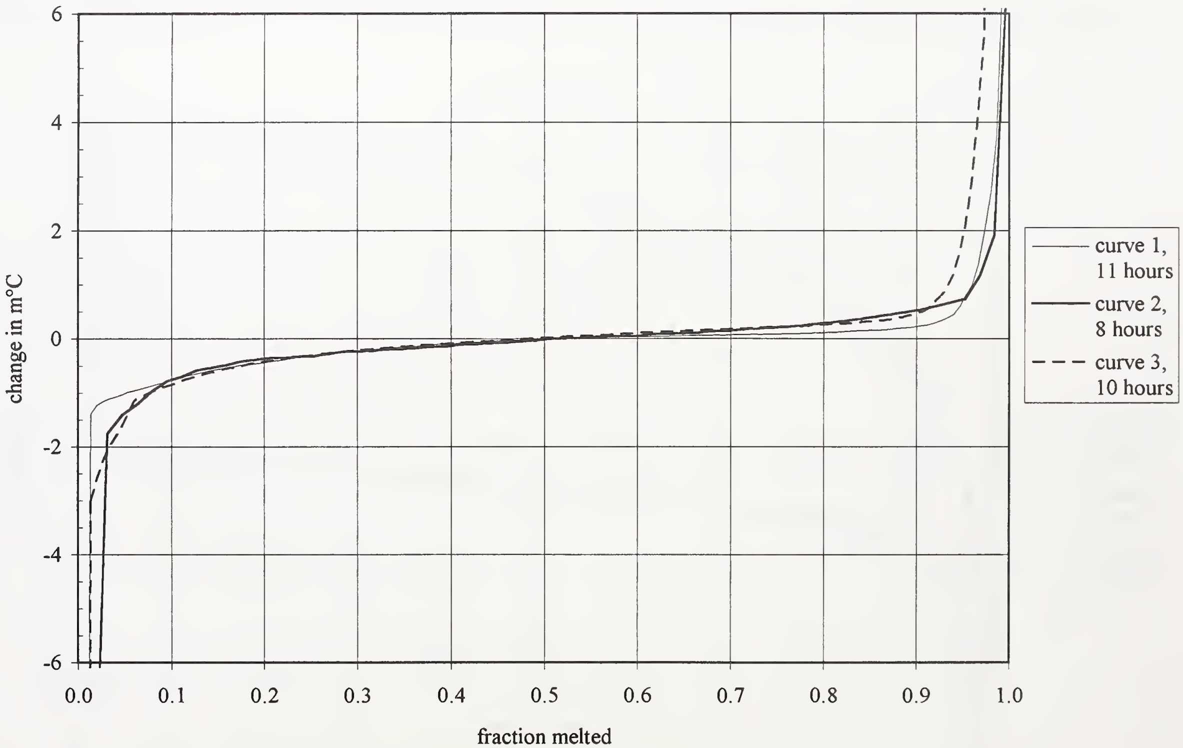

Figure 6. Three melting curves for the aluminum fixed-point cell Al 94-2 following a slow freeze. Each melt followed the respective slow freeze of Figure 3.

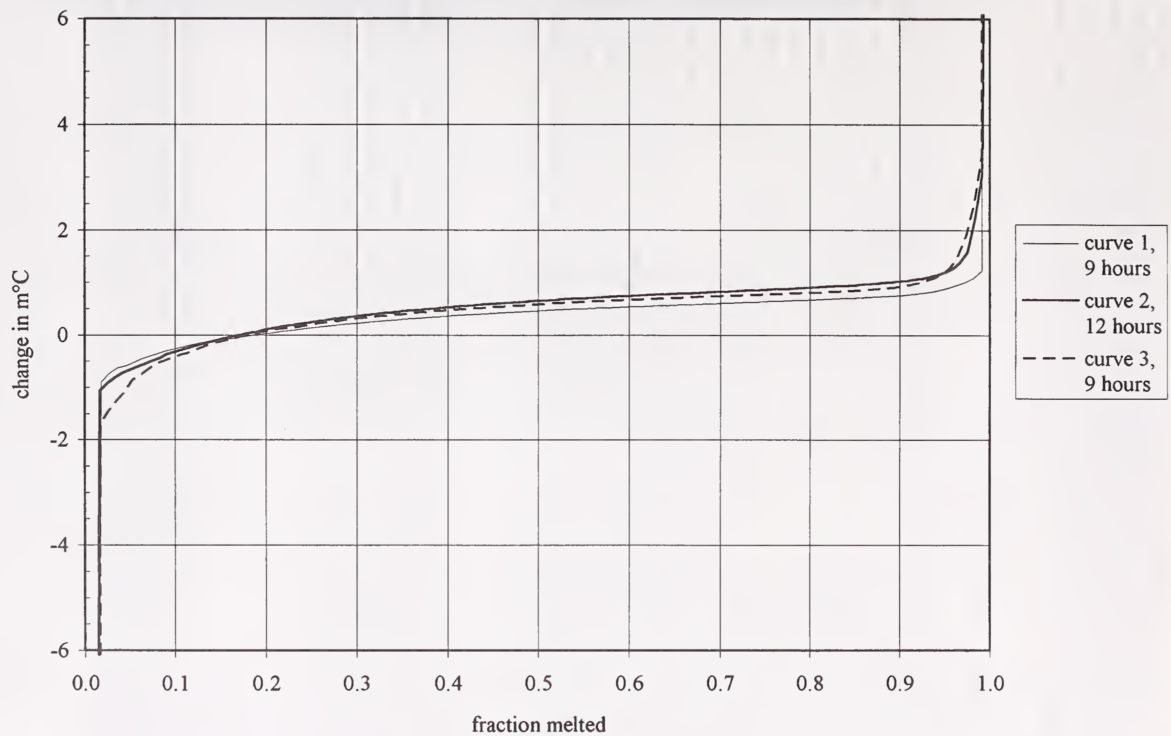

Figure 7. Three melting curves for the aluminum fixed-point cell Al 94-3 following a slow freeze. Each melt followed the respective slow freeze of Figure 4.

The temperature range of a melting curve is not indicative of the purity of the metal. Using the melting curve to indicate purity is complicated by the fact that the shape and range of a melting curve will depend upon the history of the previous freezing of the metal in the fixed-point cell [10]. A slow freeze causes the impurities to be segregated which in turn causes a large melting range. A fast freeze causes a homogenous mixture of the impurities with metal sample which in turn causes a small melting range.

3.4 Direct Comparison of Fixed-Point Cells

The second part of the evaluation was a direct comparison of the fixed-point cells under test with the laboratory standard fixed-point cell (Al 78-1) to determine their freezing-point temperatures relative to that of the reference cell. This was obtained by realizing simultaneous freezes for the two cells (Al 94-X and Al 78-1, where X was 1, 2 or 3) and making three sets of alternate measurements, at equal time intervals, on their freezing-curve plateaus, using an SPRT. Measurements of each cell were made using an SPRT with excitation currents of 1 mA and 1.414 mA to permit extrapolation to zero-power dissipation (0 mA). Corrections were made for any differences in pressure and hydrostatic head effects in each cell. Each cell was measured using an SPRT three times during the direct comparison and this procedure was repeated two times. Figure 8 shows the results of the direct comparison of the three SRM cells with the laboratory standard. The set of matching symbols (open and closed) are for the direct comparison measurements of Al 94-X (where X is 1, 2 or 3) compared with Al 78-1. Each set of symbols (open or closed) connected by lines are for the direct comparison measurements made during the simultaneous freezes. The average temperature difference of the first readings of each direct comparison showed that Al 94-1, Al 94-2 and Al 94-3 were 0.1_9 m°C, 0.3_2 m°C and 0.1_5 m°C hotter than the laboratory standard. An expanded uncertainty of 1.0 m°C (k=2) is assigned to the laboratory reference cell. The basis for that uncertainty is given in Refs. [11] and [12].

For the metal to be certified as a freezing-point standard, the fixed-point cells containing samples of the metal must have a freezing-point temperature that is in agreement with the laboratory reference cell containing high-purity metal to within the uncertainties of the measurements. If the purity of the metal in the new fixed-point cell is greater than that of the reference cell, then it may be of even higher quality, as indicated by being "hotter" than the laboratory standard. A cell that is "hotter" usually has fewer impurities, since impurities in these samples will usually decrease the freezing-point temperature.

3.5 Application

In assigning a temperature value to realizations of the aluminum freezing point, corrections must be applied for the 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) [1]. Also, if the pressure (p) over the cell during the measurements is not controlled at 101 325 Pa (1 standard atmosphere), a correction ($dt/dp = 7.0 \times 10^{-8}$ °C/Pa) must

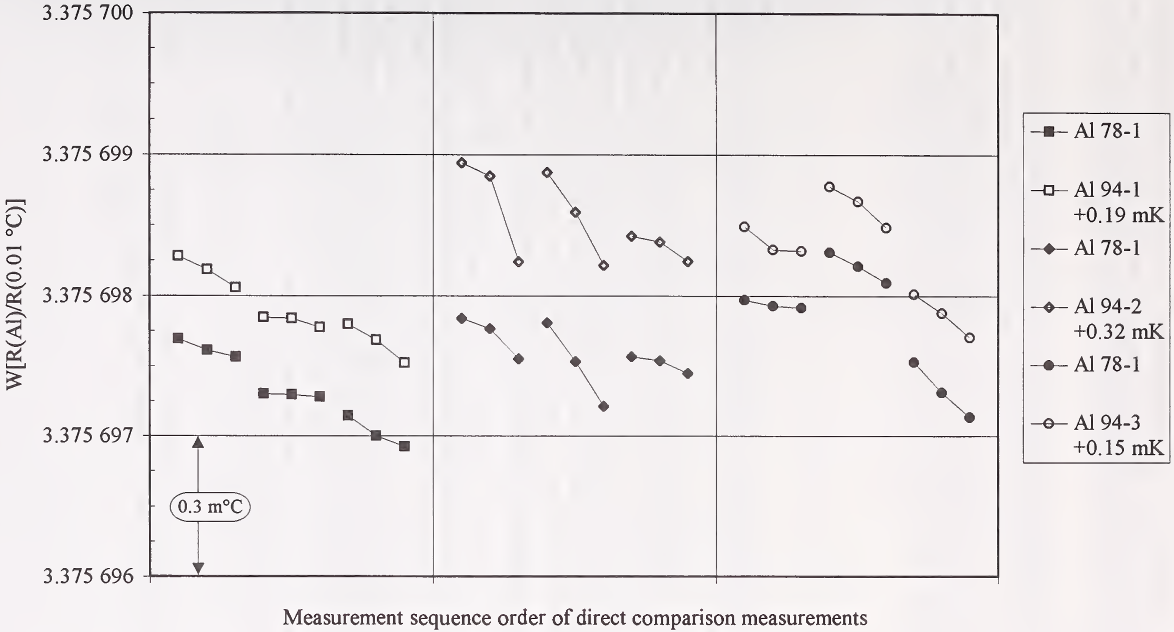

Figure 8. Direct freezing plateau comparison results of Al 94-1, Al 94-2 and Al 94-3 with Al 78-1 (laboratory standard). The differences shown in the legend represent the average temperature difference from the first readings of each direct comparison. The set of matching symbols (open and closed) are for the direct comparison measurements of Al 94-X (where X is 1, 2 or 3) compared with Al 78-1. Each set of symbols (open or closed) connected by lines are for the direct comparison measurements made during the simultaneous freezes.

be made for the difference in pressure [1]. The immersion depth for the three cells was 18 cm from the sensor mid-point of the SPRT to the top of the liquid metal surface (distance from the bottom of the graphite re-entrant well to the top of the liquid level is 20.5 cm). The pressure in the cells during use was 101 325 Pa ±27 Pa.

For those constructing their own aluminum freezing-point cell containing SRM 1744, it is necessary to confirm that the purity of the metal was maintained during construction of that cell. This confirmation is made by comparing the freezing and melting curves of the new cell with those shown in figures 2-7. As a continuing check on the overall purity of the aluminum metal contained in the fixed-point cell, melting and freezing curves should be obtained every 6 months and compared with those obtained previously.

4. Conclusions

The evaluation of SRM 1744 for use as an aluminum freezing-point standard has shown that the material is of high-purity (≥99.9999%) and is acceptable for use as a defining fixed point of the ITS-90 and has been so certified. Based on the results from the evaluation of the three fixed-point cells containing SRM 1744, the average temperature range of melting of the bulk material is not expected to exceed 1.5 m°C. Plateau temperatures of the freezing curves for this material are expected to differ by not more than 0.5 m°C from each other. The results from the evaluation of the three fixed-point cells using freezing and melting curves and direct comparisons of the freezing-point temperature with that of the laboratory standard (Al 78-1) indicates that the plateau temperature of a freezing curve of the SRM 1744 aluminum is expected to differ by not more than 1 m°C from the ITS-90 assigned temperature. A copy of the certificate for SRM 1744 is given in the appendix.

Direct comparisons of the three fixed-point cells (Al 94-1, Al 94-2 and Al 94-3) with the laboratory standard (Al 78-1) have shown that the aluminum of SRM 1744 is of slightly higher purity than any samples of aluminum previously obtained by us. Consequently, fixed-point cell Al 94-2 has been selected to be the new laboratory standard at NIST for the ITS-90 realization of the freezing point of aluminum. Of the three cells, Al 94-2 has the smallest freezing-curve temperature depression, smallest melting-curve temperature range and has the "hottest" freezing-point temperature relative to the old laboratory standard (Al 78-1). The other two fixed-point cells (Al 94-1 and Al 94-3), however, are also acceptable for use as laboratory standards and they will be kept in reserve for that purpose and for control checks of the laboratory standard Al 94-2.

5. References

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6. Appendix A

Certificate for SRM 1744: Aluminum Freezing-Point Standard.

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 $\pm$ 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.

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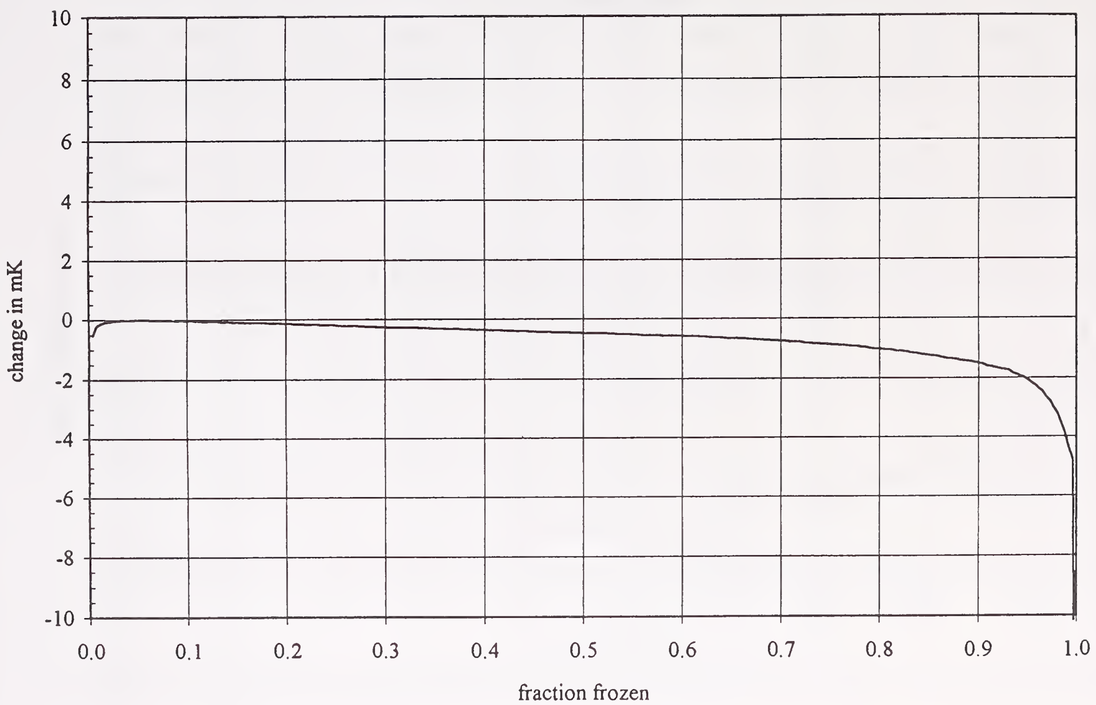

Figure 1. A freezing curve of SRM 1744 aluminum using the "induced-inner-freeze" preparation technique.

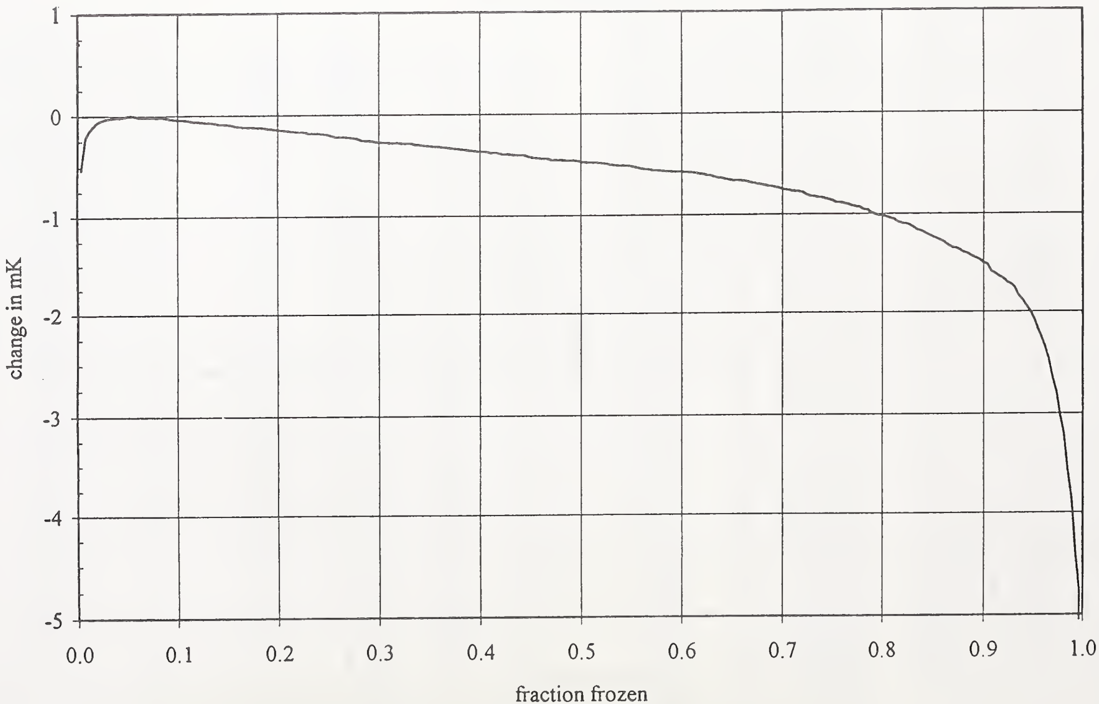

Figure 2. The freezing plateau region of Figure 1 at greater resolution.

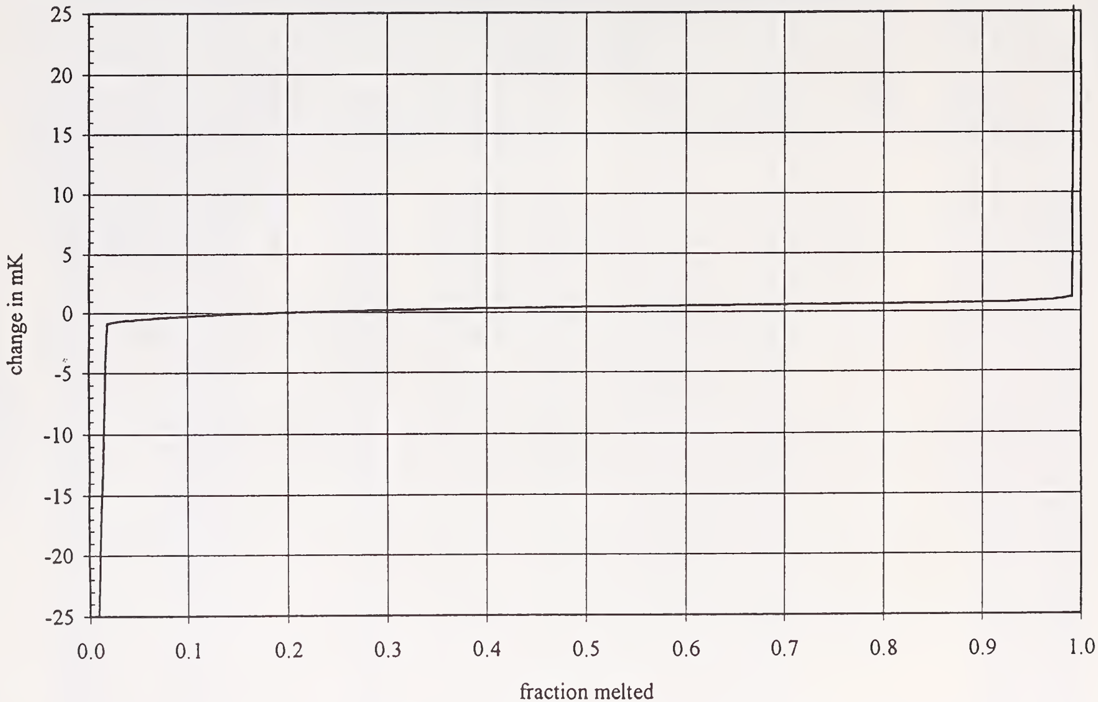

Figure 3. A melting curve of SRM 1744 aluminum following a slow freeze. This melt followed the slow freeze of Figure 1.

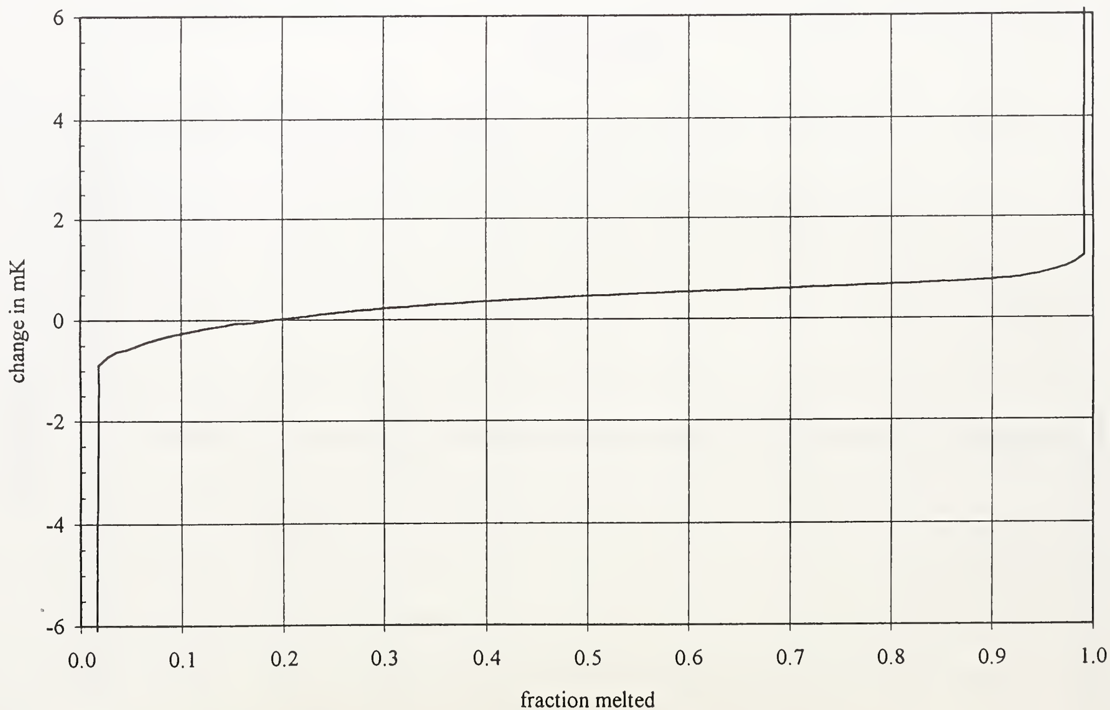

Figure 4. The melting plateau region of Figure 3 at greater resolution.

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