

NIST Standard Reference Materials for Use as Thermometric Fixed Points

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Abstract

As part of a program to disseminate the International Temperature Scale of 1990 (ITS-90), NIST provides thermometric fixed-point cells and high-purity metals for constructing fixed-point cells, certified as Standard Reference Materials[®] (SRMs[®]) for the range 29.7646 °C to 961.78 °C. SRM metals of indium, tin, zinc, aluminum and silver, $\geq 99.9999\%$ pure, may be used to construct fixed-point cells that cover the range 156.5985 °C to 961.78 °C. SRMs of large freezing-point cells of tin (231.928 °C) and zinc (419.527 °C) are for use in calibrating standard platinum resistance thermometers in accordance with the ITS-90. SRMs of small fixed-point cells are for use in calibrating small thermometers, e.g., thermistors, over the range 29.7646 °C to 156.5985 °C. These SRMs and their applications will be discussed.

1. Introduction

The National Institute of Standards and Technology (NIST) is responsible for the realization, maintenance and dissemination of the International Temperature Scale of 1990 (ITS-90) for the U.S. industrial and scientific communities. From 0.01 °C to 1085 °C, the ITS-90 has eight defining fixed points of specified metals.⁽¹⁾ NIST dissemination of the ITS-90 is provided through the calibration of (high-temperature) standard platinum resistance thermometers [(HT)SPRTs], the measurement assurance program, publications, bi-annual workshops and Standard Reference Materials[®] (SRMs[®]).

The SRM program sells certified reference materials for use as primary standards. Both fixed-point cells and high-purity metals used to fabricate fixed-point cells are certified at NIST as SRMs. Together, they cover the temperature range from 29 °C to 962 °C. Large SRM fixed-point cells of tin (231.928 °C) and zinc (419.527 °C) are for use in calibrating (HT)SPRTs in accordance with the ITS-90. Small SRM fixed-point cells are used to calibrate small thermometers over the range from 29 °C to 157 °C. High-purity SRM metals ($\geq 99.9999\%$ pure) are available for the construction of fixed-point cells of In, Sn, Zn, Al and Ag that cover the range from 156 °C to 962 °C.

These SRMs described here, facilitate the dissemination of the ITS-90 by NIST and reduce the uncertainty of the in-house calibration of thermometers. The certification process of the fixed-point cells and metals, application of the fixed-point cells, and the uncertainties of SRMs are described. The uncertainties of the user must be added to the uncertainties given here.

2. SRM freezing-point metals

The five metals that define the ITS-90 from 156 °C to 962 °C were certified as freezing-point standard SRMs. For each of the SRMs, a single lot of 20 kg to 24 kg of high-purity metal ($\geq 99.9999\%$ pure) was purchased. The metals were either in the form of 1300 g semi-cylindrical rods (Sn), small 0.1 g teardrop shot (Zn and Ag), 0.3 g buttons (Al) or 10 g ingots (In). The metals were packaged in protective containers that were either filled with argon or vacuum sealed. Depending on the SRM, the unit sizes available for purchase are either 200 g, 300 g or 1300 g (Sn). The SRM 741 Sn freezing-point standard is currently in the process of being replaced by a higher-purity, teardrop “shot” sample to be designated SRM 741a. Table 1 shows the SRM number assigned to each of the metal, freezing-point temperatures, the unit size, the purity of the sample, and the expanded uncertainty ($k=2$) associated with the freezing-point standard. These SRMs were developed for the fabrication of freezing-point cells of the ITS-90 defining fixed points and for their use in the calibration of (HT)SPRTs and other thermometers requiring high-accuracy calibrations.

2.1 Certification Procedure

For certification, three fixed-point cells, each containing random samples of the given SRM metal, were fabricated; three freezing and three melting curves of each cell were obtained and evaluated, and a direct comparison of each cell with the reference standard freezing-point cell of the NIST Platinum Resistance Thermometry (PRT) Laboratory was obtained. The results obtained were used to confirm the purity specified by the supplier and to confirm the freezing-point temperature of the SRM relative to the metal ($\geq 99.9999\%$ pure) in the reference freezing-point cell.

The fixed-point cells used in evaluation of the SRM metals were fabricated in the PRT Laboratory using high-purity cell components and following procedures to minimize contamination. As shown in figure 1, the SRM metal (In, Sn or Zn) was placed into a high-purity ($\geq 99.9999\%$ pure) graphite cell, comprised of a crucible, re-entrant well and cap. The usable volume space in the crucible for the

Table 1. SRM freezing-point metals.

SRM number	metal	t, °C	unit size, g	purity, %	expanded uncertainty (k=2), m°C
1745	In	156.5985	200	99.999 99	0.4
741 [†]	Sn	231.928	1300	99.999 9	1.0
741a [‡]	Sn	231.928	200	99.999 97	
740a	Zn	419.527	200	99.999 94 ₇	0.7
1744	Al	660.323	200	99.999 96	0.7
1746	Ag	961.78	300	99.999 97 ₄	1.1

[†]SRM 741 (Sn freezing-point standard) currently being replaced with SRM 741a.

[‡]Work on SRM 741a is currently in progress and the expanded uncertainty has yet to be determined.

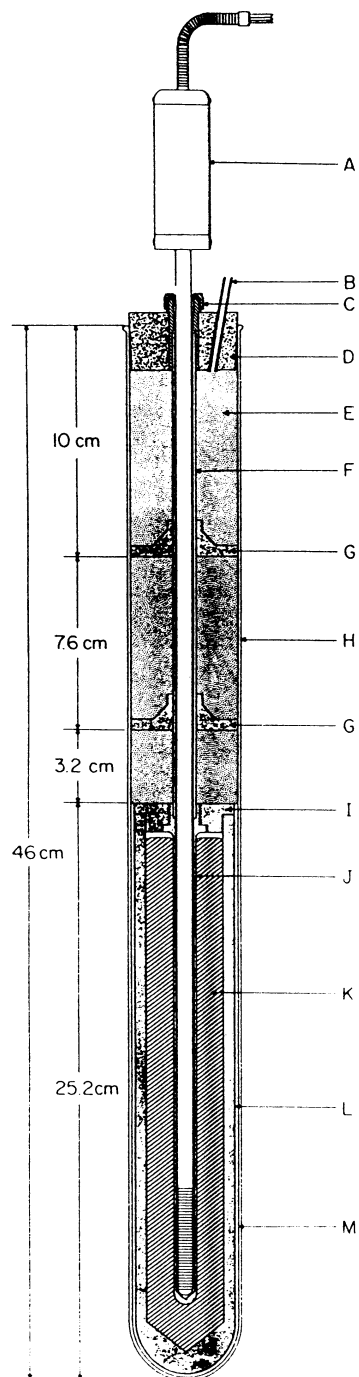


Figure 1. Cross-sectional drawing schematic of a freezing-point cell for In, Sn and Zn showing: (A) a 25.5 Ω SPRT; (B) fill tube to inert gas (Ar or He) supply and pressure gauge; (C) thermometer gas seal (a modified Swagelok fitting with a silicone rubber O-ring); (D) silicone rubber stopper; (E) thermal insulation (1.2 cm thick washed Fiberfrax disks); (F) matte-finished borosilicate-glass guide tube; (G) two graphite heat shunts; (H) precision-bore borosilicate-glass envelope; (I) graphite cap;

(J) graphite re-entrant well; (K) metal sample; (L) graphite crucible; (M) thermal insulation between the borosilicate-glass envelope and the graphite crucible.

metal, allowing for a 1 cm head space between the liquid metal and the underside of the cap, was 149 cm³. The graphite cell containing the metal sample was placed into a precision-bore borosilicate-glass envelope (5 cm o.d. and 46.2 cm length.). Above the graphite cell, there was a matte-finished borosilicate-glass guide tube, disks of washed-ceramic fiber, and two graphite heat shunts. The heat shunts were placed about 3.2 cm and 10.8 cm above the top of the graphite cap. A silicone rubber stopper glued into place at the top of the borosilicate-glass envelope had a modified compression fitting for inserting and sealing the (HT)SPRT in the fixed-point cell.

For the Al and Ag cells shown in figure 2, a slightly larger diameter graphite re-entrant well was used to accommodate an inner silica glass re-entrant well. The usable volume space in the crucible for the metal, allowing for a 1 cm head space between the liquid metal and the underside of the cap, was 135 cm³. The graphite cell containing either the Al or Ag metal was placed into a silica-glass envelope with a silica glass re-entrant well. Extending from the top of the silica-glass envelope to above the cell assembly was a silica-glass pumping tube for evacuation and then for filling the cell with purified argon. The silica-glass assembly was inserted into an Inconel protecting tube (5 cm o.d. and 61 cm length). Above the silica-glass envelope, there were 12 radiation shields separated by 1 cm long silica-glass spacers. The space remaining above the top radiation shield was filled with disks of washed ceramic fiber. A more detailed description of the filling procedures and fixed-point cell assemblies are found in Refs. (2) and (3).

The measurements of the (HT)SPRT in the freezing-point cells were made with a Guildline 9975* for the SRM Zn metal and with an ASL F18 for the In, Sn, Al and Ag metals. A description of the measurement systems may be found in Ref. (4).

The realization of the freezing point used the recommended “induced inner freeze” method.^(5,6) The minimum length of a slow freeze was 10 h. Using Raoult’s Law of dilute solutions, analysis of the freezing curves (temperature depression over the first 50% of the curve) allows an estimate to be made of the impurity concentration in the metal sample of a cell.⁽⁷⁾ This experimentally-derived estimate of impurity concentration was compared with the impurity concentration given in the metal assay provided by the supplier of the high-purity metal to confirm the overall purity of the metal sample.

After each slow freeze, a melting curve lasting at least 8 h was obtained. While the temperature range of melting curve is not simply indicative of the purity of the metal⁽⁸⁾, the liquidus-point temperatures obtained from a slow freeze and from a melt following a fast freeze should not differ by more than 0.2 m°C for a high-purity sample.⁽⁹⁾

The intercomparisons of the freezing-point temperatures of the test cells relative to that of the reference cell were obtained by realizing simultaneous freezes for the two cells in two separate, but nearly identical, furnaces and making three sets of alternate measurements, at equal time intervals, on their freezing-curve plateaus, using an (HT)SPRT. This procedure was performed three times. The fraction of the metal frozen during a set of measurements of the freezing-point temperature of each cell did not exceed 20%.

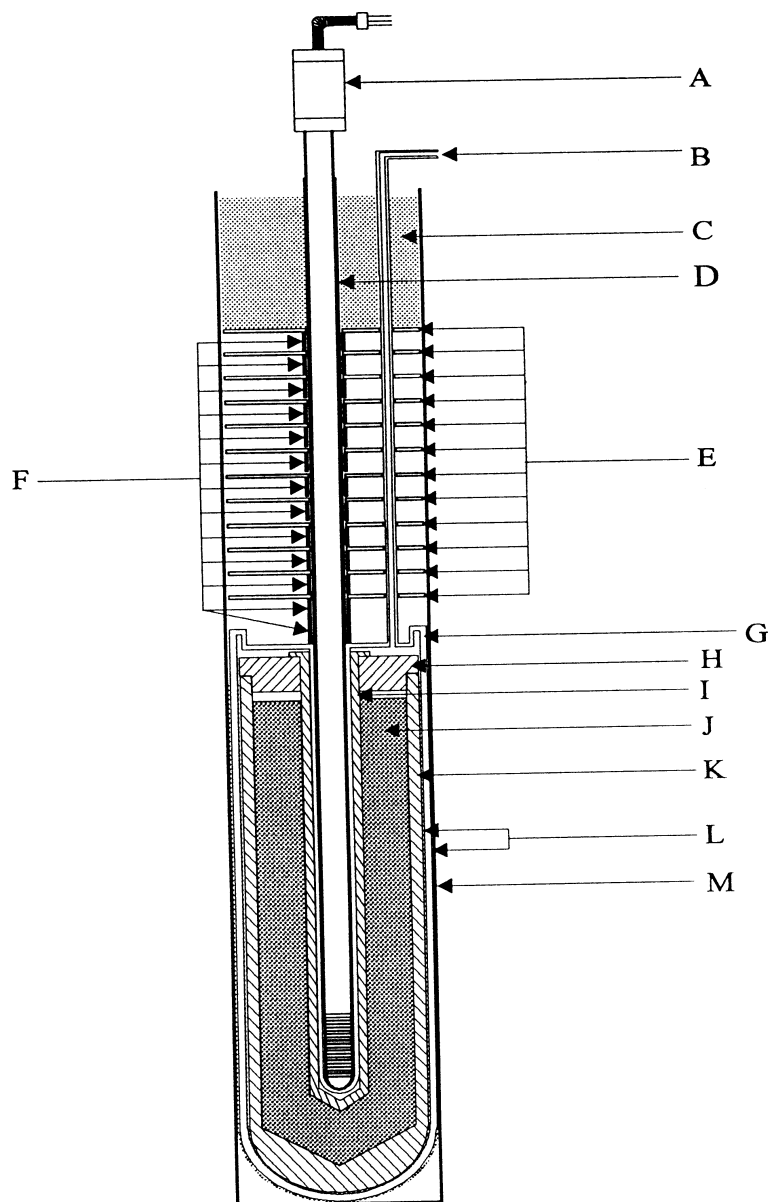


Figure 2. A cross-sectional drawing (not to scale) of a fixed-point cell for Al or Ag showing: (A) a (HT)SPRT; (B) a matte-finished silica-glass pumping tube; (C) thermal insulation (washed Fiberfrax); (D) a matte-finished, silica-glass thermometer guide tube; (E) twelve Inconel radiation shields; (F) 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.

For the metal to be certified as an SRM, the fixed-point cells fabricated using the metal samples should have freezing-point temperatures that are in agreement with the reference cell to within the uncertainties of measurement. If the new SRM has a slightly higher purity (e.g. SRM 1745, SRM 1744, SRM 1746) than that of the metal in the reference cell, then it may be of even higher quality as indicated by having a flatter freezing curve and by being “hotter” relative to the reference cell. A “hotter” cell usually indicates fewer impurities, since impurities in the metal samples will usually decrease the realized freezing-point temperature.

The expanded uncertainty ($k=2$)⁽¹⁰⁻¹²⁾ assigned to the freezing-point temperatures of each of the SRMs, based on the results of the measurements, is given in Table 1. The Type A standard uncertainty was calculated from the standard deviation of $W(t_{90})$ values of repeated measurements of the reference cell with a check SPRT. When the cells of the SRM metals were “colder” than the reference cell (indicating that the SRM sample contained more impurities than the metal sample in the reference cell), the Type B standard uncertainty was obtained from the estimated uncertainty in the freezing-point temperature of the laboratory standard. This uncertainty was calculated from the known impurities listed in the SRM metal assay, the temperature difference between the cell containing the SRM metal and the reference cell as determined from the intercomparison measurements, and the uncertainty in those intercomparison measurements. When the SRM cells were “hotter” than the reference cell (indicating that the SRM sample contained less impurities than the metal sample in the reference cell), the Type B standard uncertainty was obtained from the estimated uncertainty in the freezing-point temperature of the laboratory standard as calculated from the known impurities listed in the SRM metal assay.

3. Large SRM fixed-point cells

Two large SRM fixed-point cells contain high purity ($\geq 99.9999\%$ pure) Sn and Zn and were developed for use as ITS-90 defining fixed-point cells to calibrate (HT)SPRTs. They are designated as SRM 1747 (Sn freezing-point cell) and SRM 1748 (Zn freezing-point cell). They, together with the triple point of water cell, are used to calibrate SPRTs from 0 °C to 420 °C, used for part of the calibration of SPRTs from 0 °C to 661 °C, or used for part of the calibration of HTSPRTs from 0 °C to 962 °C.

3.1 Certification Procedure

Five SRM fixed-point cells of each type of metal were fabricated and certified in the manner described in section 2. Four of each of the five cells were selected as SRMs with the fifth cell (Sn 95-1 and Zn 95-4) kept in the PRT Laboratory as an SRM reference cell. Table 2 shows the serial number (s/n) assigned to each SRM freezing-point cell and the expanded uncertainties ($k=2$) assigned to the cells.

The cells of each type contain high-purity metals ($\geq 99.9999\%$ pure) obtained from a single lot. The metals were in the teardrop shot form contained in plastic bottles sealed in an Ar atmosphere. The method of fabrication of the fixed-point cells was identical to that described above in section 2 and in Refs. (2) and (3). The distance from the inside bottom of the graphite re-entrant well to the top

of the liquid level of the cells is 20.5 cm. These fixed-point cells were designed to fit into most commercially-available fixed-point-cell furnaces.

Table 2. Large SRM fixed-point cells.

SRM 1747			SRM 1748		
fixed-point cell, s/n	freezing-point t, °C	expanded uncertainty (k=2), m°C	fixed-point cell, s/n	freezing-point t, °C	expanded uncertainty (k=2), m°C
Sn 95-1	231.928	0.36	Zn 95-1	419.527	1.0 ₁
Sn 95-2	231.928	0.39	Zn 95-2	419.527	1.1 ₂
Sn 95-3	231.928	0.37	Zn 95-3	419.527	0.9 ₈
Sn 95-4	231.928	0.40	Zn 95-4	419.527	0.9 ₄
Sn 95-5	231.928	0.40	Zn 95-5	419.527	1.1 ₄

The Type A standard uncertainty was calculated from the standard deviation of $W(t_{90})$ values of repeated measurements of the reference cell with a check SPRT. Since the SRM cells were “colder” than the reference cell (indicating that the SRM sample contained more impurities than the metal sample in the reference cell), the Type B standard uncertainty was obtained from the estimated uncertainty in the freezing-point temperature of the laboratory standard calculated from the known impurities listed in the metal assay, the temperature difference between the SRM cell and the reference cell as determined from the intercomparison measurements, and the uncertainty in those intercomparison measurements.

Prior to the use of one of these SRM cells in the laboratory of the purchaser of the cell, the cell should be evacuated and backfilled with an inert gas (He or Ar) several times to remove any air present in the cell. During use, the cell should have a slight overpressure of an inert gas (He or Ar) of about 250 Pa above atmospheric pressure to prevent air from entering the cell. In assigning a measured temperature value to the realization of the fixed-point cell, a correction ($dt/dl = 2.2 \times 10^{-3} \text{ }^\circ\text{C/m}$ for Sn and $2.7 \times 10^{-3} \text{ }^\circ\text{C/m}$ for Zn) must be applied for the depth of immersion (l) of the thermometer sensing element below the metal surface⁽¹⁾ Also, if the pressure (p) over the cell during the measurements is not controlled at 101 325 Pa, a correction ($dt/dp = 3.3 \times 10^{-8} \text{ }^\circ\text{C/Pa}$ for Sn and $4.3 \times 10^{-8} \text{ }^\circ\text{C/Pa}$ for Zn) must be made for the difference in pressure.⁽¹⁾

In using a high-quality fixed-point cell, such as one of these SRM cells, a long-stem SPRT should be associated with the cell as a check thermometer. The check thermometer should be used to measure the beginning of each freeze prior to measurement of the test (HT)SPRTs and then at the end of the freeze to ensure that the plateau has progress as expected. As a continuing check on the overall purity of the metal sample contained in the fixed-point cell, a freezing curve should be obtained every 6 months and compared with those obtained previously.

4. Small SRM fixed-point cells

A series of six small, sealed fixed-point cells were developed to cover the temperature range from 29.7646 °C to 156.5985 °C for the purpose of calibrating small thermometers and for use as check points in which to verify the calibration of small thermometers. Initially, materials were chosen that have a freezing-point, melting-point, or a triple-point temperature, closely matching critical temperature values used in clinical, biomedical and pharmaceutical laboratories as reference-temperature check points. Additionally, these cells may be used for the calibration of thermometers (e.g. thermistors, diode thermometers, industrial-grade resistance thermometers) that do not adhere to the definition of the ITS-90. Two of these SRM cells (SRM 1968 and SRM 1971) contain high-purity metals that have defining fixed points on the ITS-90 and the other four are secondary fixed points. Table 3 gives the SRM number, sample material, cell type, fixed-point temperature, and expanded uncertainty ($k=2$) of the fixed-point cell.

4.1 Certification Procedure

The certification of each cell was performed by measuring the appropriate freezing, melting or triple-point temperature with a set of stable bead-in-glass probe-type thermistors that were regularly calibrated against a NIST-calibrated SPRT. An oil bath or a simple furnace was used provide the thermal environment for evaluating the SRM cells. The measurement system for the thermistors included a constant current source, a calibrated 10 k Ω reference resistor and a 6.5-digit digital voltmeter. The uncertainty of the resistance measurements of the thermistors was about 0.25 m°C.⁽¹³⁾

The expanded uncertainties ($k=2$) assigned to each SRM cell were determined from the uncertainty in the calibration of the SPRT, calibration of the thermistor, the irreproducibility in the fixed-point temperature of a given SRM cell and the scatter in the fixed-point temperature measurements for all of the SRM cells of one type. An overview of each small SRM cell is given below.

SRM 1968 (gallium melting-point cell) has a melting-point temperature of 29.7646 °C (a defining fixed point of the ITS-90) and an expanded uncertainty ($k=2$) of 0.7 m°C. As shown in figure 3, each cell containing about 25 g of 99.999 99% pure gallium was placed in a Teflon crucible with a Nylon cap and Nylon re-entrant well (i.d. 3.6 mm). During the first realization of the melting point, the cell was evacuated and then backfilled with purified argon to a pressure of 101.3 kPa and sealed. This cell is for use in realizing the melting point and is best used with an oil bath. A seven hour duration of the melting-point plateau was achieved by setting the oil bath to 0.18 °C above the Ga melting-point temperature. A more detailed description of the cell and realization technique is found in Ref. (14).

Table 3. Small SRM fixed-point cells.

SRM number	sample material	cell type [†]	fixed-point t_f , °C	expanded uncertainty ($k=2$), m°C
1968	gallium	MP	29.7646	0.7
1972	ethylene carbonate	TP	36.3143	1.5
1969	rubidium	TP	39.265	10
1973	n-docosane	TP	43.879	2.5

1970	succinonitrile	TP	58.0642	1.5
1971	indium	FP	156.5985	2

†MP = melting point, FP = freezing point and TP = triple point

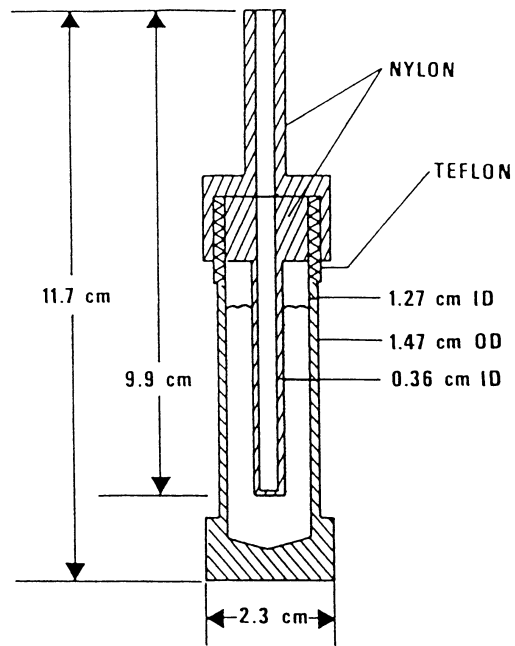


Figure 3. Cross-sectional drawing of SRM 1968, Gallium Melting-Point cell.

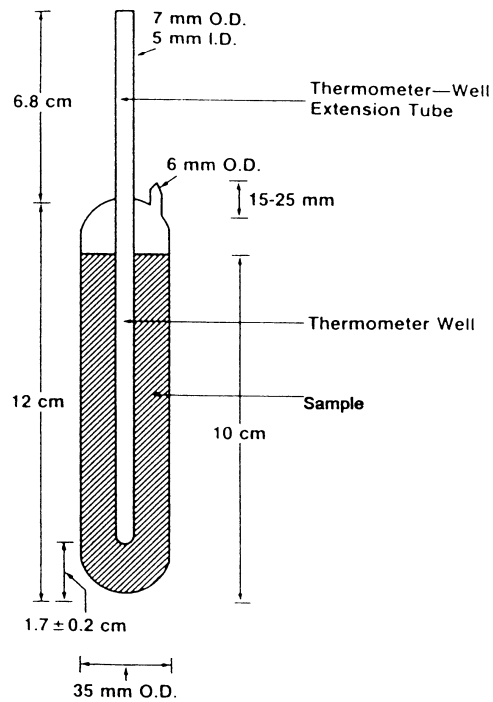


Figure 4. Cross-sectional drawing of SRM cells 1972 (Ethylene Carbonate Triple Point), 1973 (n-Docosane Triple Point), and 1970 (Succinonitrile Triple Point).

SRM 1972 (ethylene carbonate triple-point cell) provides a secondary reference point; it has a triple-point temperature of 36.3143 °C and an expanded uncertainty ($k=2$) of 1.5 m°C. As shown in figure 4, each cell containing about 60 g of 99.999% to 99.9999% pure sample was sealed under vacuum in a borosilicate glass cell with a borosilicate re-entrant well. Thermometers having an o.d. of less than 4.5 mm may be measured in this SRM if the immersion of the sensor is adequate. The triple-point temperature was determined during freezing of the sample in which the “mush” method was used. A more detailed description of the cell and realization technique is found in Ref. (15).

SRM 1969 (rubidium triple-point cell) provides a secondary reference point; it has a triple-point temperature of 39.265 °C and an expanded uncertainty ($k=2$) of 10 m°C. As shown in figure 5, each cell containing about 154 g of 99.9+% pure sample was sealed under vacuum in an all-welded stainless steel cell. This cell can be used to measure a thermometer having an o.d. of less than 5 mm. For improved repeatability, the triple-point temperature was determined from the midpoint of the melting curves obtained during the melting of the sample. With a bath temperature of 39.4 °C, a melting-curve plateau lasted approximately 2 h. A more detailed description of the cell and realization technique is found in Refs. (16) and (17).

SRM 1973 (n-docosane triple-point cell) provides a secondary reference point; it has a triple-point temperature of 43.879 °C and an expanded uncertainty ($k=2$) of 2.5 m°C. As shown in figure 4, each cell containing of approximately 60 g of 99.999% to 99.9999% pure sample was sealed under vacuum in a borosilicate glass cell with a borosilicate re-entrant well. Thermometers having an o.d. of less than 4.5 mm may be measured in this SRM if the immersion of the sensor is adequate. The triple-point temperature was determined during freezing of the sample in which the “induced inner freeze” method was used. With the bath temperature set at 43.5 °C, the freezing curve had a duration of about 9 h. A more detailed description of the cell and realization technique is found in Ref. (18).

SRM 1970 (succinonitrile triple-point cell) provides a secondary reference point; it has a triple-point temperature of 58.0642 °C and an expanded uncertainty ($k=2$) of 1.5 m°C. As shown in figure 4, each cell containing of approximately 60 g of 99.999% to 99.9999% pure sample was sealed under vacuum in a borosilicate glass cell with a borosilicate re-entrant well. Thermometers having an o.d. of less than 4.5 mm may be measured in this SRM if the immersion of the sensor is adequate. The triple-point temperature was determined during freezing of the sample in which the “induced inner freeze” method was used. With the bath temperature set at 57.48 °C, the freezing curve had a duration of about 3.5 h. A more detailed description of the cell and realization technique is found in Refs. (13), (19) and (20).

SRM 1971 (indium freezing-point cell) has a freezing-point temperature of 156.5985 °C (a defining fixed point of the ITS-90) and an expanded uncertainty ($k=2$) of 2 m°C. As shown in figure 6, each cell containing of approximately 100 g of 99.9999% pure sample was placed in an all Teflon cell. During the first realization of the freezing point, the cell was evacuated and then backfilled with purified argon to a pressure of 101.3 kPa and sealed. Thermometers having an o.d. of less than 4.4 mm may be measured in this SRM if the immersion of the sensor is adequate. The freezing-point curve was obtained by using the “induced inner freeze” method. With the bath temperature set at 155.8 °C, the freezing curve had a duration of about 6 h. A more detailed description of the cell and realization technique is found in Ref. (21).

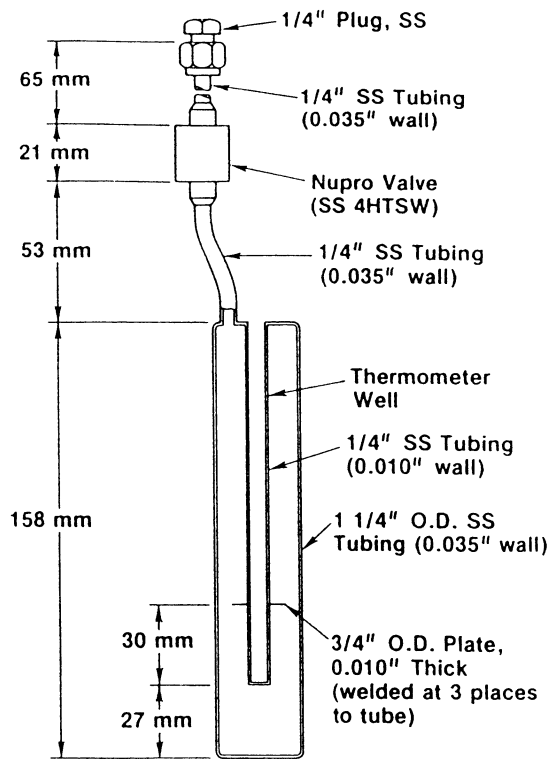


Figure 5. Cross-sectional drawing of SRM 1969, Rubidium Triple-Point cell.

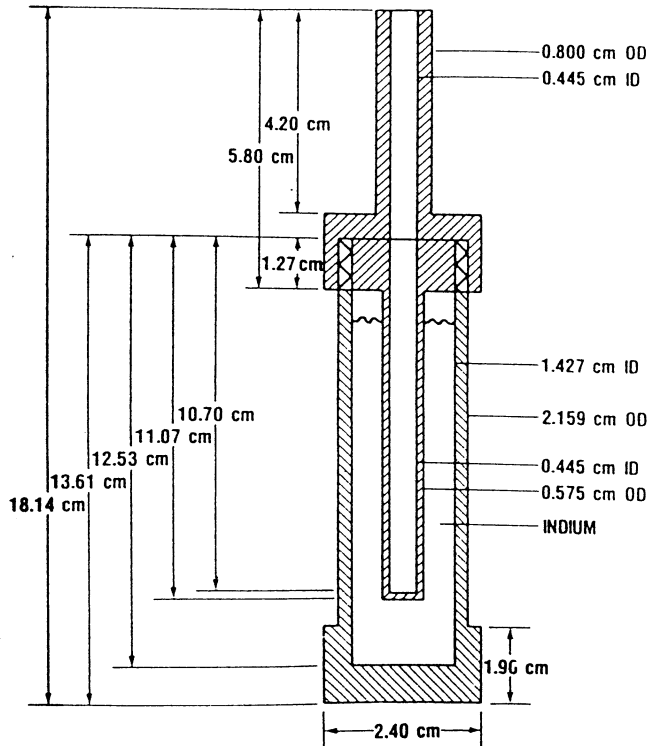


Figure 6. Cross-sectional drawing of SRM 1971, Indium Freezing-Point cell.

5. Conclusions

These NIST-certified SRMs are available to U.S. industry and the scientific community and if they are employed, the users can reduce the uncertainty in their calibration of thermometers and, consequently, in their use. The certification processes for the SRM metals and fixed-point cells have shown that the SRMs are of high quality and are suitable for use in realizing ITS-90 defining fixed points and secondary reference points. SRM 741 (Sn freezing-point standard) is in the process of being replaced by a higher-purity, teardrop “shot” sample to be designated SRM 741a. The large SRM freezing-point cells have small uncertainties and are suitable for calibrating (HT)SPRTs in accordance with the ITS-90. The small fixed-point cells provide a means to both calibrate small thermometers and check of the calibration of small thermometers.

Acknowledgment

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6. References

*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.

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