Certificate of Analysis

Standard Reference Material® 741a

Tin Freezing-Point Standard

Certified Freezing-Point Temperature: (231.928 ± 0.000 53) °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 certified value of 231.928 °C ± 0.000 53 °C is the temperature assigned to the fixed point of SRM 741a. The fixed point is realized as the plateau temperature (or liquidus point) of the freezing curve of the slowly frozen high purity tin. SRM 741a consists of 200 g of tin in the form of millimeter size teardrop shot sealed in an argon atmosphere in a plastic bottle.

Based on samples tested, the temperature range of melting of this current lot of material is not expected to exceed 0.0013 °C. Temperatures of freezing curve plateaus (see Figure 2) for samples of this material are expected to differ by not more than ± 0.0002 °C from each other and by not more than ± 0.000 53 °C from the ITS-90 assigned temperature.

An expanded uncertainty ($k=2$) of 0.000 53 °C is assigned to the freezing-point temperature of SRM 741a. The Type A standard uncertainty component of 0.000 15 °C is the standard deviation of $W(t_{90})$ values of repeated measurements of the laboratory standard tin cell with a check Standard Platinum Resistance Thermometer (SPRT) [2]. The Type B standard uncertainty of 0.000 22 °C is obtained from the temperature difference between the three SRM cells and the laboratory standard tin cell as determined from the direct comparison measurements of 0.000 37 °C, and the uncertainty in those direct comparison measurements of 0.000 07 °C. The two Type B standard uncertainties are considered to be a rectangular distribution and must be divided by $\sqrt{3}$.

The tin for this SRM is of high purity, with the total of all elements that affect the freezing-point temperature being 0.3 mg/kg, resulting from 0.1 mg/kg of silver, 0.1 mg/kg of calcium, and 0.1 mg/kg of silicon.

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 instructions given in the Notice and Warnings to Users section of this certificate. The certification is nullified if the SRM is damaged, contaminated, or modified.

Source of Material: The tin metal (Lot M6755) for this SRM was obtained from Johnson Matthey Company of Spokane, Washington.

Temperature measurements of the fixed-point cells were performed by G.F. Strouse of the NIST Process Measurements Division and N.P. Moiseeva of D.I. Mendeleyev Research Institute of Metrology, St. Petersburg, Russia.

The 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
Certificate Issue Date: 2 September 1998

Thomas E. Gills, Chief
Standard Reference Materials Program

Notice and Warnings to Users: Because any handling of high purity material is apt to introduce contamination, this SRM is provided in "shot" form in order to minimize 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 while handling. Also, a clean laboratory environment is essential.

Instructions for Use: In assigning a temperature value to realizations of the tin 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 ($dt/d\ell = 2.2 \times 10^{-3}$ °C/m). Also, if the pressure (p) over the cell during the measurements is not controlled at $1.013\,25 \times 10^{2}$ kPa (1 standard atmosphere), a correction, $dt/dp = 3.3 \times 10^{-8}$ °C/Pa, must be made for the difference in pressure.

Certification Testing: The thermal tests for the certification of this SRM were performed on three fixed-point cells prepared in a manner similar to that described in reference [3]. Each cell contains approximately 1071 g of tin obtained from randomly selected bottles of lot M6755.

The freezing points were prepared using the recommended "induced inner freeze" method. Due to the deep supercool of as much as 25 °C below the freezing-point temperature of high purity tin, the technique for the realization of the tin freezing point is different from that of the other ITS-90 freezing-point metals. The freezing point was achieved by heating the cell overnight to approximately 5 °C above the freezing-point temperature and then setting the furnace to a temperature of about 0.5 °C below the freezing-point temperature of the metal and monitoring the temperature of the metal with the check thermometer. When the tin had cooled to its freezing-point temperature, the freezing-point cell with the check thermometer was removed from the furnace until the start of recalescence. At the beginning of that recalescence, the fixed-point cell was placed back into the furnace, the check thermometer was removed and two fused-silica glass rods were inserted three minutes each into the reentrant well of the cell to induce an inner solid-liquid interface. Finally, the "cold" thermometer was reinserted into the cell and, after equilibrium was obtained, the measurements were started. After equilibrium was established, the temperature of the plateau on the freezing curve was found to vary no more than ± 0.000 16 °C during the first 50 % of the duration of the freeze. Three freezing curves obtained under such conditions are shown in Figure 1 (the region of supercooling and recalescence is not shown, as the curves begin 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 to a temperature of about 1 °C above the freezing-point temperature to slowly melt the metal over an average time of about 10 h. Thermometer readings were recorded continuously until the melting was complete. Three melting curves obtained under such conditions are shown in Figure 3; some of the same data are plotted at greater resolution in Figure 4.

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 maintained by the NIST Platinum Resistance Thermometer Calibration Laboratory, using a 25.5 Ω SPRT. The method of direct comparison is described in detail in reference [5].

During the freezing and melting curve measurements, an inert environment of argon gas at 101 325 Pa ± 27 Pa was maintained in the cells.

The electronic measurement equipment included an ASL F18[1] 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 (25.000 ± 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 $\sqrt{2}$ mA, with a 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.

[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.

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] Furukawa, G.T., Riddle, J.L., Bigge, W., 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)," NIST Tech. Note 1265, 190 pages, (1990).

[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 Cakiroglu, 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**, (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 http://ts.nist.gov/srm.

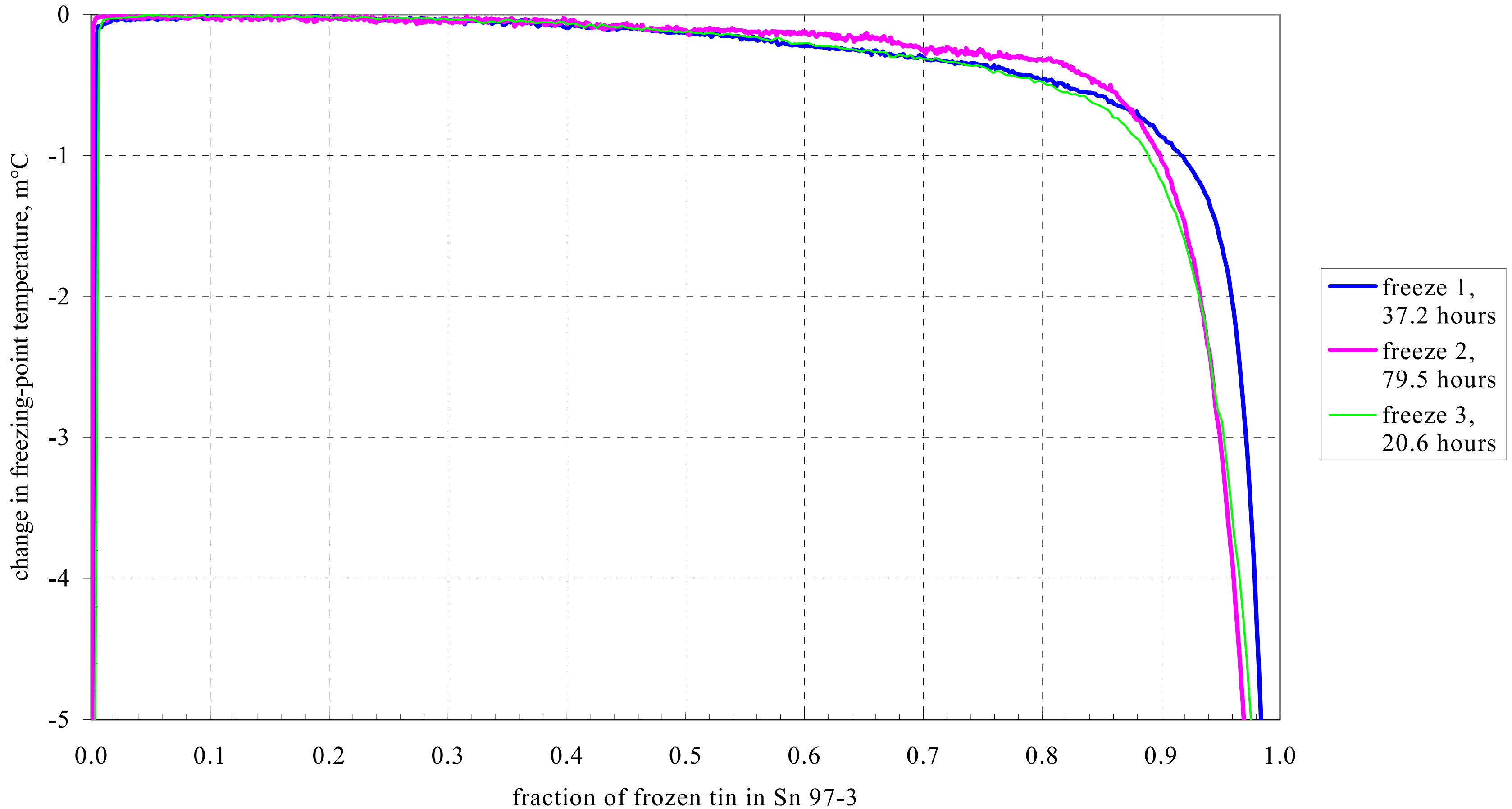

Figure 1. Three freezing curves for SRM 741a using the "induced inner freeze" preparation technique.

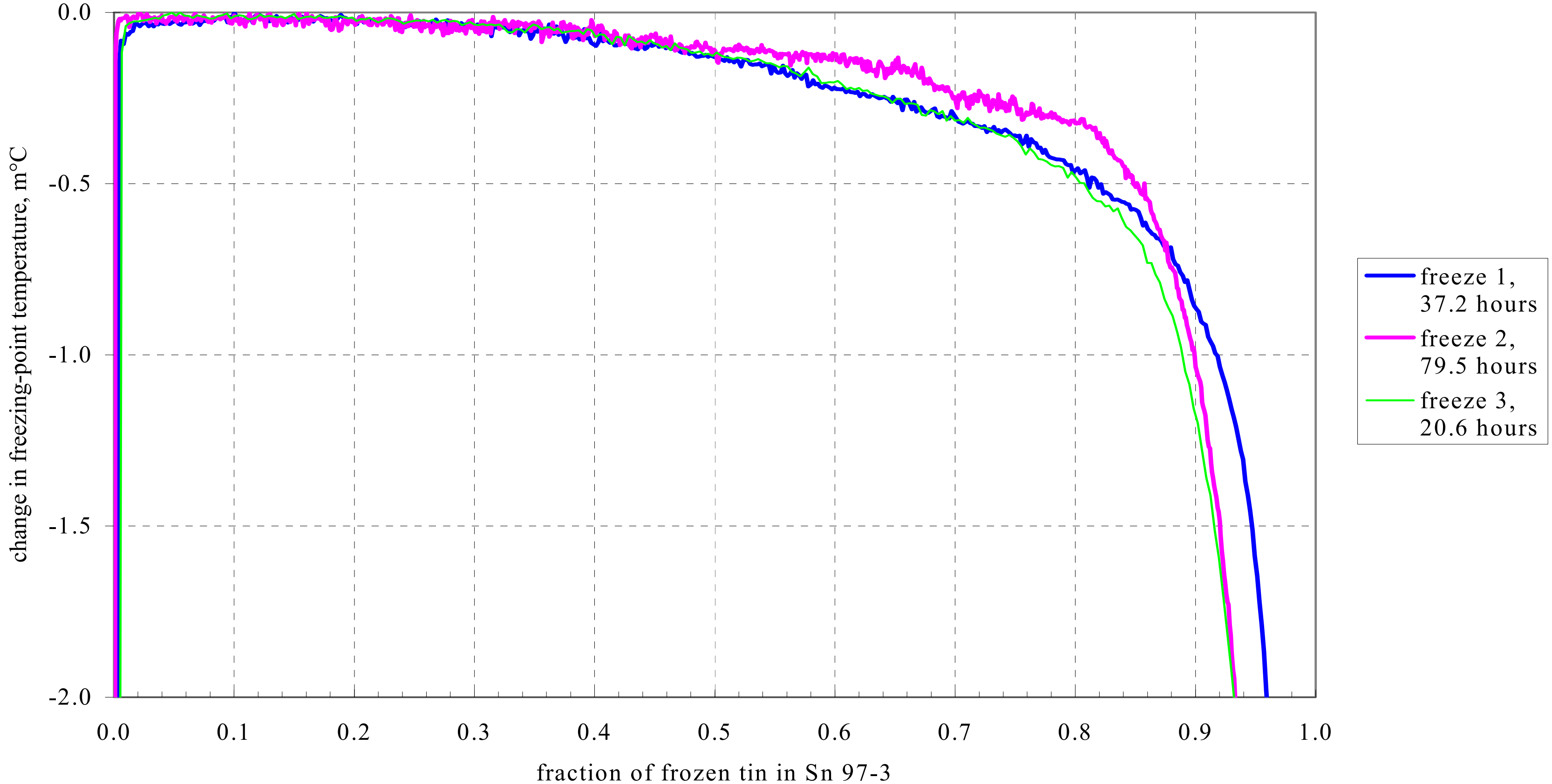

Figure 2. The freezing plateau regions of Figure 1 at greater resolution.

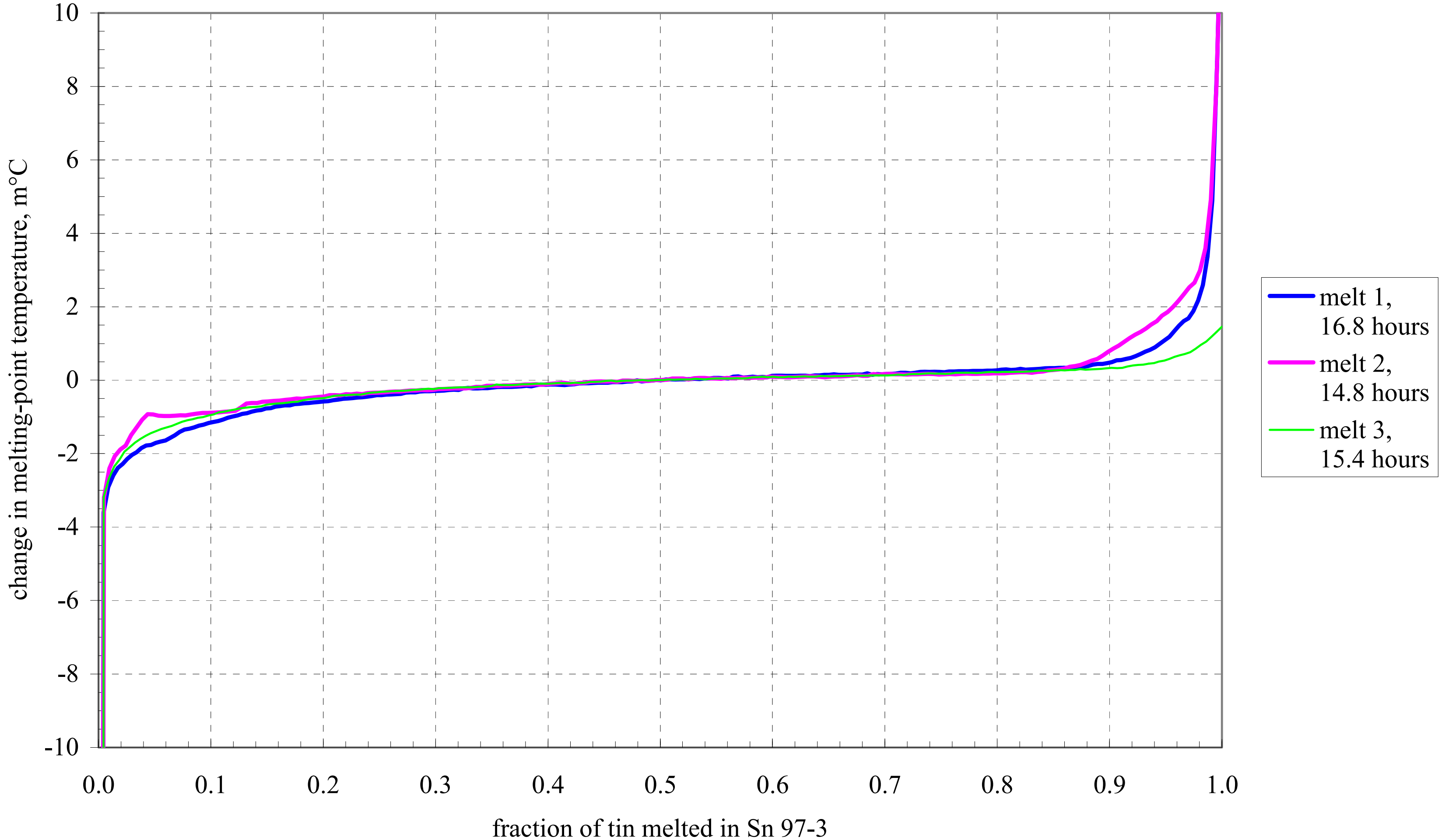

Figure 3. Three melting curves of SRM 741a tin following a slow freeze. Each melt followed the respective slow freeze of Figure 1.

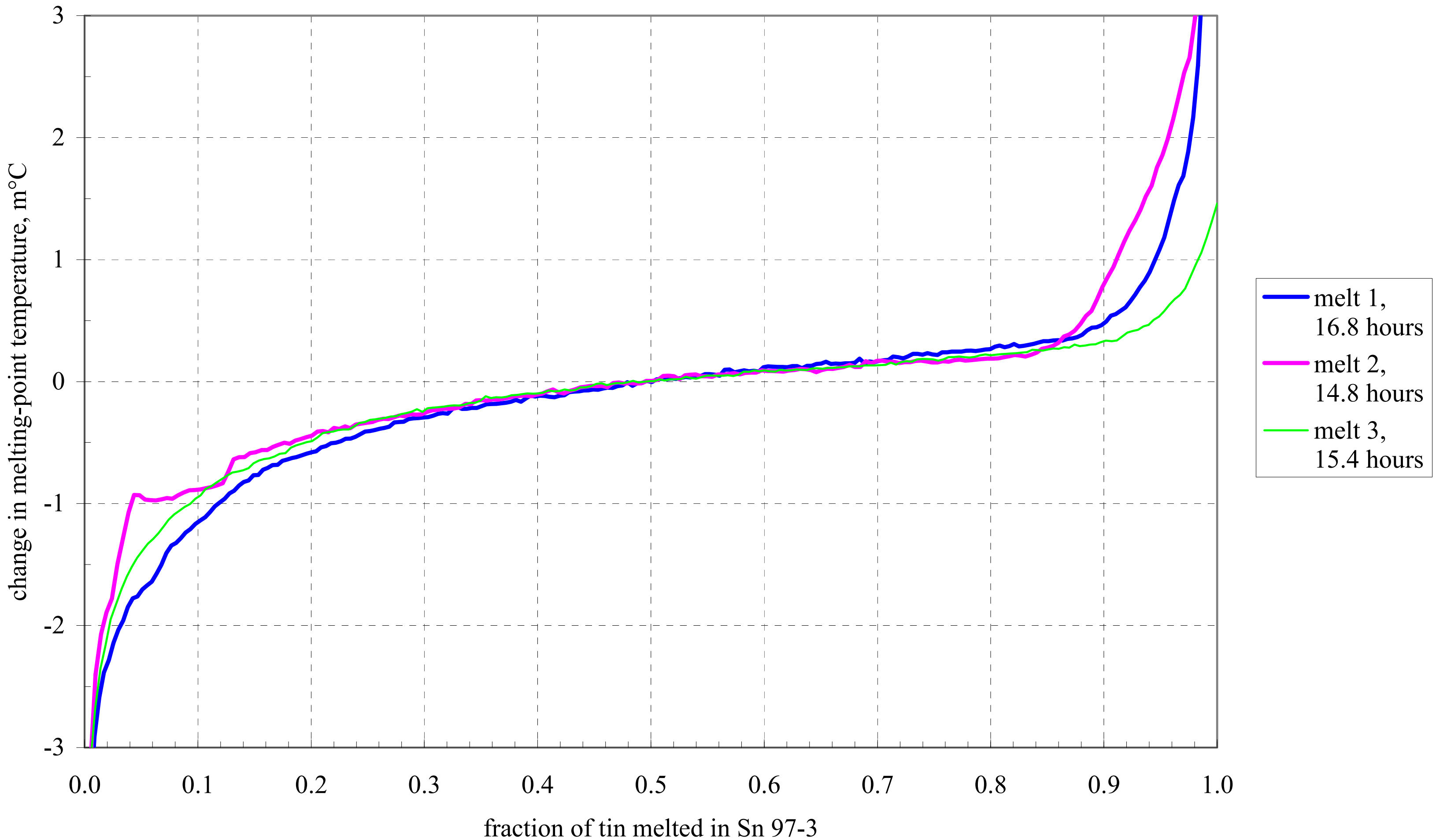

Figure 4. The melting plateau regions of Figure 3 at greater resolution.