# SIM.T-K9.1: Canada/Peru bilateral comparison of standard platinum resistance thermometers from 0.01 °C to 419.527 °C

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**Abstract.** A bilateral comparison of standard platinum resistance thermometers is reported that links the INDECOPI (Peru) realization of the International Temperature Scale of 1990 to the NRC realization for the temperature range 0.01 °C to 419.527 °C. The values can be linked to CCT-K9 when that comparison is completed.

## 1. Introduction

The Consultative Committee for Thermometry organized Key Comparison 9 (CCT-K9) as an updated replacement for CCT-K3. At the time of writing, CCT-K9 remains a work in progress. Nonetheless, it was decided to undertake a comparison of the fixed-point temperature standards of Peru with those of Canada following a protocol that closely resembles CCT-K9. The comparison was limited to the fixed points of tin and zinc. NRC was the pilot laboratory for SIM.T-K9.1 and the eventual link to CCT-K9. The comparison protocol requires the participants to select, characterize, and transport (i.e. hand carry) the SPRTs to the pilot. Following the measurements at NRC, the SPRTs were returned and re-measured at INDECOPI to close the loop and assess any changes that occurred in the SPRTs over the course of the measurements.

## 2. Experimental details

### *2.1 Measurements performed at INDECOPI (Peru)*

Two metal-sheathed, 25 ohm, standard platinum resistance thermometers (Hart Scientific Model 5699, S/N 0220 and S/N 0493) were chosen for the comparison. These SPRTs were chosen because acceptable results were obtained with them during an informal fixed point comparison exercise carried out with CEM, Spain in February 2012. Additionally, these SPRTs have shown acceptable stability and drift over several years.

#### *2.1.1 Experimental set up*

INDECOPI´s temperature laboratory has an ISOTECH Model 465 three-zone furnace (S/N 281614-1) specifically designed for ITS-90 fixed-point realizations (200 °C to 1200 °C, Sn to Cu). The furnace is equipped with three temperature controllers, each with a resolution of 0.1 °C. The furnace´s technical specifications indicate that acceptable plateaus can be achieved even with variations in the furnace temperature of up to ± 2 °C. In practice, the furnace temperature variations encountered at INDECOPI ranged from ± 0.20 °C at 200 °C to ± 0.35 °C at 962 °C with the controller settings optimized. The cell holder has a depth of 610 mm and a diameter of 100 mm. At the top of the furnace, there are insulating ceramic blocks and insulating material (Kabul) to control thermal gradients. Likewise, at the bottom there are more insulating ceramic blocks to help minimize the thermal gradient.

The triple point of water cells are maintained in an ice box constructed according to the one described in the BIPM publication *Supplementary Information for the ITS-90*.

INDECOPI´s Temperature Laboratory has controlled environmental conditions. The variations inside the laboratory are within ± 2 °C in ambient temperature and ± 5% in ambient relative humidity. The electrical power is stabilized at 220 VAC ± 2.5%, 60 Hz, and the installation includes peak suppressors and a controlled ground line.

### *2.1.2 Resistance measurements*

The bridge-type measurement system uses a stable DC current source (Guildline PRT adapter model 99301) powered by a UPS (model M-1000P) that stabilizes the electrical signal at 220 VAC ± 1%, with harmonic distortion less than 3%. Two currents are used (1 mA and 1.18 mA) in order to correct for the self-heating of the SPRT. The DC current is measured with a calibrated Fluke Model 8808A 5½-digit multimeter.

The DC current passes through a 25 ohm Fluke Model 742 A-25 standard resistor calibrated by INMETRO, Brazil (certificate DIMCI 2009/2008). The resistor temperature is continuously measured and controlled within a thermostatted environment with maximum variations of ±0.15 °C. Because the temperature coefficient of the resistor is only 2 ppm/°C, these temperature variations contribute less than 0.1 mK uncertainty to the SPRT measurements.

The DC current passing through the standard resistor also passes through the SPRT under calibration. The fundamental measurement is the ratio of the voltage across the SPRT to the voltage across the standard resistor, so systematic errors in the voltages tend to be minimized. This voltage ratio, with minor additional corrections, is equivalent to the resistance ratio between the SPRT and the standard resistor. The SPRT´s resistance is calculated from the measured ratio and the known value of the standard resistor.

The voltage ratio is measured by an Agilent Model 34420 7-½ digit Nanovolt Micro-ohmmeter, calibrated by INTI, Argentina (certificate FM 102-15759).

The linearity of this bridge-type electrical measurement system has been assessed using two sets of calibrated resistors in different combinations (series and parallel) to obtain ratios from 1.000 000 to 10.000 000. The maximum deviation from linearity (4.3 ppm) contributes to the measurement uncertainty.

Measurements are made with both current polarities to take into account DC offsets within the measurement system.

### *2.1.3 Traceability*

The following sealed cells were used:

Zinc Cell: Isotech Model 17671MO, S/N Zn 260
Tin Cell: Isotech Model 17669MO, S/N Sn 216
Water Cell: Isotech Model B11-65-270, S/N 794

These cells were calibrated by NIST with the following Certificates:
836/279319-10 for Sn and Zn Cells
836/278242-09 for Water Triple Point Cell

The resistance values measured at the triple point of water were not corrected for isotopic composition. This influence factor (about 0.04 mK according to the manufacturer's specifications and the NIST certificate) contributes to the measurement uncertainty. The NIST calibration of the cells was used to "confirm" that the combined effect of the purity and gas pressure uncertainty components are consistent with the certificate uncertainty, so a correction was not applied and the certificate uncertainty was used.

### *2.1.4 Measurements*

Table 1 list the resistance ratios (averages of 3 realizations) and final triple point of water resistances for the measurements carried at INDECOPI. The "*after – before*" differences of 0.33 mK (S/N 0493) and -1.36 mK (S/N 0220) at the zinc freezing point and 0.12 mK (S/N 0493) and 0.83 mK (S/N 0220) at the tin freezing point are well within the uncertainty. Likewise, the TPW resistances differ by the equivalent of only 0.3 mK. Overall, these data indicate that the SPRTs have travelled well and therefore there is no compelling evidence that an additional uncertainty term is needed to account for instability of the transfer standards. The SPRT resistances at the TPW were sufficiently stable that annealing of the thermometers was considered unnecessary.

**Table 1.** Resistance ratios determined at INDECOPI before and after the SPRTs were measured at NRC. Each resistance ratio in the table is the average of 3 freezing point realizations. The values in italics are the resistances of the thermometers (in ohms) at the triple point of water (0.010 °C).

| Fixed point | Cell identification | $t_{90}$ / °C | *W*(S/N 0493) | $u_L$ / mK | *W*(S/N 0220) | $u_L$ / mK |
|---|---|---|---|---|---|---|
| *prior to measurements at NRC* | | | | | | |
| Zinc | Zn 260 | 419.527 | 2.5686946 | 2.0 | 2.5686913 | 2.0 |
| Tin | Sn 216 | 231.928 | 1.8926606 | 1.9 | 1.8926690 | 2.0 |
| $H_2O$ (final) | 794 | 0.010 | *25.45692* Ω | | *25.09751* Ω | |
| *after measurements at NRC* | | | | | | |
| Zinc | Zn 260 | 419.527 | 2.5686958 | 2.0 | 2.5686865 | 2.0 |
| Tin | Sn 216 | 231.928 | 1.8926610 | 1.9 | 1.8926721 | 2.0 |
| $H_2O$ (final) | 794 | 0.010 | *25.45695* Ω | | *25.09742* Ω | |

## *2.2 Measurements performed at NRC with the INDECOPI SPRTs*

### *2.2.1 Fixed-point measurements*

The SPRTs arrived at NRC September 17, 2012. Upon delivery, the resistances at the triple point of water (TPW) differed by the equivalent of 3.06 mK (S/N 0493) and -0.55 mK (S/N 0220) from the values measured at INDECOPI. Next, the SPRTs were measured at the Zn freezing point, but both SPRTs exhibited a downward drift in resistance that made it difficult to determine a meaningful plateau value. Subsequent measurements at the TPW revealed that S/N 0493 had decreased by the equivalent of 0.5 mK and S/N 0220 by 1.0 mK. With the aim of stabilizing the resistances, both SPRTs were annealed overnight at 480 °C. The resistances of both SPRTs at the TPW decreased a further 0.3 mK from the annealing. Another attempt at the Zn freezing point showed that S/N 0493 was still drifting downward in resistance while S/N 0220 appeared to be stabilizing. Additional heat treatment of S/N 0493 consisted of 1.5 hours at 660 °C (in the Al fixed-point furnace) followed by 1 hour in the annealing furnace at 480 °C. Following this heat treatment, the resistance at the TPW was 1.8 mK below the value when it was received. From this point on, both SPRTs were adequately stable as revealed by the repeated measurements at the TPW. Further heat treatment was unnecessary.

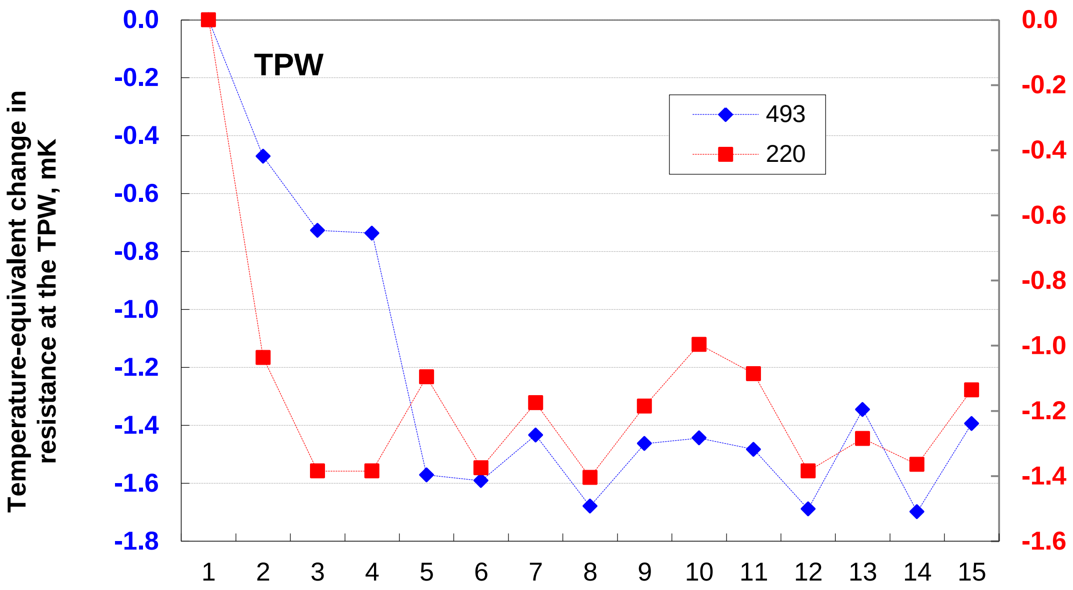


**Figure 1.** The evolution of the electrical resistances of the INDECOPI SPRTs observed at the triple point of water while at NRC.

Over the course of their 2-week stay at NRC, 4 freezing points of Sn and Zn were obtained with SPRT 0493, and 5 freezing points of Sn and Zn with SPRT 0220. The detailed behaviour is best illustrated graphically (see Figures 2 and 3). Relatively stable behaviour was observed at the freezing point of tin for both thermometers, and the agreement of the two is good in light of the uncertainties claimed by INDECOPI ($U(k = 2) \sim 4$ mK).

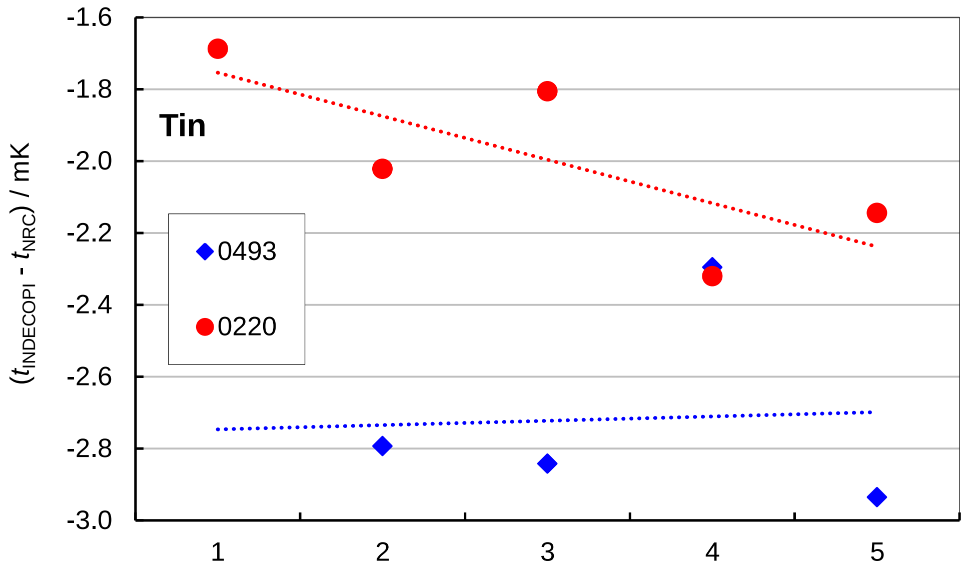


**Figure 2.** Temperature-equivalent differences of the INDECOPI SPRTs observed at NRC at the freezing point of tin. The differences are with respect to the resistance ratios measured at INDECOPI prior to the measurements at NRC.

This is less true for the zinc freezing point, where the two SPRTs are in greater disaccord. Additionally, SPRT 0220 exhibits a modest decrease in resistance ratio over the course of the measurements whereas the trend for SPRT 0493 is primarily increasing, and of greater slope.

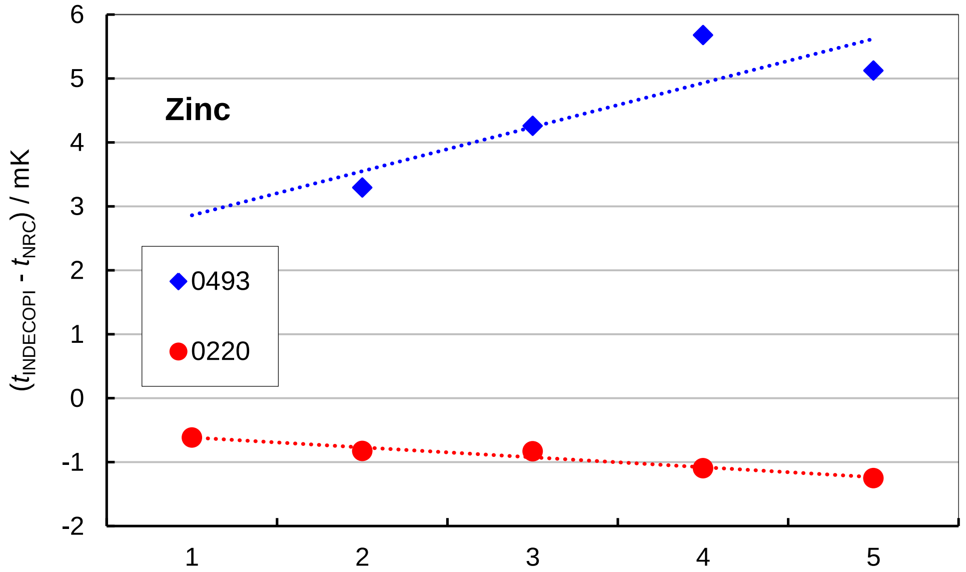


**Figure 3.** Temperature-equivalent differences of the INDECOPI SPRTs observed at NRC at the freezing point of zinc. The differences are with respect to the resistance ratios measured at INDECOPI prior to the measurements at NRC.

In an effort to ensure the quality and comparability of the measurements, the insulation resistance between the sheath and the leads was measured under various operating conditions (i.e. with the SPRTs in the TPW cell, the tin cell, and the zinc cell). With a 100 V test voltage applied, the insulation resistance for S/N 0220 exceeded 110 MΩ under all measurement conditions. This was generally true for SPRT 0493 as well, but on one occasion an insulation resistance of 11 MΩ was measured when the SPRT was left for a prolonged period (> 1 hour) in the Zn fixed point. It is possible that this behaviour was due to the migration of moisture within the sheath. Unfortunately, there was insufficient time for more extensive investigation. Without compelling and repeatable evidence of a moisture problem, we feel that the results for the two thermometers can be averaged.

**Table 2.** Average resistance ratios obtained at NRC. The values in italics are the final resistances of the thermometers (in ohms) at 0.01 °C.

| Fixed point | Cell identification | $t_{90}$ / °C | *W* (S/N 0493) | $u_L$/mK (S/N 0493) | *W* (S/N 0220) | $u_L$/ mK (S/N 0220) |
|---|---|---|---|---|---|---|
| Zinc | Zn-6 | 419.527 | 2.5686786 | 0.60 | 2.5686945 | 0.31 |
| Tin | Sn-4 | 231.928 | 1.8926707 | 0.21 | 1.8926764 | 0.19 |
| $H_2O$ (final) | B11-270 | 0.010 | *25.457058* Ω | | *25.097344* Ω | |

### *2.2.2 Resistance measurements*

The resistance measurements were made with an Automatic Systems Laboratories Model F18 resistance bridge. A 100 Ω Tinsley Model 5685A reference resistor was used, thermostatted at 25 °C ± 2 mK in a Guildline Model 9732VT oil bath. Currents of 1 mA and √2 mA were used to correct for the self-heating of the SPRTs.

### *2.2.3 Traceability*

The measurements were made using open cells fabricated at NRC. Their laboratory designations are Sn-4 and Zn-6. These cells are not the fixed points used during the measurements for CCT-K3, and so we are relying on the values from CCT-K9 to link the measurements made at NRC to those of the other participants. Figures 4 and 5 visually indicate the before-and-after repeatability of the INDECOPI measurements and their compatibility with the NRC fixed points. Detailed analysis of the differences can be found in Section 3.

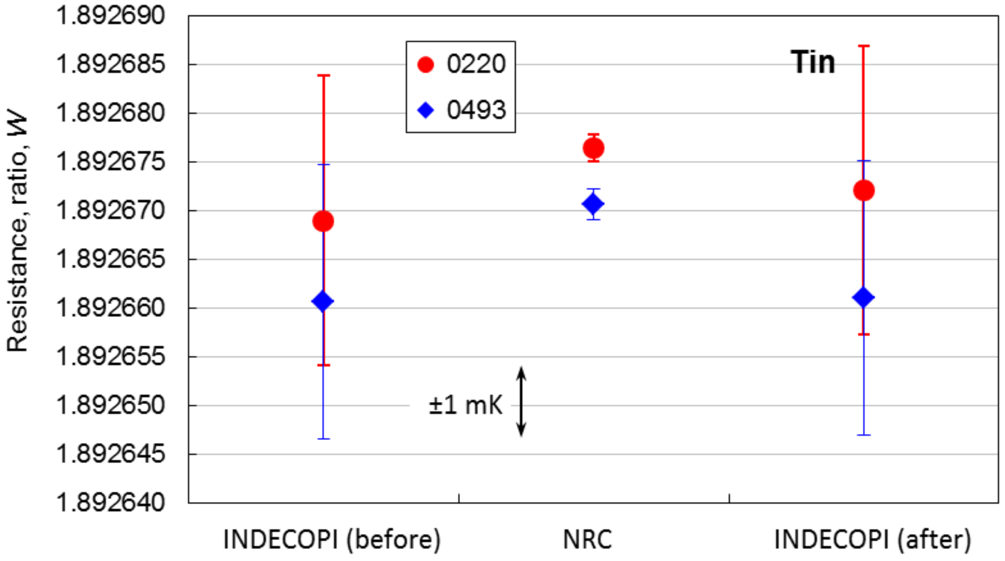


**Figure 4.** Resistance ratios of the INDECOPI SPRTs observed at the freezing point of tin.

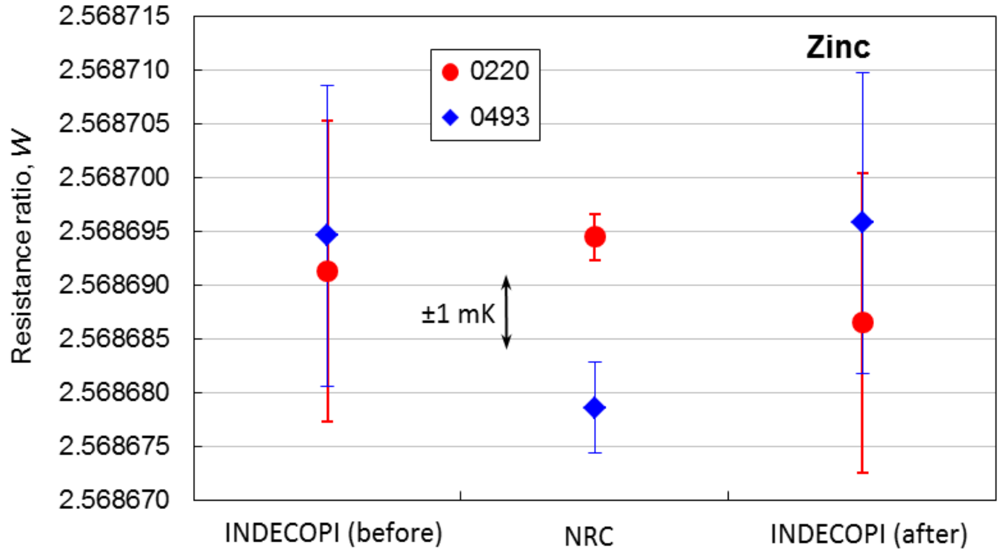


**Figure 5.** Resistance ratios of the INDECOPI SPRTs observed at the freezing point of zinc.

## 3. Results

For this comparison, the averages of the fixed-point resistance ratios measured at NRC constitute the reference values. When the SPRT is well-behaved and "before" and "after" values are in reasonable agreement, the degree of equivalence is calculated from

$$D_{NMI} = ((W_{NMI-before} + W_{NMI-after})/2 - W_{NRC})/(\mathrm{d}W_r/\mathrm{d}T) \tag{1}$$

Both SPRTs are capable of contributing data to the comparison, so the resulting *D*-values can be averaged.

The expanded uncertainty, *U*, is obtained by combining the uncertainties of the terms in Equation 1.

$$U = 2\sqrt{u_{NMI}^2 + u_{NRC}^2} \tag{2}$$

For the purposes of this comparison, the NRC uncertainty in the above equation was obtained by adding in quadrature the Type B uncertainties from Appendix A with the standard deviation of the temperature-equivalent differences obtained at NRC with the INDECOPI thermometers.

**Table 3.** The degrees of equivalence, *D*, and expanded uncertainties, *U* ($k$ = 2), for this comparison.

| $T$ / °C | $D_{\mathrm{INDECOPI}}$ / mK | $U$ / mK |
|---|---|---|
| 419.527 | 1.58 | 4.6 |
| 231.928 | -2.12 | 3.9 |

## 4. Conclusion

This comparison has demonstrated compatibility of the fixed points of the participants within the combined uncertainties, and therefore serves to verify the intended Calibration and Measurement Capabilities envisioned for INDECOPI, Peru. The results can be linked to NMIs globally following CCT-K9, with NRC as the linking participant.

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**Appendix A**

**<u>The uncertainty budget for the measurements at INDECOPI, Peru.</u>**

| | Sn | | | | Zn | | | |
|---|---|---|---|---|---|---|---|---|
| **SPRT** | S/N 0493 | | S/N 0220 | | S/N 0493 | | S/N 0220 | |
| **Type A** | mK | df | mK | df | mK | df | mK | df |
| Phase transition realization repeatability | 0.14 | *179* | 0.23 | *179* | 0.05 | *179* | 0.11 | *179* |
| Bridge (repeatability, non-linearity) | 0.67 | *179* | 0.67 | *179* | 0.71 | *179* | 0.71 | *179* |
| Reference resistor stability | 0.54 | *59* | 0.54 | *59* | 0.57 | *59* | 0.57 | *59* |
| **Total A** | **0.87** | | **0.89** | | **0.91** | | **0.92** | |
| **Type B** | mK | df | mK | df | mK | df | mK | df |
| Chemical impurities | 0.72 | *200* | 0.72 | *200* | 0.39 | *200* | 0.39 | *200* |
| Hydrostatic-head | 0.04 | *200* | 0.04 | *200* | 0.05 | *200* | 0.05 | *200* |
| Propagated TPW | 1.37 | *200* | 1.37 | *200* | 1.47 | *200* | 1.47 | *200* |
| SPRT self-heating | 0.40 | *179* | 0.40 | *179* | 0.57 | *179* | 0.4 | *179* |
| Heat flux | 0.20 | *59* | 0.20 | *59* | 0.30 | *59* | 0.30 | *59* |
| Moisture | 0.10 | *200* | 0.10 | *200* | 0.10 | *200* | 0.10 | *200* |
| SPRT Pt oxidation | 0.28 | *200* | 0.28 | *200* | 0.39 | *200* | 0.39 | *200* |
| Gas pressure | 0.02 | *200* | 0.02 | *200* | 0.02 | *200* | 0.02 | *200* |
| Slope of plateau | 0.50 | *59* | 0.61 | *59* | 0.67 | *59* | 0.66 | *59* |
| **Total B** | **1.71** | | **1.75** | | **1.83** | | **1.78** | |
| Combined standard uncertainty | **1.9** | *630* | **2.0** | *644* | **2.0** | *593* | **2.0** | *558* |
| *k using effective df in t-distribution* | 1.96 | | 1.96 | | 1.96 | | 1.96 | |
| Expanded uncertainty (*k*=2) | **3.8** | | **3.9** | | **4.1** | | **4.0** | |

**<u>Note:</u>** Repeatability in the above table includes the variability from 3 freezes (in addition to a component associated with the variability of 60 readings per freeze).

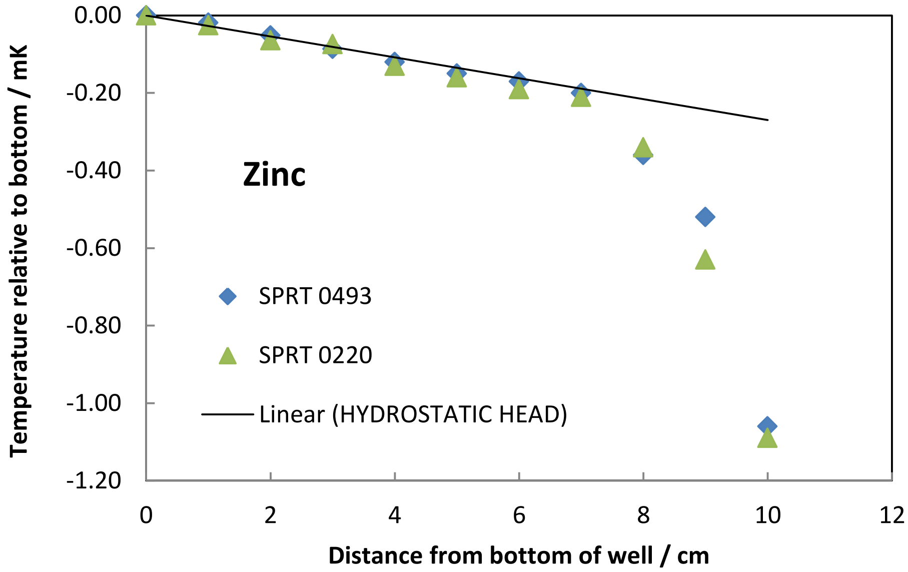
**The uncertainty budget for the NRC fixed points.**

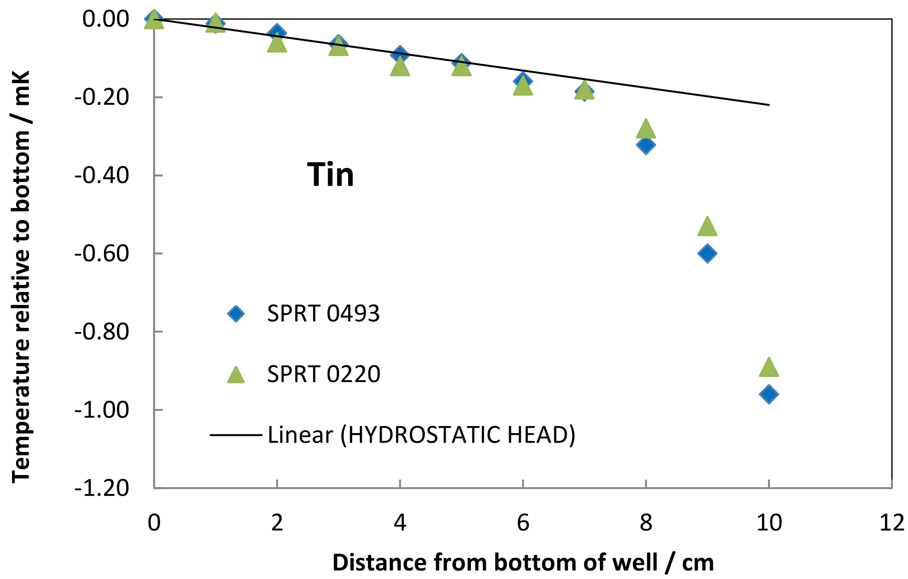
| | Sn-4 | | Zn-6 | |
|---|---|---|---|---|
| **Type A** | mK | *df* | mK | *df* |
| Phase transition realization repeatability (sample standard deviation) | 0.170 | *10* | 0.050 | *10* |
| **Total A** | **0.170** | | **0.050** | |
| **Type B** | mK | *df* | mK | *df* |
| Bridge (repeatability, non-linearity) | 0.030 | *8* | 0.030 | *8* |
| Reference resistor stability | 0.004 | *50* | 0.004 | *50* |
| Chemical impurities | 0.127 | *8* | 0.267 | *8* |
| Hydrostatic-head (10 mm) | 0.022 | *50* | 0.027 | *50* |
| Propagated TPW | 0.081 | *16* | 0.110 | *16* |
| SPRT self-heating | 0.010 | *50* | 0.010 | *50* |
| Heat flux | 0.005 | *8* | 0.005 | *8* |
| Moisture | - | | - | |
| SPRT Pt oxidation | - | | - | |
| Gas pressure | 0.004 | *8* | 0.006 | *8* |
| Slope of plateau | 0.010 | *8* | 0.010 | *8* |
| **Total B** | **0.156** | | **0.293** | |
| Combined standard uncertainty | **0.231** | *24* | **0.297** | *12* |
| Expanded uncertainty ($k$ = 2) | **0.462** | | **0.594** | |

## Appendix B

**Immersion profiles measured at INDECOPI, Peru.**

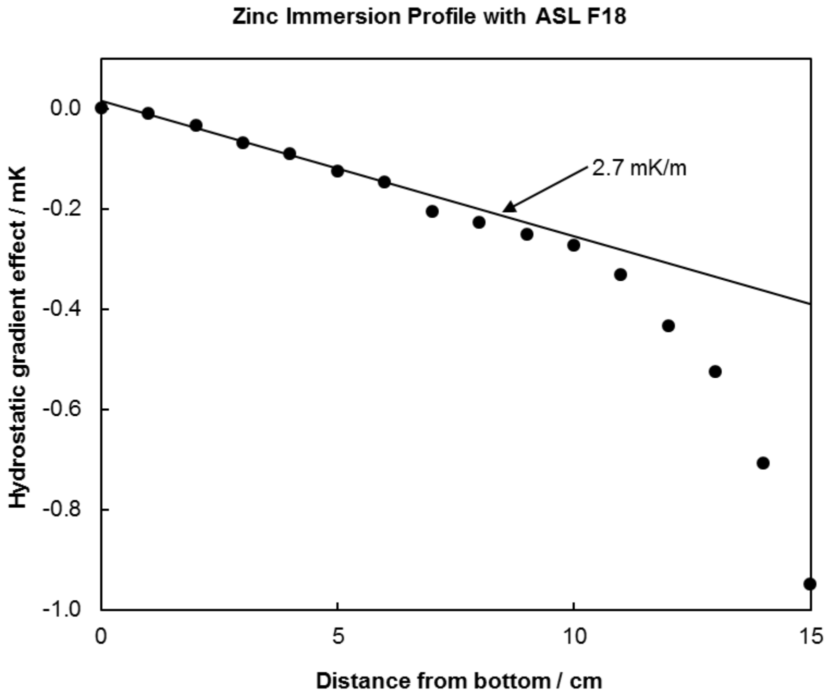
The SPRT resistances that provide the basis for the relative temperature differences plotted below were not corrected for self-heating.

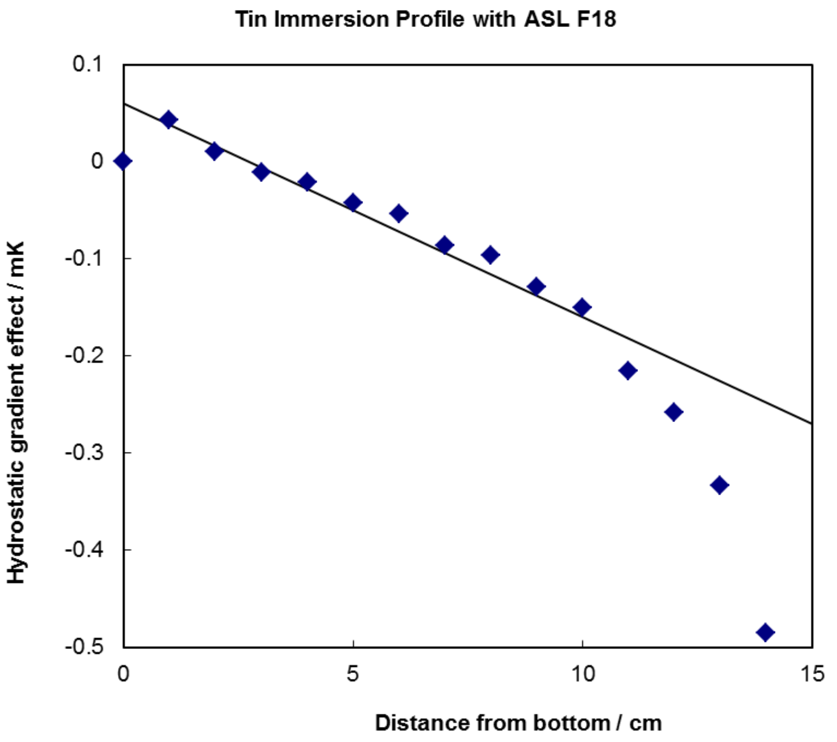


## Immersion profiles measured at NRC

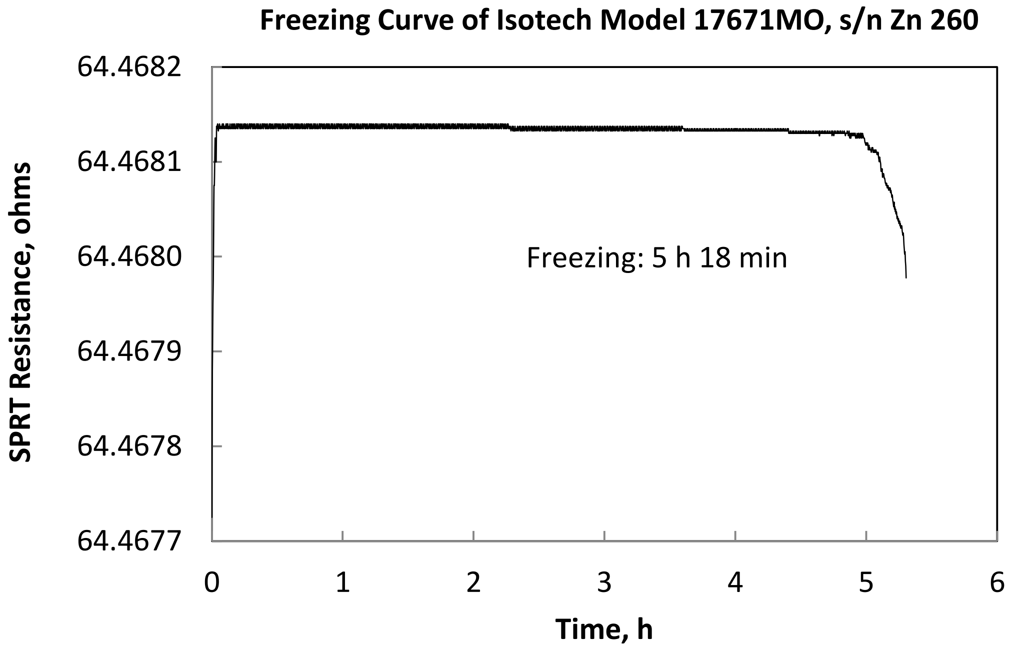
The immersion profiles shown here were recorded during the CCT-K3 comparison and may be found in that report. They are duplicated here for the convenience of the reader.

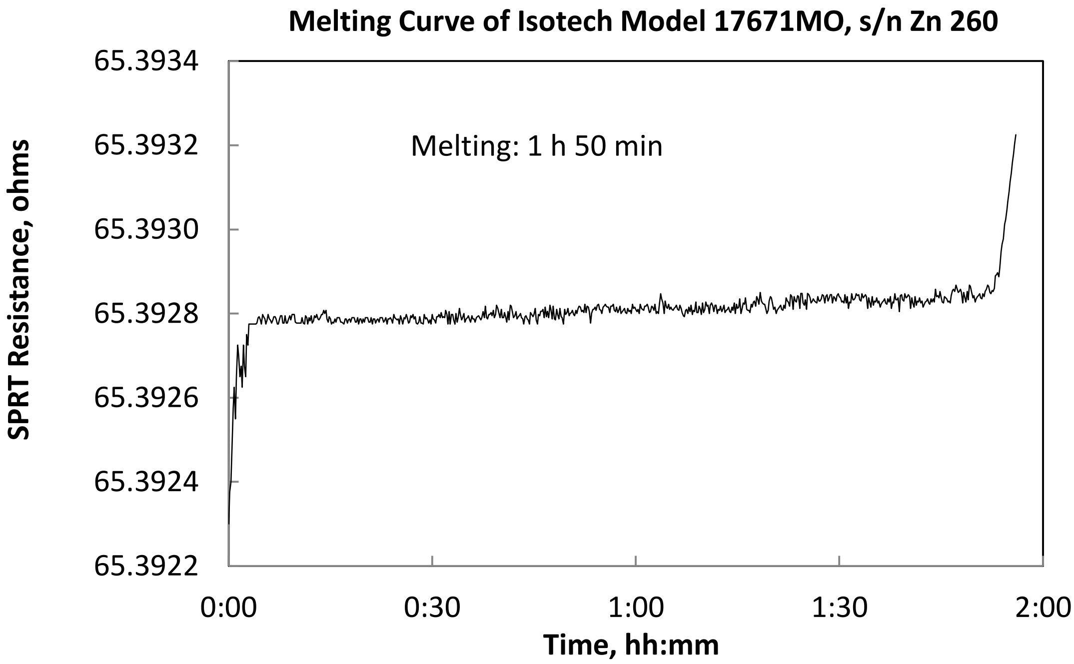


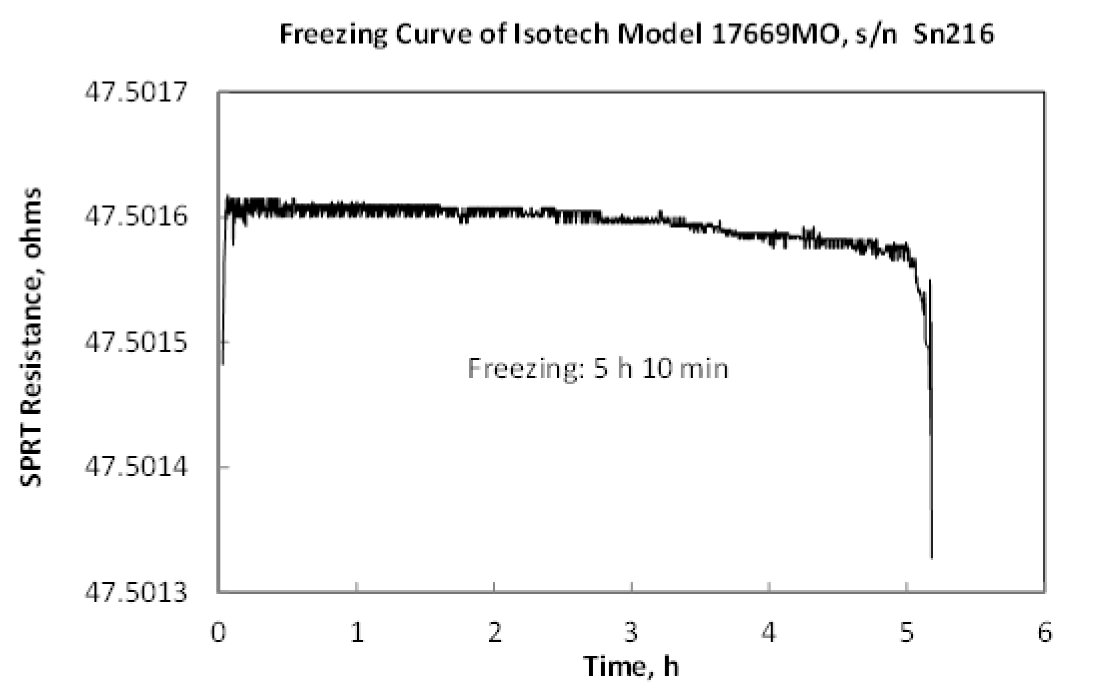



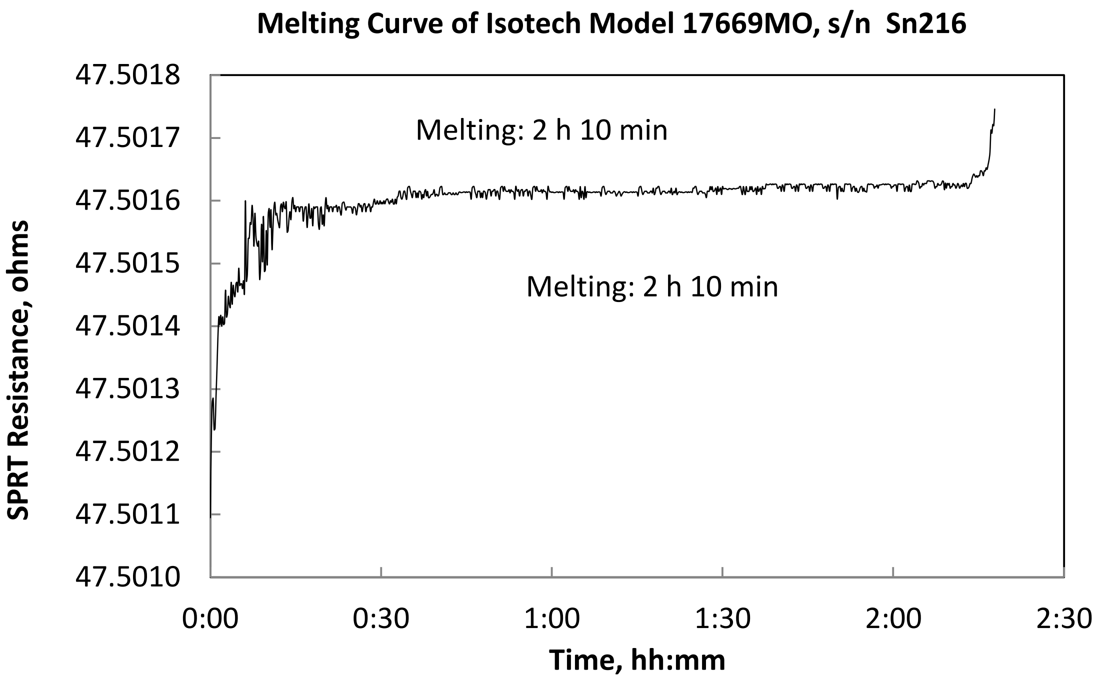
## Appendix C

**Freezing and melting curves measured at INDECOPI, Peru.**

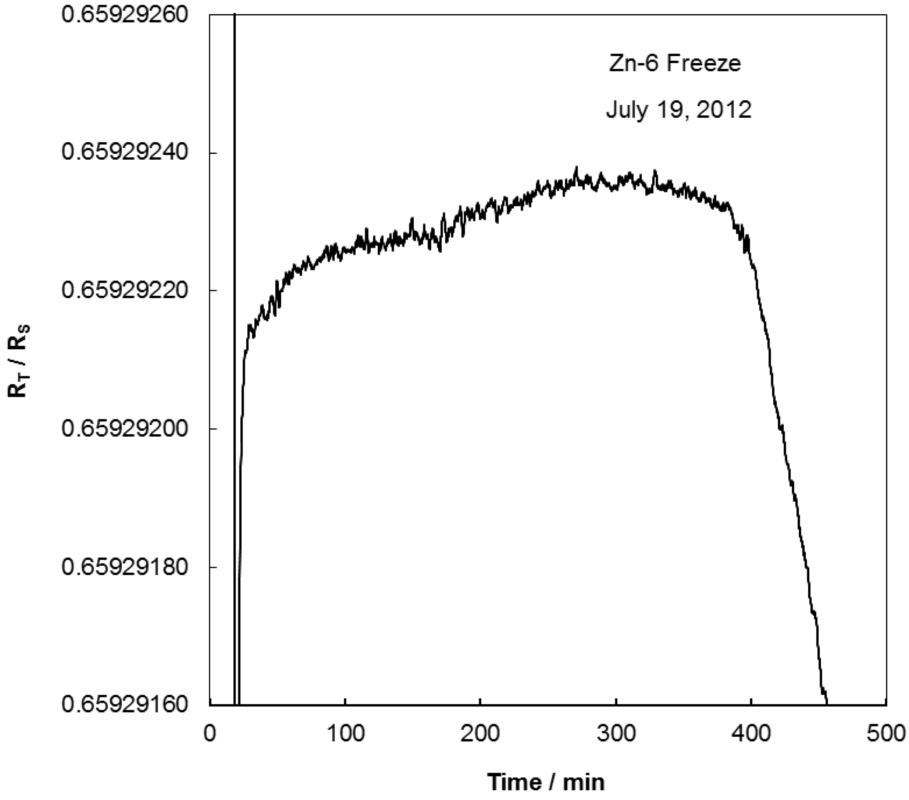


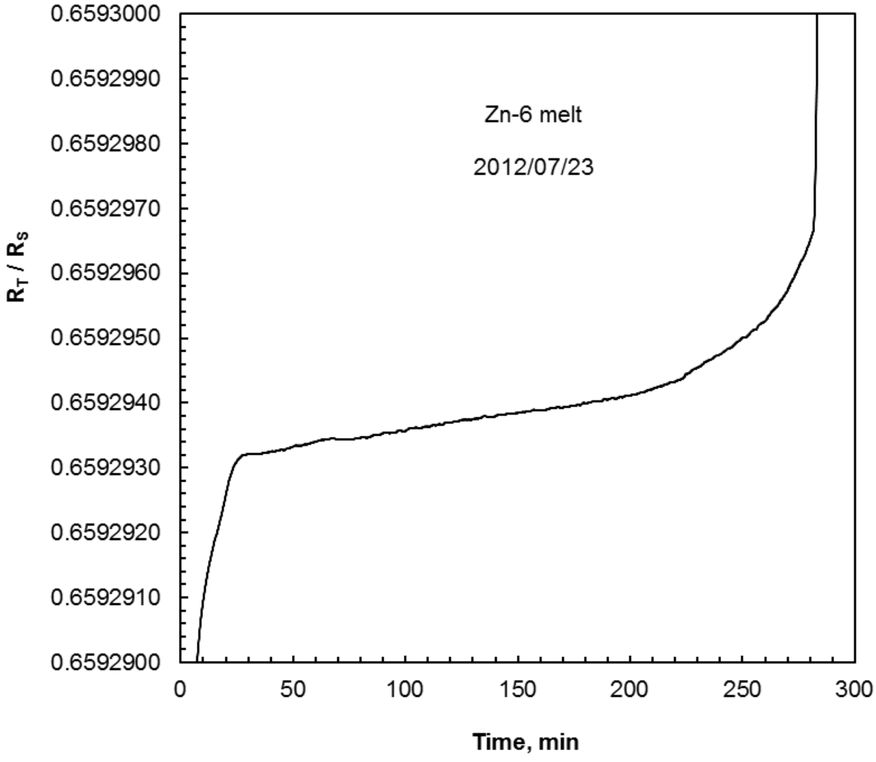



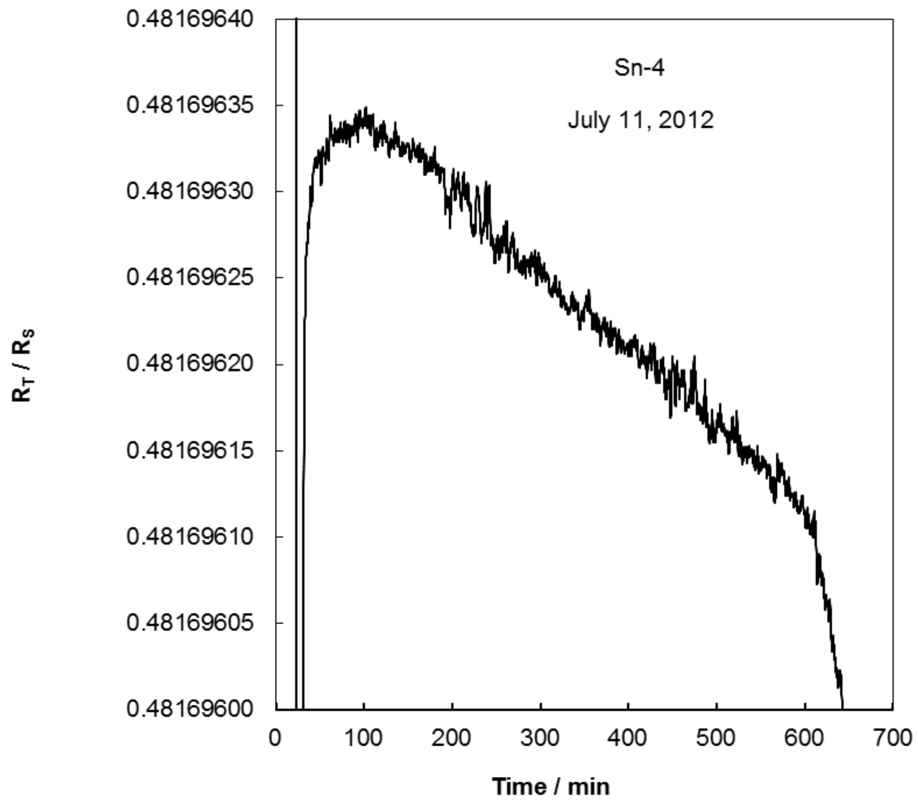

Freezing Curve of Isotech Model 17669MO, s/n Sn216
47.5017
47.5016
47.5015
47.5014
47.5013
SPRT Resistance, ohms
Freezing: 5 h 10 min
0
1
2
3
4
5
6
Time, h


Melting Curve of Isotech Model 17669MO, s/n Sn216
47.5018
47.5017
47.5016
47.5015
47.5014
47.5013
47.5012
47.5011
47.5010
SPRT Resistance, ohms
Melting: 2 h 10 min
Melting: 2 h 10 min
0:00
0:30
1:00
1:30
2:00
2:30
Time, hh:mm

**Freezing and melting curves measured at NRC, Canada**




0.48169640
0.48169635
0.48169630
0.48169625
0.48169620
0.48169615
0.48169610
0.48169605
0.48169600
R_T / R_S
Sn-4
July 11, 2012
0
100
200
300
400
500
600
700
Time / min

## Appendix D

**List of equipment**

| **Laboratory name** | **INDECOPI** | **NRC** |
|---|---|---|
| | | |
| **Resistance measurement** | | |
| Manufacturer | Agilent | ASL |
| Model | 34420A | F18 |
| Serial number | MY42001189 | 1131-5/089 |
| AC or DC | DC | AC |
| If AC, give Frequency | n/a | 90 Hz |
| If DC, give Period of reversal | 5 min | n/a |
| Normal measurement current | 1 mA | 1 mA |
| Self-heating current | 1.18 mA | √2 mA |
| Evaluation of linearity of resistance | yes | yes |
| | | |
| **Reference Resistor** | | |
| Manufacturer | Fluke | Tinsley |
| Model | 742 A-25 | 5685A |
| Serial number | 8691005 | 274676 |
| Nominal value | 25 Ω | 100 Ω |
| Reference resistor – how maintained | Thermostatted enclosure | Guildline 9732VT oil bath |
| Reference resistor - temperature control | ± 0.15 °C | ± 0.002 °C |
| | | |
| **TPW Cell** | | |
| Manufacturer | Isotech | Isotech |
| Model | B11-65-270 | B11 |
| Serial number | 794 | 270 |
| Immersion depth to middle of the SPRT sensitive element | 24 cm | 24 cm |
| How are mantles maintained (ice, bath, …) | Insulated ice box | Insulated ice box |
| | | |
| **Zn Cell** | | |
| Manufacturer | Isotech | NRC |
| Model | 17671MO | n/a |
| Serial number | Zn 260 | Zn-6 |
| Sealed cell or open | sealed | open |
| Nominal purity | 99.9999% | 99.9999% |
| Immersion depth to middle of the SPRT sensitive element | 17 cm | 19.5 cm |

| **Laboratory name** | **INDECOPI** | **NRC** |
|---|---|---|
| | | |
| **Zn Furnace** | | |
| Manufacturer | Isotech | NRC |
| Model | 465 | n/a |
| Type (1 zone, 3 zones, heat pipe, …) | 3 zone | 1 zone |
| Typical duration of the melting / freezing plateau | 5 hours | 7 hours |
| | | |
| **Sn Cell** | | |
| Manufacturer | Isotech | NRC |
| Model | 17669MO | n/a |
| Serial number | Sn 216 | |
| Sealed cell or open | sealed | open |
| Nominal purity | 99.9999% | 99.9999% |
| Immersion depth to middle of the SPRT sensitive element | 17 cm | 18.5 cm |
| | | |
| **Sn Furnace** | | |
| Manufacturer | Isotech | NRC |
| Model | 465 | n/a |
| Type (1 zone, 3 zones, heat pipe, …) | 3 zone | 1 zone |
| Typical duration of the melting / freezing plateau | 5 hours | 10 hours |
| | | |
| **Ga Cell** | | |
| Manufacturer | | NRC |
| Model | | n/a |
| Serial number | | Ga-2 |
| Sealed cell or open | | open |
| Nominal purity | | 99.9999% |
| Immersion depth to middle of the SPRT sensitive element | | 17 cm |
| | | |
| **Ga Furnace** | | |
| Manufacturer | | NRC |
| Model | | n/a |
| Type (1 zone, 3 zones, heat pipe, …) | | 1 zone |
| Typical duration of the melting plateau | | 3 days |

# Appendix E

## INDECOPI Fixed-point Calibration Certificates

Triple Point of Water

Isotech Model B11-65-270, Serial Number 794

NIST Certificate 836/278242-09

Tin Freezing Point

Isotech Model 17669MO, Serial Number Sn 216

NIST Certificate 836/279319-10

Zinc Freezing Point

Isotech Model 17671MO, Serial Number Zn 260

NIST Certificate 836/279319-10

UNITED STATES DEPARTMENT OF COMMERCE
National Institute of Standards and Technology
Gaithersburg, Maryland 20899-

# CERTIFICATE OF ANALYSIS

International Temperature Scale of 1990

Water Triple-Point Cell
Isotech Model B11-65-270
Serial Number 794

Tested for
Servicio Nacional de Metrologia - INDECOPI
Lima, Peru

11 June 2009

**UNITED STATES DEPARTMENT OF COMMERCE**
**National Institute of Standards and Technology**
Gaithersburg, Maryland 20899-

11 June 2009

In reply refer to: 836/278242-09

Servicio Nacional de Metrologia - INDECOPI
Calle La Prosa N°. 138
San Borja
Lima, Peru

Subject: Certification of a water triple-point cell
(Isotech Model B11-65-270, s/n 794)
Purchase Order No.: 20547

Dear Sir:

A direct comparison of your water triple-point cell (Isotech Model B11-65-270, s/n 794) was made against one of our laboratory reference water triple-point cells (s/n A-Q5011). The measurement system included an ASL Model F18 operating at a frequency of 30 Hz with a 100 Ω Tinsley Model 5685A reference resistor, temperature controlled to 298.15 K ± 8 mK, and a 25.5 Ω SPRT. Corrections were made to account for the differences in immersion depth and for the isotopic composition of your cell and the NIST reference cell.

As shown in figure 1, the triple-point temperature of your cell is 0.01 mK colder than that of the NIST reference cell. As given in Appendix A, the expanded uncertainty ($k$=2) of the direct comparison is 0.06 mK. As given in Appendix B, the expanded uncertainty ($k$=2) assigned to the realization of the NIST reference cell is 0.05 mK.

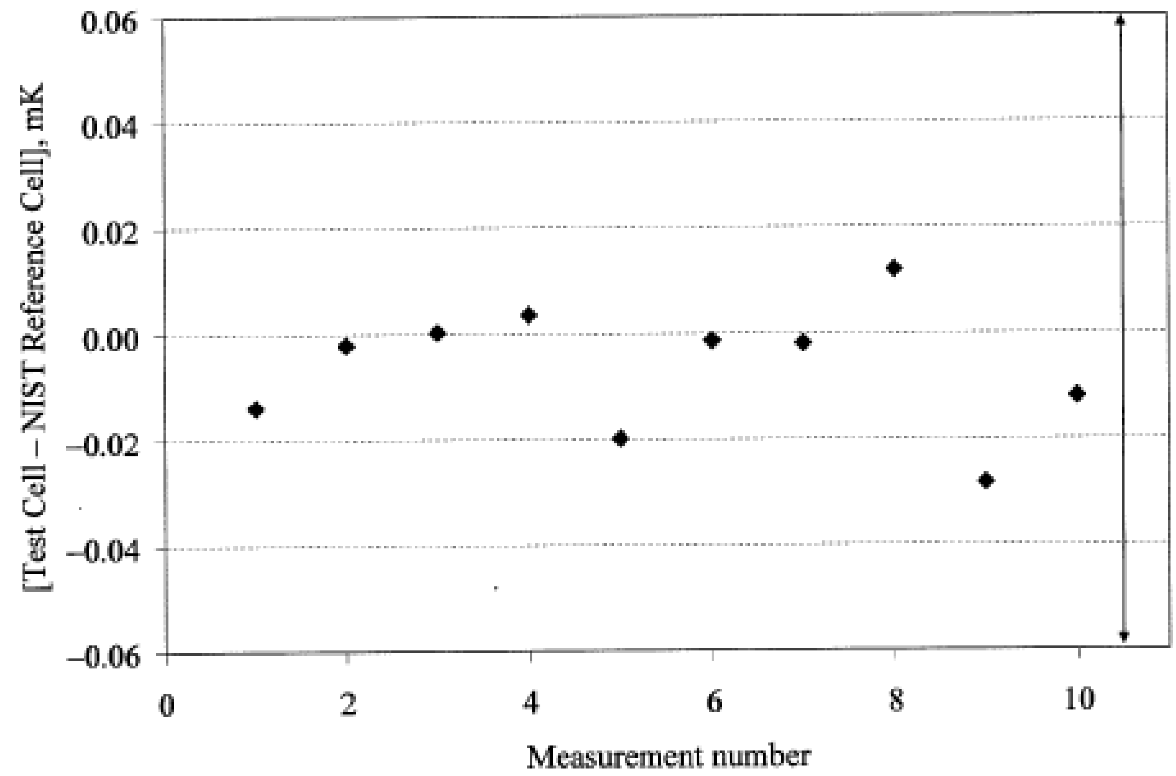



Figure 1. Direct comparison of the INDECOPI water triple-point cell (Isotech Model B11-65-270, s/n 794) with a NIST reference water triple-point cell (s/n A-Q5011). The vertical double ended arrow line indicates the direct comparison uncertainty ($k$=2).

Figure 2 gives an example of the immersion characteristics (heat-flux test), using a Hart Scientific Model 5681 SPRT, of an Isotech Model B11-65-270 water triple-point cell relative to the ITS-90 assigned hydrostatic-head effect for water. A thermometer must track the hydrostatic-head effect over the bottommost 3 cm of the reentrant well to exhibit proper immersion in a fixed-point cell.

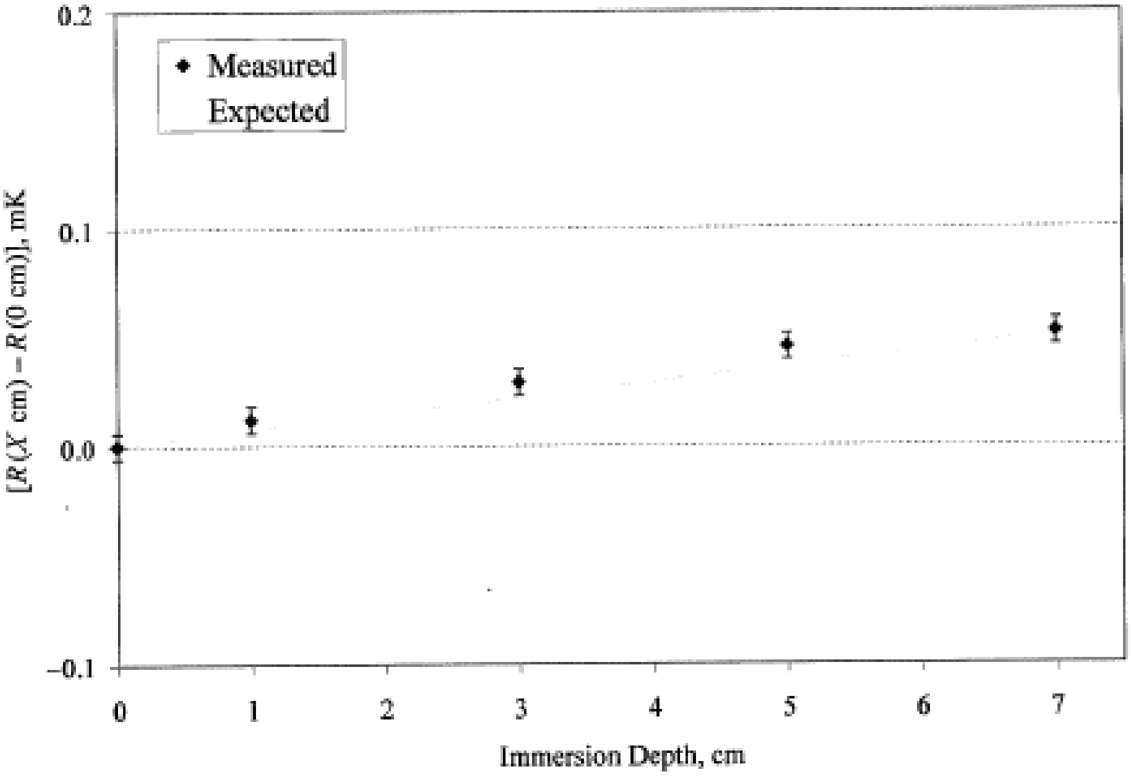



Figure 2. Heat-flux test of an Isotech Model B11-65-270 water triple-point cell using a Hart Scientific Model 5681 SPRT.

Appendix C supplies a suggested uncertainty budget for you to use in assigning an uncertainty to your water triple-point cell as realized in your laboratory. The values left blank are to be determined by yourself in your laboratory using your equipment and personnel. The supplied values are derived from this certification of your cell and the NIST reference cell uncertainty budget. The suggested application of the supplied values is a conservative approach.

Sincerely,

Dean Ripple
Leader, Thermometry Group
Process Measurements Division

**Appendix A: Uncertainty budget for direct comparison of the INDECOPI water triple point cell (Isotech Model B11-65-270, s/n 794) with one of the NIST reference water triple point cells (s/n A-Q5011)**

**Type A**

| | $u_i$ / mK | |
|---|---|---|
| Bridge Repeatibility | 0.01 | both cells |
| Direct Comparison Repeatibility | 0.01 | pooled s.d. of pair differences |
| Total A | 0.01 | |

**Type B (rectangular distribution unless otherwise noted)**

| | | |
|---|---|---|
| Isotopic correction | 0.002 | NIST cell, normal distribution |
| Hydrostatic-head | 0.01 | both cells |
| SPRT self-heating | 0.04 | both cells |
| Immersion (Heat Flux) | 0.01 | both cells, normal distribution |
| Gas pressure | 0.00 | both cells |
| Total B | 0.03 | |
| Total Standard Uncertainty ($k$=1) | 0.03 | |
| Total Expanded Uncertainty ($k$=2) | 0.06 | |

**Appendix B: Uncertainty budget for the realization of a NIST reference water triple point cell (s/n A-Q5011)**

| | $u_i$ / mK |
|---|---|
| Bridge Repeatibility | 0.002 |
| Bridge Non-Linearity | 0.02 |
| Bridge Quadrature Effects (AC only) | 0.01 |
| Reference Resistor Resistance | 0.01 |
| Phase Transistion Realization Repeatibility | 0.005 |
| Chemical Impurities | 0.01 |
| Hydrostatic Head Correction | 0.00 |
| SPRT Self-Heating Correction | 0.02 |
| Heat Flux | 0.003 |
| Gas Pressure | 0.00 |
| Slope of Plateau | 0.00 |
| Isotopic Variation | 0.002 |
| | |
| $u_c$ | 0.024 |
| **$U$ ($k$=2)** | **0.05** |

**Appendix C: Suggested uncertainty budget for the INDECOPI water triple point cell (Isotech Model B11-65-270, s/n 794)**

| | $u_i$ / mK | |
|---|---|---|
| Bridge Repeatibility | | Values and type (e.g. A or B) determined by customer |
| Bridge Non-Linearity | | |
| Bridge Quadrature Effects (AC only) | | |
| Reference Resistor Resistance | | |
| Phase Transistion Realization Repeatibility | | |
| Hydrostatic Head Correction | | |
| SPRT Self-Heating Correction | | |
| Heat Flux | | |
| Isotopic Variation | 0.04 | Value supplied by manufacturer |
| Absolute Value of Direct Comparison Difference | 0.01 | Type B, normal distribution -- NIST suggested method of applying these uncertainties |
| Direct Comparison Measurements | 0.03 | |
| Chemical Impurities in NIST cell | 0.01 | |
| $u_c$ | | |
| $U$ ($k$=2) | | |

Please contact Gregory Strouse at either (301) 975-4803 or gstrouse@nist.gov for assistance in determining the values left blank in Appendix C. These values are necessary for calculating your own fixed-point cell realization uncertainty.

UNITED STATES DEPARTMENT OF COMMERCE
National Institute of Standards and Technology
Gaithersburg, Maryland 20899-

# CERTIFICATE OF ANALYSIS

International Temperature Scale of 1990

Tin Freezing-Point Cell
Isotech Model 17669MO
Serial Number Sn 216

Tested for
Servicio Nacional de Metrologia (INDECOPI)
Lima, Peru

28 February 2010

UNITED STATES DEPARTMENT OF COMMERCE
National Institute of Standards and Technology
Gaithersburg, Maryland 20899-

Servicio Nacional de Metrologia (INDECOPI)
Ing. Edwin Guillén Mestas
Calle La Prosa N°. 138
San Borja
Lima, Peru

Subject: Certification of a Sn FP cell (Isotech Model 17669MO, s/n Sn 216)
Purchase Order No.: 20650

Dear Ing. Edwin Guillén Mestas:

A direct comparison of your tin freezing-point cell (Isotech Model 17669MO, s/n Sn 216) was made against our laboratory reference tin freezing-point cell (Sn 88A). The measurement system included an ASL Model F18 operating at a frequency of 30 Hz with a 100 Ω Tinsley Model 5685A reference resistor, temperature controlled to 298.15 K ± 8 mK, and a 25.5 Ω SPRT. The immersion depth from the inside bottom of the thermometer well to the liquid surface of your tin freezing-point cell is stated to be 20 cm; the depth of our cell is 20.5 cm The internal pressure of your sealed cell is stated by the manufacturer to be 101.3 kPa during realization. The NIST tin freezing-point cell was set to 101.3 kPa during realization. The direct comparison results are corrected for the differences in immersion depth.

As shown in Fig. 1, the freezing-point temperature of your cell is 0.86 mK colder than that of the NIST reference cell Sn 88A. As given in Appendix A, the expanded uncertainty ($k$=2) of the direct comparison is 0.22 mK. As given in Appendix B, the expanded uncertainty ($k$=2) assigned to the realization of the NIST reference cell Sn 88A is 0.28 mK.

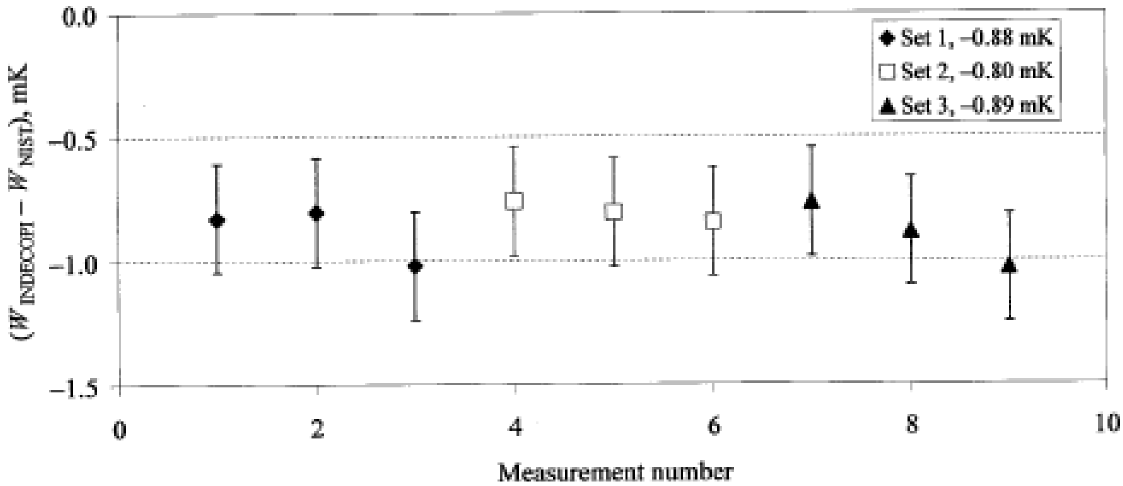



Figure 1. Direct comparison results of the INDECOPI Sn FP cell (Isotech Model 17669MO, s/n Sn 216) with the NIST reference Sn FP cell (Sn 88A).

836/279319-10 Page 2 of 8

NIST

Figures 2 and 3 give examples of freezing and melting curves for your cell, respectively.

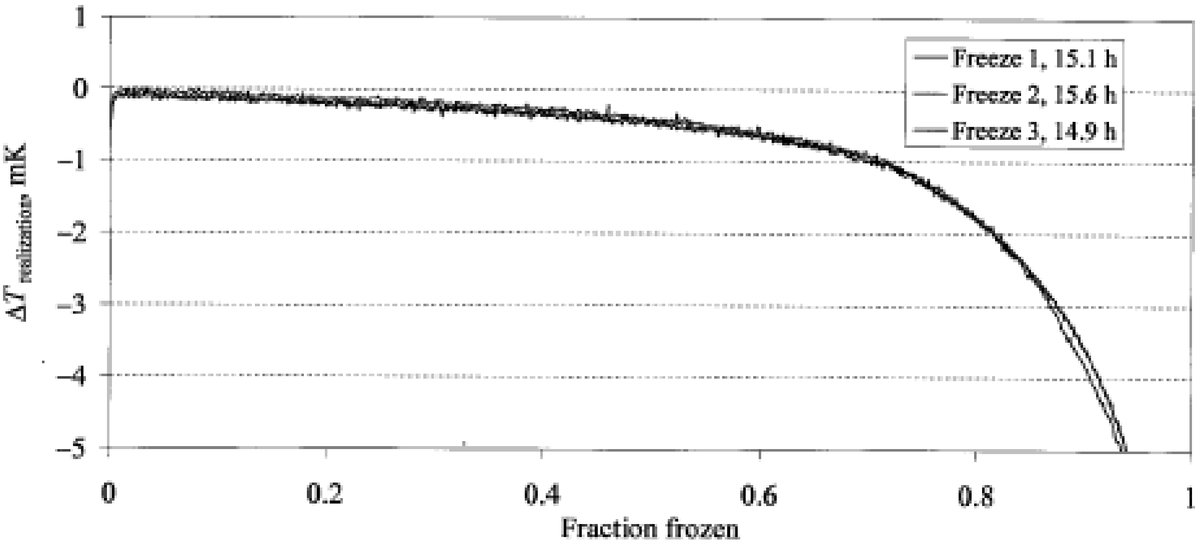



Figure 2. Freezing curves for the INDECOPI Sn FP cell (Isotech Model 17669MO, s/n Sn 216).

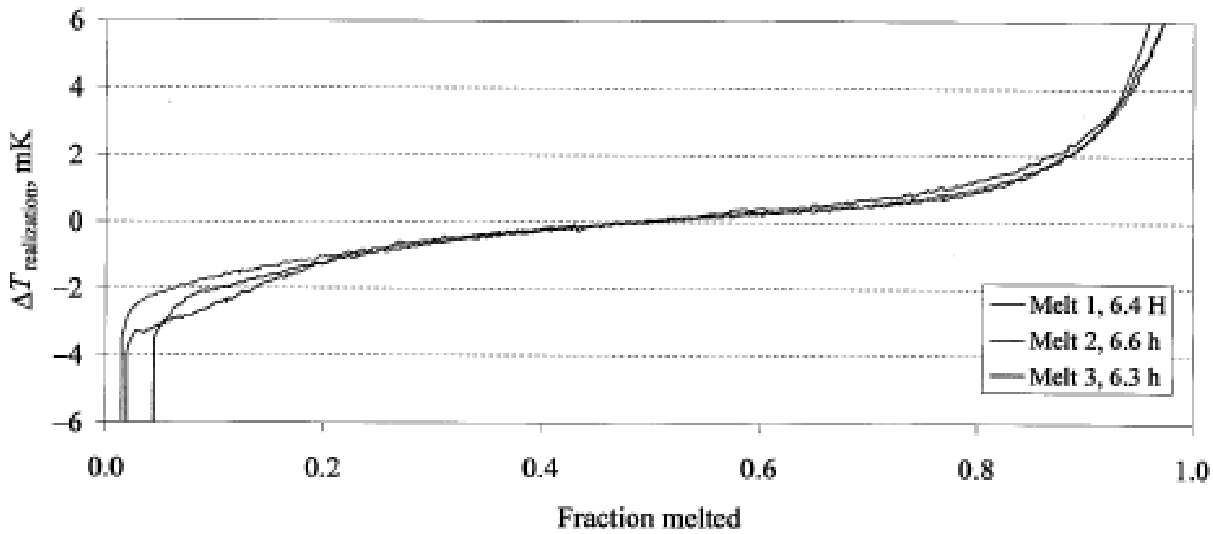



Figure 3. Melting curves for the INDECOPI Sn FP cell (Isotech Model 17669MO, s/n Sn 216).

An estimate of the impurity of the Sn sample contained with your cell (s/n Sn 216) may be estimated using different methods of analysis as described in Strouse, G. F., "NIST Method of Estimating the

Impurity Uncertainty Components for the ITS-90 Fixed-Point Cells from the Ar TP to the Ag FP," CCT 03-19 (2003); and Fellmuth, B. and Hill, K.D., "Estimating the influence of impurities on the freezing point of tin," *Metrologia* **43**, 71-83 (2006). Table 1 gives the results for the estimation of impurity for one method (e.g., freezing curve). No metal analysis information was available from Isotech to perform a mole fraction impurity analysis. Additionally, as a cross check on the impurity calculation, the difference between the NIST reference cell and the tested cell is within 0.21 mK of the freezing curve estimate of the impurity effect.

Table 1. Estimated impurity uncertainty component ($k$=1) results for two methods of analysis.

| **Method of analysis** | **Estimated impurity uncertainty component, mK** |
|---|---|
| Mole fraction sum of impurities | unknown |
| Freezing curve | 0.72 |

Figure 4 gives an example of a heat flux test (immersion characteristics) of an SPRT in your cell relative to the ITS-90 assigned hydrostatic-head effect for tin (2.2 mK/m). A thermometer must track the hydrostatic-head effect over the bottommost 3 cm of the reentrant well to exhibit proper immersion in a fixed-point cell.

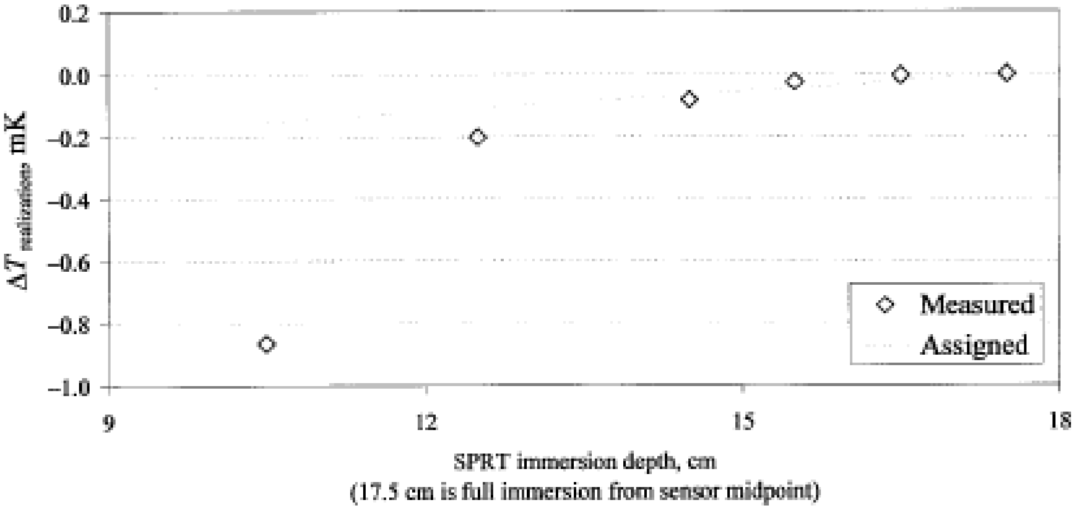



Figure 4. Heat-flux test results of the INDECOPI Sn FP cell (Isotech Model 17669MO, s/n Sn 216) during realization using an SPRT L&N Model 8167.

Appendix C supplies a suggested uncertainty budget for you to use in assigning an uncertainty to your tin freezing-point cell as realized in your laboratory. The values left blank must be determined by yourself, in your laboratory using your equipment and personnel. Values given in Appendix B are not be used to complete Appendix C. The supplied values are derived from this certification of your cell and the NIST Sn reference-cell uncertainty budget. The suggested application of the supplied values is a conservative approach.

Further information, see Strouse, Gregory F., "NIST Certification of ITS-90 Fixed-Point Cells from 83.8058 K to 1234.93 K: Methods and Uncertainties," *Proc. Tempmeko 2004*, pp. 879-884.

Sincerely,

Gregory F. Strouse
Acting Group Leader, Thermometry
Process Measurements Division

**Appendix A: Uncertainty budget for NIST direct comparison of the INDECOPI Sn FP cell (Isotech Model 17669MO, s/n Sn 216) with the NIST reference Sn FP cell (Sn 88A)**

**Type A**

| | $u_i$ / mK | |
|---|---|---|
| Bridge Repeatibility | 0.007 | both cells |
| Direct Comparison Repeatibility | 0.10 | pooled s.d. of pair differences ($n$=9) |
| Total A | 0.10 | |

**Type B (rectangular distribution unless otherwise noted)**

| | | |
|---|---|---|
| Hydrostatic-head | 0.03 | both cells |
| SPRT self-heating | 0.03 | both cells |
| Immersion (Heat Flux) | 0.03 | both cells, normal distribution |
| Gas pressure | 0.02 | both cells |
| Total B | 0.04 | |
| Total Standard Uncertainty ($k$=1) | 0.11 | |
| Total Expanded Uncertainty ($k$=2) | 0.22 | |

**Appendix B: Uncertainty budget for the realization of the NIST reference Sn FP cell (Sn 88A)**

| | $u_i$ / mK |
|---|---|
| Bridge Repeatibility | 0.003 |
| Bridge Non-Linearity | 0.02 |
| Bridge Quadrature Effects (AC only) | 0.02 |
| Reference Resistor Resistance | 0.01 |
| Phase Transistion Realization Repeatibility | 0.12 |
| Chemical Impurities | 0.06 |
| Hydrostatic Head Correction | 0.01 |
| SPRT Self-Heating Correction | 0.02 |
| Heat Flux | 0.003 |
| Gas Pressure | 0.02 |
| Slope of Plateau | 0.00 |
| Isotopic Variation | 0.00 |
| Propagation of TPW | 0.05 |
| $u_c$ | 0.14 |
| **$U$ ($k$=2)** | **0.28** |

**Appendix C: Suggested uncertainty budget for the INDECOPI Sn FP cell (Isotech Model 17669MO, s/n Sn 216)**

| | $u_i$ / mK | |
|---|---|---|
| Bridge Repeatibility | | Values and type (e.g. A or B) determined by customer |
| Bridge Non-Linearity | | |
| Bridge Quadrature Effects (AC only) | | |
| Reference Resistor Resistance | | |
| Check SPRT Repeatibility | | |
| Hydrostatic Head Correction | | |
| SPRT Self-Heating Correction | | |
| Heat Flux | | |
| Gas Pressure | | |
| Slope of Plateau | | |
| Propagation of TPW | | |
| Absolute Value of Direct Comparison Difference | 0.86 | Type B, normal distribution -- NIST suggested method of applying these uncertainties |
| Direct Comparison Measurements | 0.11 | |
| Chemical Impurities in NIST cell | 0.06 | |
| $u_c$ | | |
| $U$ ($k$=2) | | |

Please contact Gregory Strouse at either (301) 975-4803 or gstrouse@nist.gov for assistance in determining the values left blank in Appendix C. These values are necessary for calculating your own fixed-point cell realization uncertainty.

UNITED STATES DEPARTMENT OF COMMERCE
National Institute of Standards and Technology
Gaithersburg, Maryland 20899-

# CERTIFICATE OF ANALYSIS

International Temperature Scale of 1990

Zinc Freezing-Point Cell
Isotech Model 17671MO
Serial Number Zn 260

Tested for
Servicio Nacional de Metrologia (INDECOPI)
Lima, Peru

28 February 2010

UNITED STATES DEPARTMENT OF COMMERCE
National Institute of Standards and Technology
Gaithersburg, Maryland 20899-

Servicio Nacional de Metrologia (INDECOPI)
Ing. Edwin Guillén Mestas
Calle La Prosa N°. 138
San Borja
Lima, Peru

Subject: Certification of a Zn FP cell (Isotech Model 17671MO, s/n Zn 260)
Purchase Order No.: 20650

Dear Ing. Edwin Guillén Mestas:

A direct comparison of your zinc freezing-point cell (Isotech Model 17671MO, s/n Zn 260) was made against our laboratory reference zinc freezing-point cell (Zn 08A). The measurement system included an ASL Model F18 operating at a frequency of 30 Hz with a 100 Ω Tinsley Model 5685A reference resistor, temperature controlled to 298.15 K ± 8 mK, and a 25.5 Ω SPRT. The immersion depth from the inside bottom of the thermometer well to the liquid surface of your zinc freezing-point cell is stated to be 20 cm; the depth of our cell is 20.5 cm The internal pressure of your sealed cell is stated by the manufacturer to be 101.3 kPa during realization. The NIST zinc freezing-point cell was set to 101.3 kPa during realization. The direct comparison results are corrected for the differences in immersion depth.

As shown in Fig. 1, the freezing-point temperature of your cell is 0.86 mK colder than that of the NIST reference cell Zn 08A. As given in Appendix A, the expanded uncertainty ($k$=2) of the direct comparison is 0.22 mK. As given in Appendix B, the expanded uncertainty ($k$=2) assigned to the realization of the NIST reference cell Zn 08A is 0.51 mK.

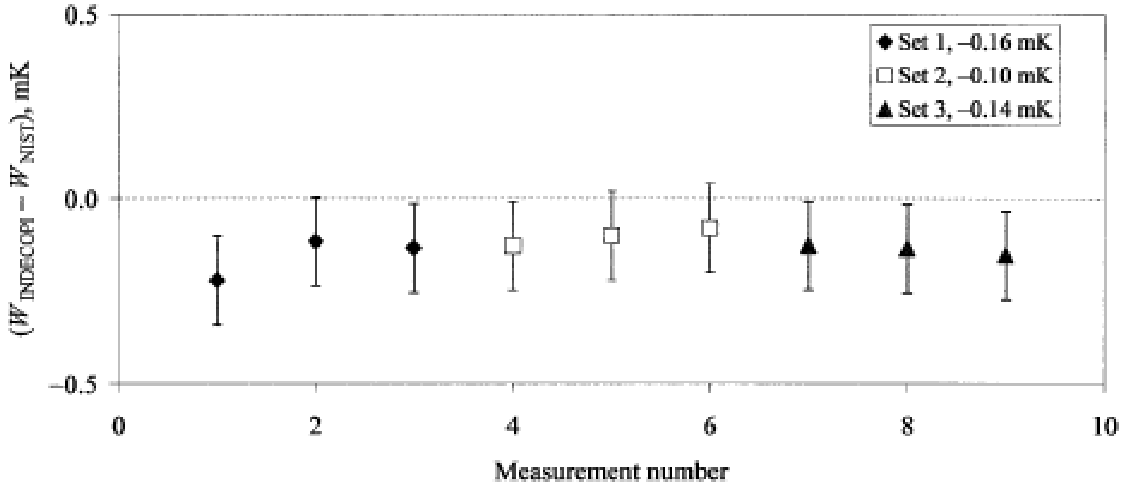



Figure 1. Direct comparison results of the INDECOPI Zn FP cell (Isotech Model 17671MO, s/n Zn 260) with the NIST reference Zn FP cell (Zn 08A).

Figures 2 and 3 give examples of freezing and melting curves for your cell, respectively.

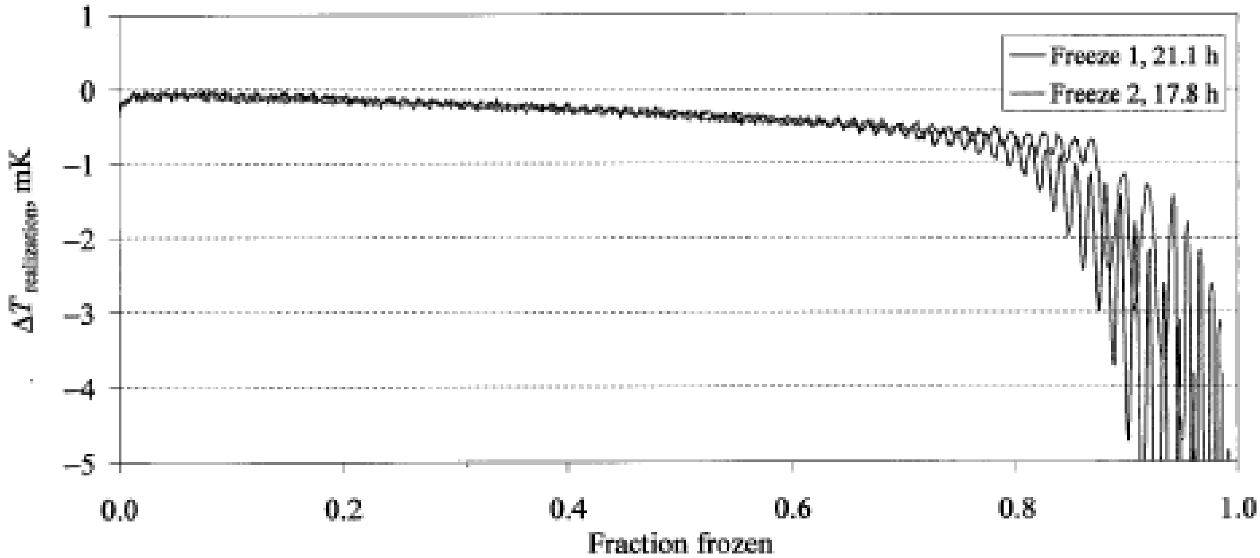



Figure 2. Freezing curves for the INDECOPI Zn FP cell (Isotech Model 17671MO, s/n Zn 260).

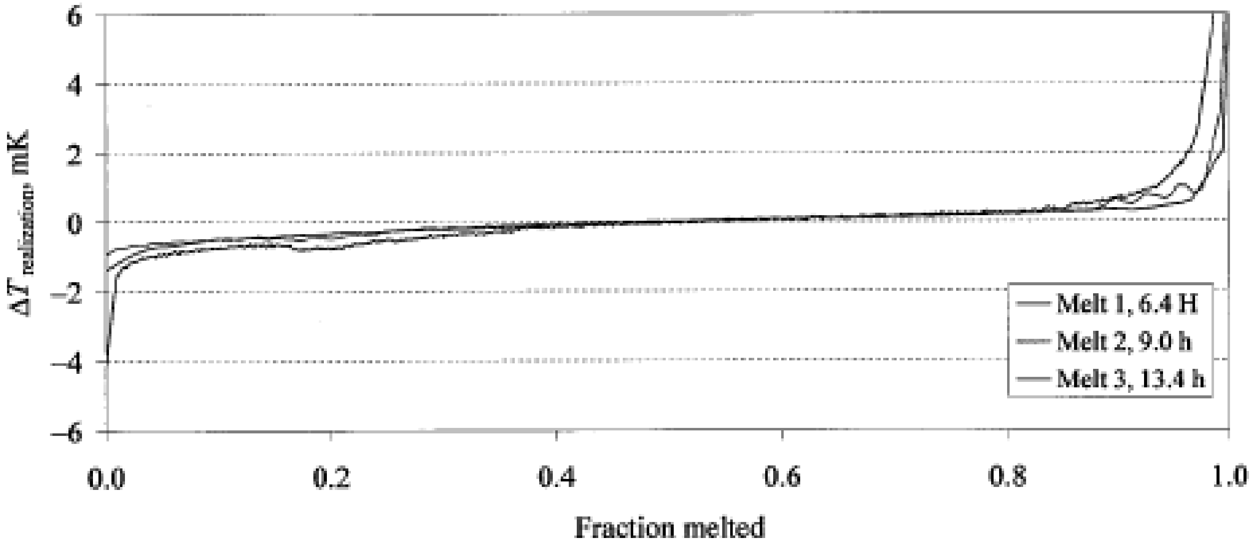



Figure 3. Melting curves for the INDECOPI Zn FP cell (Isotech Model 17671MO, s/n Zn 260).

An estimate of the impurity of the Zn sample contained with your cell (s/n Zn 260) may be estimated using different methods of analysis as described in Strouse, G. F., "NIST Method of Estimating the

Impurity Uncertainty Components for the ITS-90 Fixed-Point Cells from the Ar TP to the Ag FP," CCT 03-19 (2003); and Fellmuth, B. and Hill, K.D., "Estimating the influence of impurities on the freezing point of tin," *Metrologia* **43**, 71-83 (2006). Table 1 gives the results for the estimation of impurity for one method (e.g., freezing curve). No metal analysis information was available from Isotech to perform a mole fraction impurity analysis. Additionally, as a cross check on the impurity calculation, the difference between the NIST reference cell and the tested cell is within 0.08 mK of the freezing curve estimate of the impurity effect.

Table 1. Estimated impurity uncertainty component ($k$=1) results for two methods of analysis.

| **Method of analysis** | **Estimated impurity uncertainty component, mK** |
|---|---|
| Mole fraction sum of impurities | unknown |
| Freezing curve | 0.39 |

Figure 4 gives an example of a heat flux test (immersion characteristics) of an SPRT in your cell relative to the ITS-90 assigned hydrostatic-head effect for zinc (2.7 mK/m). A thermometer must track the hydrostatic-head effect over the bottommost 3 cm of the reentrant well to exhibit proper immersion in a fixed-point cell.

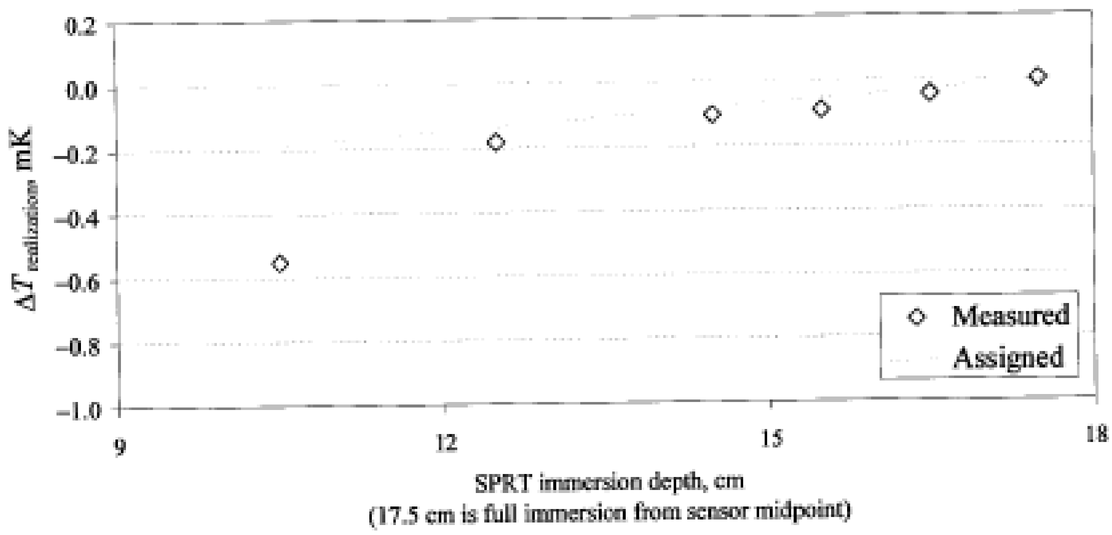



Figure 4. Heat-flux test results of the INDECOPI Zn FP cell (Isotech Model 17671MO, s/n Zn 260) during realization using an SPRT L&N Model 8167.

Appendix C supplies a suggested uncertainty budget for you to use in assigning an uncertainty to your zinc freezing-point cell as realized in your laboratory. The values left blank must be determined by yourself, in your laboratory using your equipment and personnel. Values given in Appendix B are not be used to complete Appendix C. The supplied values are derived from this certification of your cell and the NIST Zn reference-cell uncertainty budget. The suggested application of the supplied values is a conservative approach.

Further information, see Strouse, Gregory F., "NIST Certification of ITS-90 Fixed-Point Cells from 83.8058 K to 1234.93 K: Methods and Uncertainties," *Proc. Tempmeko 2004*, pp. 879-884.

Sincerely,

Gregory F. Strouse
Acting Group Leader, Thermometry
Process Measurements Division

**Appendix A: Uncertainty budget for NIST direct comparison of the INDECOPI Zn FP cell (Isotech Model 17671MO, s/n Zn 260 with the NIST reference Zn FP cell (Zn 08A)**

**Type A**

| | $u_i$ / mK | |
|---|---|---|
| Bridge Repeatibility | 0.007 | both cells |
| Direct Comparison Repeatibility | 0.04 | pooled s.d. of pair differences ($n$ =9) |
| Total A | 0.04 | |

**Type B (rectangular distribution unless otherwise noted)**

| | | |
|---|---|---|
| Hydrostatic-head | 0.03 | both cells |
| SPRT self-heating | 0.03 | both cells |
| Immersion (Heat Flux) | 0.03 | both cells, normal distribution |
| Gas pressure | 0.02 | both cells |
| Total B | 0.05 | |
| Total Standard Uncertainty ($k$ =1) | 0.06 | |
| Total Expanded Uncertainty ($k$ =2) | 0.12 | |

**Appendix B: Uncertainty budget for the realization of the NIST reference Zn FP cell (Zn 08A)**

| | $u_i$ / mK |
|---|---|
| Bridge Repeatibility | 0.010 |
| Bridge Non-Linearity | 0.02 |
| Bridge Quadrature Effects (AC only) | 0.02 |
| Reference Resistor Resistance | 0.01 |
| Phase Transistion Realization Repeatibility | 0.18 |
| Chemical Impurities | 0.17 |
| Hydrostatic Head Correction | 0.01 |
| SPRT Self-Heating Correction | 0.02 |
| Heat Flux | 0.003 |
| Gas Pressure | 0.03 |
| Slope of Plateau | 0.00 |
| Isotopic Variation | 0.00 |
| Propagation of TPW | 0.08 |
| $u_c$ | 0.25 |
| **$U$ ($k$=2)** | **0.51** |

**Appendix C: Suggested uncertainty budget for the INDECOPI Zn FP cell (Isotech Model 17671MO, s/n Zn 260**

| | $u_i$ / mK | |
|---|---|---|
| Bridge Repeatibility | | Values and type (e.g. A or B) determined by customer |
| Bridge Non-Linearity | | |
| Bridge Quadrature Effects (AC only) | | |
| Reference Resistor Resistance | | |
| Check SPRT Repeatibility | | |
| Hydrostatic Head Correction | | |
| SPRT Self-Heating Correction | | |
| Heat Flux | | |
| Gas Pressure | | |
| Slope of Plateau | | |
| Propagation of TPW | | |

| | | |
|---|---|---|
| Absolute Value of Direct Comparison Difference | 0.13 | Type B, normal distribution -- NIST suggested method of applying these uncertainties |
| Direct Comparison Measurements | 0.06 | |
| Chemical Impurities in NIST cell | 0.17 | |

| | |
|---|---|
| $u_c$ | |
| $U$ ($k$=2) | |

Please contact Gregory Strouse at either (301) 975-4803 or gstrouse@nist.gov for assistance in determining the values left blank in Appendix C. These values are necessary for calculating your own fixed-point cell realization uncertainty.