FINAL REPORT

CCT-K9.2

Report on the comparison of the realization of the ITS-90 over the range 234.315 6 K to 692.677 K between Centro Español de Metrología (CEM) and Servicio Ecuatoriano de Normalización (INEN)

April 2025

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1. Introduction

The comparison of the realization of the International Temperature Scale of 1990 (ITS- 90) over the range 234.315 6 K (triple point of mercury) to 692.677 K (freezing point of Zinc) in the National Metrology Institutes of Spain (Centro Español de Metrología - CEM) and Ecuador (Servicio Ecuatoriano de Normalización - INEN) has been organized with the aim to provide support to the Calibration Measurement Capabilities claimed by INM in this range. Due to the participation of CEM in the regional comparison EURAMET.T-K9, the linkage with the corresponding key comparison, CCT-K9, is possible.

The measurements of this comparison were performed during January 2022 and January 2023.

Participants

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2. Protocol

The protocol of this comparison (see annex 1) was initially agreed between CEM, INEN and CESMEC (INM Chile). After receiving some comments from the CCT-WG-KC reviewers in September 2021, a new version of the protocol, addressing all the reviewers' comments was sent, approved by the CCT-WG-KC and registered in the BIPM KCDB on December 2021. Nevertheless CESMEC finally was unable to join the comparison so a new version of the protocol was edited by February 2021 withdrawing CESMEC participation.

The scheme of measurements finally carried out is described in table 1.

Date	Laboratory	Action
January 2022	INEN	Al, Zn, Sn, In, Ga and Hg measurements. (Before).
April 2022 - July 2022	CEM	Zn, Sn, In, Ga and Hg measurements.
December 2022 – January 2023	INEN	Zn, Sn, In, Ga and Hg measurements (after).

Table 1. Schedule of the comparison

Initially the comparison was planned for the fixed points of Hg, Ga, In, Sn, Zn and Al. As the SPRTs, dedicated for the Al measurements was damaged during the travel from Ecuador to Spain, it was decided to eliminate this fixed point from this comparison. Table 2 summarizes the equipment used for both laboratories during the comparison.

CEM maintains their fixed points by means of a group of cells, periodically comparisons are performed to assure their integrity. In addition control SPRTs are assigned to each fixed point and all the plateaux performed are initiated and finalised using them. CEM has used in this comparison the same reference cells used in the EURAMET.T-K9 comparison.

To ensure the traceability of the measurement, INEN's zinc, tin, indium and mercury fixed-point cells were calibrated by the National Institute of Metrology (NIST) of the United States, the water cells have internal traceability through of a triple point water cell No. serial 420-A053 calibrated at CENAM. Table 3 present the traceability of the INEN cells.

Laboratory name	CEM	INEN
Bridge		
Manufacturer	A.S.L.	ISOTECH
Type	F18 /F900	Microk
AC or DC	AC	AC
If AC, give Frequency	75 Hz	
If DC, give Period of reversal	-	-
Normal measurement current	1 mA	1 mA
Self-heating current	√2 mA	√2 mA
Evaluation of linearity of resistance	Yes	Yes
Bridge calibration (yes or not)	Yes	Yes
If yes, How?	Using Resistance Bridge Calibrator (RBC)	Certificate of Analysis NIST 685/292668-19
Reference resistor		
Manufacturer / type	Tinsley / Wilkins (model 5685 A)	Tinsley/5685A; serie 17894/08
Reference resistor temperature control (yes or not)	Yes	Yes
If yes, How?	Oil bath: (23 ± 0,01) ºC	ISOTECH maintenance bath 20 ºC
TPW Cell		
Manufacturer / model / sn	Jarret / A13 / 1179	Fluke/D-G1402
Is it a primary reference? (if not explain its traceability)	Yes	Certificate os Analysis
Immersion depth of middle of the SPRT sensitive element/cm	25	23,5
How are mantles maintained (ice, bath,….)	Stirred water bath	Fluke Hart Scientific 7312 maintenance bath
Zn Cell		
Manufacturer / model /sn	Isotech / ITLM17671 / Zn 11	Isotech / 17671 MO / Zn 252
Is it a primary reference? (if not explain its traceability)	Yes	Certificate of analysis NIST 685/283181-13
Immersion depth of middle of the SPRT sensitive element / cm	12	17
Closed cell or open	Open	Closed
Nominal purity	99,999 9 %	99.999 9 %
Zn Furnace		
Type (1 zone, 3 zones, heat pipe, …….)	3 zones	Isotech/ITL 17703 (3 zones)
Typical duration of the melting / freezing plateaux	9 h / 8,5 h	9 h
Sn Cell		
Manufacturer / model /sn	L&N / 8411 / 742876	Isotech / 17669 MO / Sn 250
Is it a primary reference? (if not explain its traceability)	Yes	Certificate of analysis NIST 685/283181-13
Immersion depth of middle of the SPRT sensitive element/cm	15	17
Closed cell or open	Open	Closed
Nominal purity	99,999 9 %	99.999 9 %
Sn Furnace		
Type (1 zone, 3 zones, heat pipe, …….)	3 zones	Isotech/ITL 17703 (3 zones)
Typical duration of the melting / freezing plateaux	13 h / 9,5 h	7 h
In Cell		
Manufacturer / model /sn	Isotech 7 ITL-M.17688-O / In 97	Isotech / 17668 MO / In 214
Is it a primary reference? (if not explain its traceability)	Yes	Certificate of analysis NIST 685/283181-13
Immersion depth of middle of the SPRT sensitive element/cm	13	17
Closed cell or open	open	Closed
Nominal purity	99,999 9 %	99.999 9 %
In Furnace		
Type (1 zone, 3 zones, heat pipe, …….)	3 zones	Isotech/ITL 17703 (3 zones)
Typical duration of the melting / freezing plateaux	8.5 h	10 h
Ga Cell		
Manufacturer / model /sn	YSI / 17401 / L8256	ISOTECH / 17401 / Ga 506
Is it a primary reference? (if not explain its traceability)	Yes	Certificate of analysis NIST 685/283181-13
Immersion depth of middle of the SPRT sensitive element/cm	25	20
Closed cell or open	Closed	Closed
Nominal purity	99,999 9+ %	99.999 99 %
Ga Furnace		
Type (1 zone, 3 zones, heat pipe, …….)	1 zone	Isotech/17402B (Thermoelectric heat pump module)
Typical duration of the melting / freezing plateaux	9 h / 8 h	12 h
Hg Cell		
Manufacturer / model	Isotech / ITLM-17924 / Hg 62	Isotech / 17724 / Hg 258
Is it a primary reference? (if not explain its traceability)	yes	Certificate of analysis NIST 685/283181-13
Immersion depth of middle of the SPRT sensitive element/cm	17	17
Closed cell or open	Closed	Closed
Nominal purity	99,999 95 %	99.999 99 %
Hg cryostat		
Type (cryostat, bath, …….)	Alcohol stirred bath	Isotech/ITL-M-17725 (cryostat)
Typical duration of the melting / freezing plateaux	9 h / 8,5 h	8 h / 9 h

Table 2. Summary of the equipment used

Fixed point	Calibration date	Source of traceability
Zn	2013/5/22	NIST
Sn	2013/5/22	NIST
In	2013/5/22	NIST
Ga	2013/5/22	NIST
H_2O	2022/10/12	Internal
Hg	2013/1/10	NIST

Table 3. INEN cell traceability

3. Transfer standards

The transfer standard was a 25 Ω SPRT (initially, one more was planned for the Al fixed point but it arrived damaged after travel from Ecuador to Spain). The thermometer had proven stability and was provided by INEN.

Manufacturer	Model	Serial number	Calibration points
Fluke Hart Scientific	5681	1502	Zn, Sn, In, Ga and Hg

Table 4. Transfer standards

The resistance of the travelling SPRT was measured at two currents, in order to determine the zero-power value. All the measurements were corrected for the hydrostatic head to obtain the resistance values.

4. Results

Table 5 presents the *W* and uncertainty values calculated for a coverage probability of about 95 %, reported by CEM and INEN. Two realizations were performed for each fixed point, and for each of the values presented below (two for the "INEN before" value, two for the "CEM" value, and two for the "INEN after" value).

	INEN before		CEM		INEN after	
Fixed Point	***W***	***U* [mK]**	***W***	***U* [mK]**	***W***	***U* [mK]**
Zn	2.568 785 5	4.8	2.568 789 10	0.90	2.568 784 8	4.8
Sn	1.892 728 8	2.6	1.892 730 98	0.49	1.892 729 5	2.6
In	1.609 757 2	2.3	1.609 756 16	0.80	1.609 756 5	2.3
Ga	1.118 133 0	1.1	1.118 129 81	0.26	1.118 132 4	1.1
Hg	0.844 148 7	1.8	0.844 149 55	0.43	0.844 148 9	1.8

Table 5. Results reported by the participants

Figures 1 to 5 present the results of the comparison in graphical form showing also the good behaviour of the travelling standards.

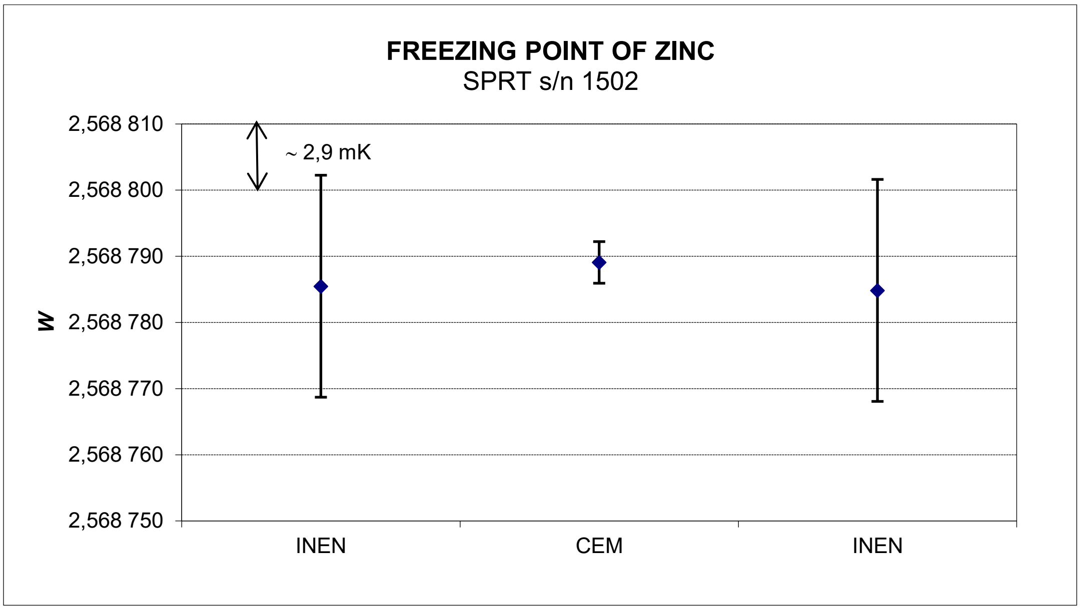

Figure 1. Freezing point of zinc results

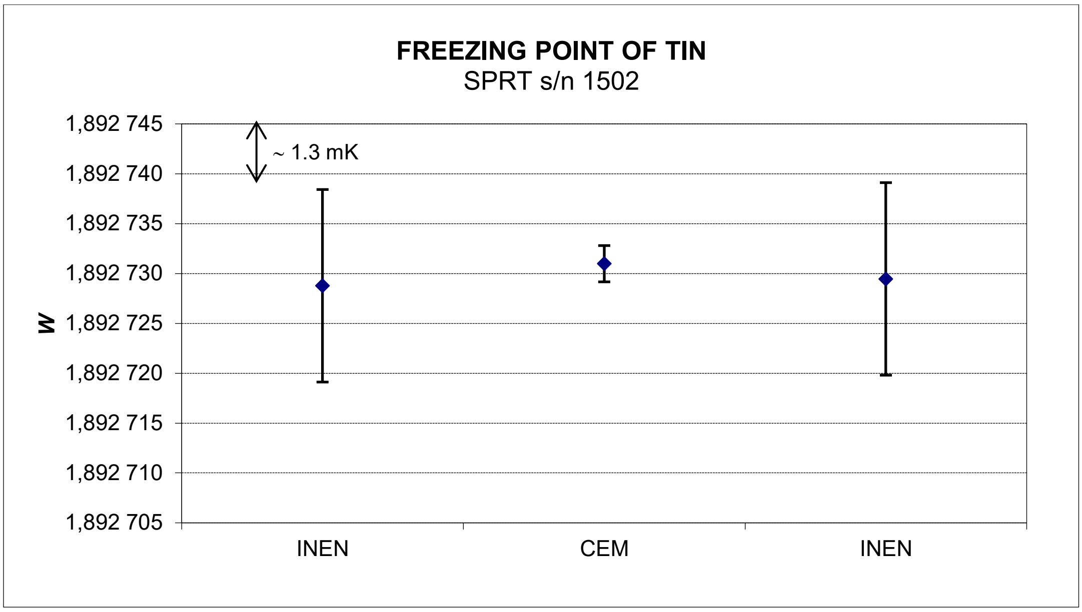

Figure 2. Freezing point of tin results

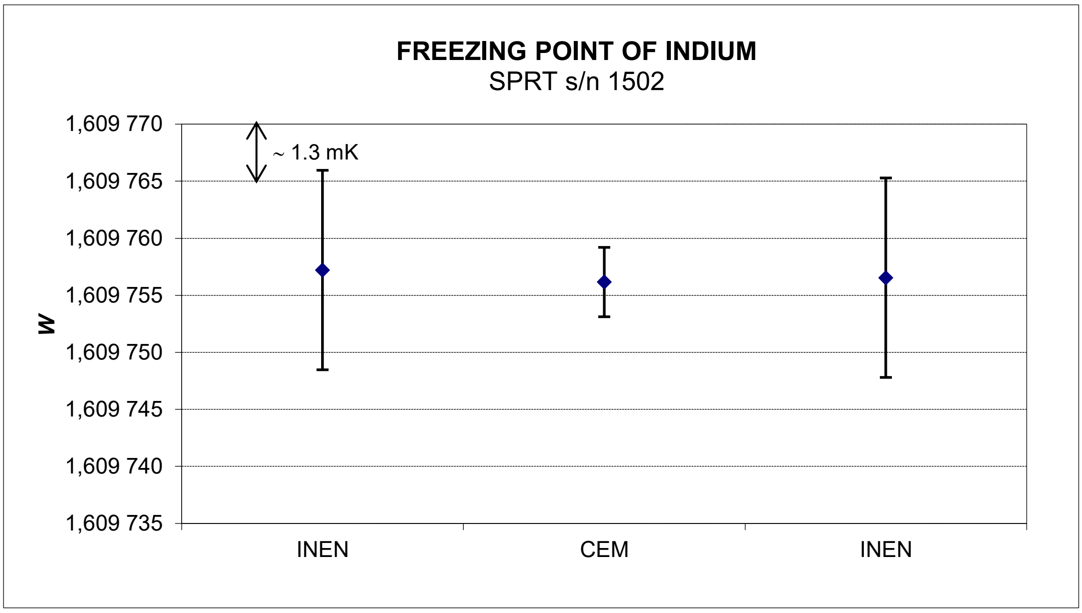

Figure 3. Freezing point of indium results

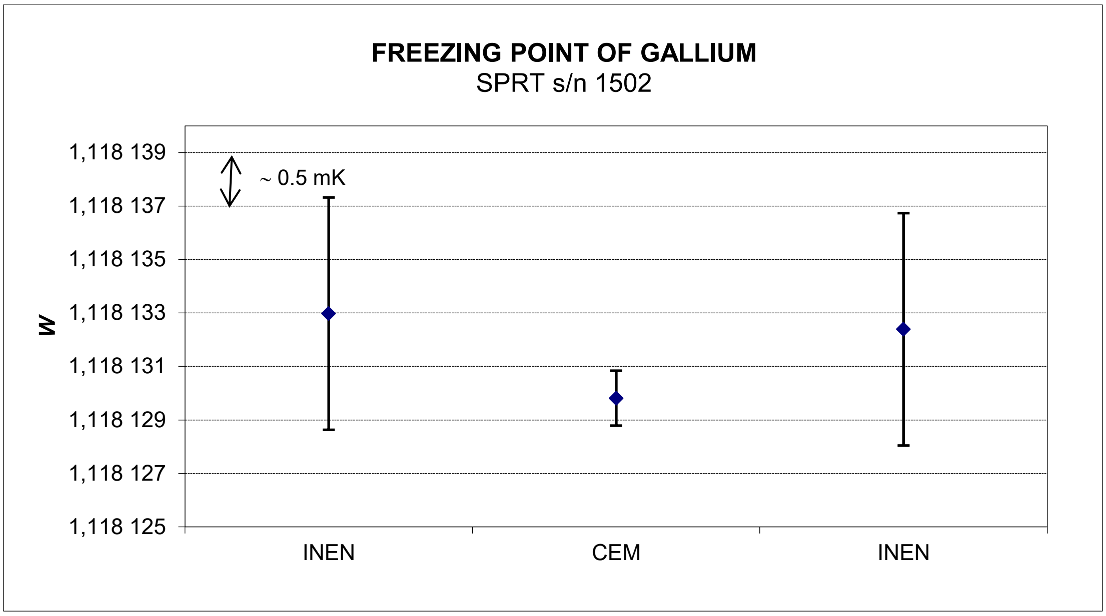

Figure 4. Melting point of gallium results

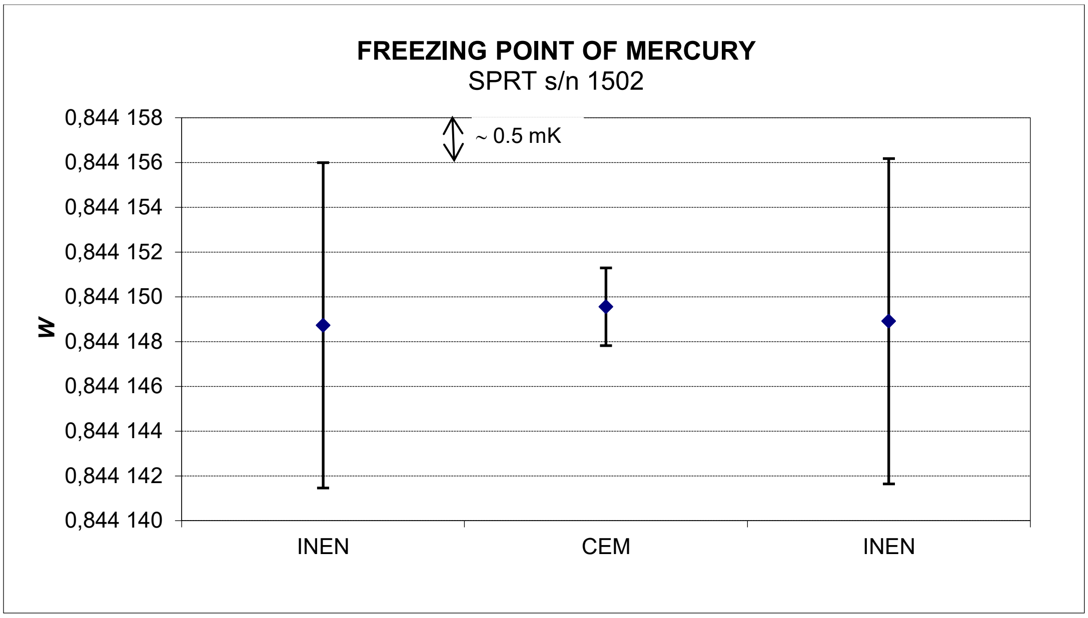

Figure 5. Triple point of mercury results

5. Uncertainties

The participants were requested to supply the uncertainty budget associated with the calibration at the different fixed points. The complete uncertainty budgets can be found in Appendix II.

The combined uncertainties were computed by the root-sum-of-squares of the contributions. In the case of CEM, some of the contributions were estimated using type A method but the coverage factors were very close to 2 due to the large number of the calculated degrees of freedom using the Welch-Satterwhite formula. INEN used the same approach for the calculation of the degrees of freedom.

The expanded uncertainty, *U*, reported for both participants are showed in table 6.

Expanded uncertainty, *U*/mk (coverage probability ~95%)									
Zn		**Sn**		**In**		**Ga**		**Hg**	
CEM	INEN	CEM	INEN	CEM	INEN	CEM	INEN	CEM	INEN
0.90	4.77	0.49	2.57	0.80	2.25	0.26	1.08	0.43	1.79

Table 6. CEM and INEN's uncertainties, all values are expressed in mK.

For each fixed point, the fixed point realization temperature differences between CEM and INEN have been calculated according to:

$$\Delta T_{XX,INEN-CEM} = T_{XX,INEN} - T_{XX,CEM} \quad (1)$$

Where

$T_{XX,INEN}$ is the arithmetic mean of the two results reported by INEN, before and after;

$T_{XX,CEM}$ is the value measured for XX fixed point by CEM; and

XX represents the different fixed points: Zn, Sn, In, Ga and Hg.

The uncertainty of this difference has been calculated based on the uncertainties reported by CEM and INEN and the drift of the travelling standards, calculated based on the W differences measured by INEN at the beginning and end of the comparison and assuming a rectangular probability distribution:

$$u^2(\Delta T_{XX,INEN-CEM}) = u^2(T_{XX,INEN}) + u^2(T_{XX,CEM}) + u^2(T_{XX,drift}) \quad (2)$$

$u(T_{XX,INEN})$, is the uncertainty of INEN. As $T_{XX,INEN}$ is the arithmetic mean of the values measured by this laboratory, before and after, there were be some uncertainty components whose are correlated. The approach followed is that of the EURAMET.T-K9 key comparison where the systematic components were considered fully correlated (and treated accordingly GUM 5.2.2) and random components were considered completely uncorrelated (following in this case the GUM 5.1.1).

$u(T_{XX,CEM})$ is the CEM uncertainty for the XX fixed point.

$u(T_{XX,drift})$ is the uncertainty due to the drift of the travelling standards estimated assuming the differences measured by INEN as the maximum interval of a rectangular distribution.

$$\Delta T_{XX,drift} = T_{XX,INEN\,before} - T_{XX,INEN\,after} \quad (3)$$

$$u(T_{XX,drift}) = \frac{|\Delta T_{XX,drift}|}{2\sqrt{3}} \quad (4)$$

	$\Delta T_{XX,drift}$	$u(T_{drift})$
Fixed point	**[mK]**	**[mK]**
Zn	0.18	0.052
Sn	-0.19	0.054
In	0.18	0.052
Ga	0.15	0.043
Hg	-0.05	0.013

Table 7. Uncertainty due to the drift of the travelling standard

Table 8 presents the differences obtained together with their calculated uncertainties.

	$\Delta T_{XX,INEN-CEM}$	$U(T_{INEN} - T_{CEM})$
Fixed point	**[mK]**	**[mK]**
Zn	-1.12	4.22
Sn	-0.50	2.26
In	0.19	2.35
Ga	0.73	1.12
Hg	-0.18	1.61

Table 8. Differences between INEN and CEM

$$\Delta T_{XX,INEN-CEM} = T_{XX,INEN} - T_{XX,CEM} \quad (5)$$

At the moment of writing this report, the EURAMET.T-K9 Final report was already approved by the CCT and published so the link to the CCT-K9 is available for INEN via CEM. The EURAMET.T-K9 report gives the values (with its uncertainty) for $\Delta T_{XX,CEM-CCT-K9}$. These values, for the different fixed points, are presented in table 9.

	$\Delta T_{XX,CEM-CCT-K9}$	$U(\Delta T_{XX,CEM-CCT-K9})$
Fixed point	**[mK]**	**[mK]**
Zn	1.43	0.98
Sn	1.10	0.96
In	0.48	0.75
Ga	0.07	0.48
Hg	0.65	0.78

Table 9. Differences between CEM and CCT-K9 KCRV

So the values for $\Delta T_{XX,INEN-CCT-K9}$ could be obtained from the following expression:

$$\Delta T_{XX,INEN-CCT-K9} = \Delta T_{XX,INEN-CEM} + \Delta T_{XX,CEM-CCT-K9} \quad (6)$$

To evaluate the uncertainty of the difference between INEN values and the CCT-K9 key comparison reference value (via EURAMET.T-K9) it is necessary to consider the correlations between the CEM uncertainties components from this CCT-K9.2 and EURAMET.T-K9. The approach followed here is similar to that of the section 2.4 of the EURAMET.T-K9 final report and the equations (10), (6), (10) and (12) of that report were used.

In the CEMs uncertainty budget, presented in appendix II.1, it is explicitly declared which components are systematic or random. The systematic components are treated fully correlated and the random components are considered completely uncorrelated.

Therefore the resulting values are presented in the next table 10:

	$\boldsymbol{\Delta T_{XX,CEM-CCT-K9}}$	$\boldsymbol{U(\Delta T_{XX,CEM-CCT-K9})}$	$\boldsymbol{\Delta T_{XX,INEN-CCT-K9}}$	$\boldsymbol{U(\Delta T_{XX,INEN-CCT-K9})}$
Fixed point	**[mK]**	**[mK]**	**[mK]**	**[mK]**
Zn	1.43	0.98	0.31	4.14
Sn	1.10	0.96	0.60	2.37
In	0.48	0.75	0.67	2.21
Ga	0.07	0.48	0.80	1.19
Hg	0.65	0.78	0.47	1.75

Table 10. Differences between INEN and CEM with the CCT-K9 KCRV

Figure 6 presents the results of previous tables showing the degrees of equivalence between both laboratories, INEN and CEM, and the degree of equivalence for the INEN with the CCT.K9 KCRV.

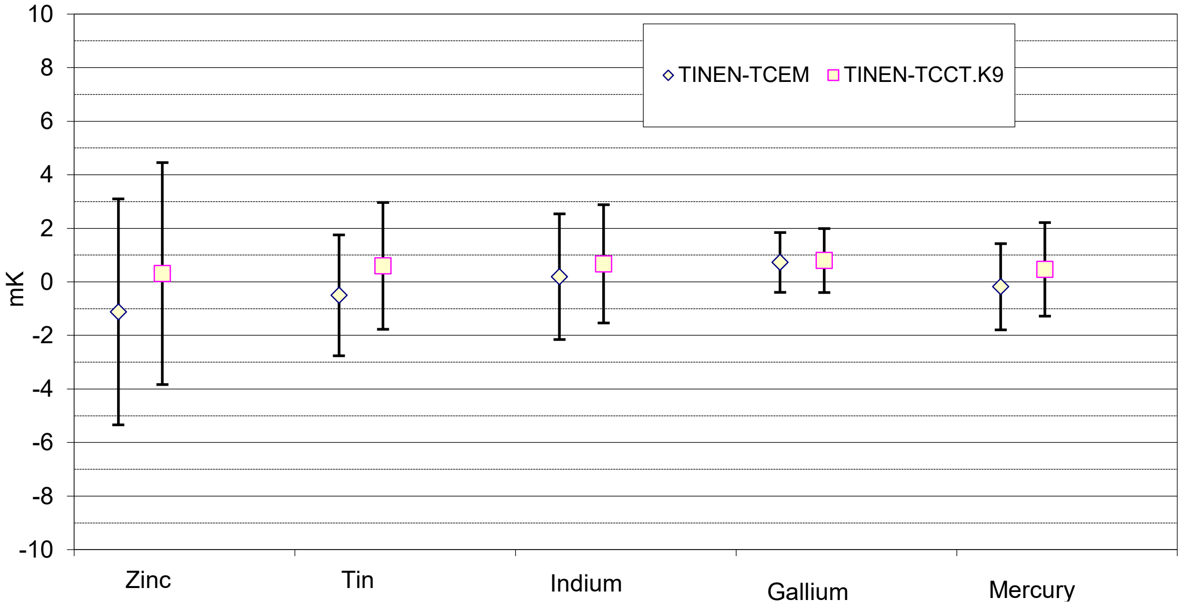

Figure 6. INM degrees of equivalence between INEN and CEM, and between INEN and CCT.K9 KCRV.

6. Conclusion

The results of the comparison have been quite successful. The link for INEN with the CCT.K9 KCRV has been established via EURAMET.T-K9, comparison in which CEM has participated, and could be used to support the INEN CMCs.

7. References

[1] *EURAMET.T-K3.3 Final Report*, 2012, 47 pages Metrologia, 2012, 49, Tech. Suppl., 03007

[2] "*A Bilateral Comparison Between CEM and LACOMET in the Range from 83.8058 K to 933.473 K, Linking to CCT Comparisons*" del Campo, D., García, C. & Solano,. Int J Thermophys 32, 120–126 (2011). https://doi.org/10.1007/s10765-010-0884-8

[3] *"A New Method for the Quantification and Correction of Thermal Effects on the Realization of Fixed Points"* M. Fahr and S. Rudtsch, Int. J. Thermophys., vol. 29, no. 1, pp. 126–138, 2008, doi: 10.1007/s10765-007-0351-3.

[4] *"EURAMET.T-K9 regional key comparison ITS-90 SPRT calibration from the Ar TP to the Zn FP*" Fernando Sparasci et al 2024 Metrologia 61 03005, DOI 10.1088/0026-1394/61/1A/03005

[5] JCGM 100:2008, Evaluation of measurement data — Guide to the expression of uncertainty in measurement,

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8. ANNEX 1. PROTOCOL

Bilateral comparison

CCT-K9.2

Comparison of the realisations of the ITS-90 over the range 234.315 6 K to 933.473 K.

Technical Protocol

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February 22nd, 2022

Version 04

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1. Introduction

The instructions and procedures given here shall be followed by the participants in this comparison. By accepting this technical protocol, the participant laboratories agree to follow the general and technical instructions written in this document and the content in the MRA document *Measurement comparisons in the CIPM MRA* [1].

This comparison is designed as a bilateral comparison. In this bilateral comparison CEM and INEN will calibrate two Standards Platinum Resistance Thermometers (SPRTs) one for the fixed points of Hg, Ga, In, Sn and Zn and another for the fixed point of Al. The method for calibration of the SPRTs will be the fixed point method. The range of temperature covered in this comparison is from the triple point of Hg (234.315 6 K) to the freezing point of Al (993.473 K).

The resulting calibration results will be calculated by NMI personnel and submitted to the pilot laboratory (see section 8).

The final results of the comparison will be determined by the pilot laboratory and presented as degrees of equivalence between the participants. A link to the parent CCT-K9 comparisons will be also provided for the fixed points of Hg, Ga, In, Sn and Zn. For the Al the link will be to the CCT-K4 (via the EURAMET.T-K4) and to the CCT-K3 (via the ARV-K3)[2]

1. Participants

Two National Metrology Institutes will participate in this comparison: Centro Español de Metrología (CEM), España, and Servicio Ecuatoriano de Normalización (INEN). Where CEM is the pilot laboratory. Contact persons with their addresses are given in Table 1.

Table 1. Participating NMIs

Country	NMI	Acronym	Shipping Address	Contact Person
España	Centro Español de Metrología	CEM	C/Alfar, 2. 28760 Tres Cantos, Madrid. España.	Raúl Caballero e-mail: rcaballero@cem.es Phone :_+34 918 074 822 Mª José Martín e-mail: mjmartinh@cem.es Phone: +34 918 074 714
Ecuador	Servicio Ecuatoriano de Normalización	INEN	Autopista General Rumiñahui, Puente Peatonal No.5. Quito. Ecuador	William Paucar wpaucar@normalizacion.gob.ec Phone: +59 3998783062

2. Comparison methodology

Bilateral comparison between CEM (pilot) and INEN with two travellers standards. These two SPRTs 25 Ω will be calibrated by the fixed points method. The measurement sequence is as follows:

1) The SPRTs are calibrated by INEN.

2) The SPRTs are calibrated by CEM

3) The SPRTs are calibrated by INEN.

Detailed timetable of comparison is given in Table 2 below:

Table 2. Timetable of comparison

Measurement sequence	Description	Time Period
1	INEN completes measurements.	Planned December 1st, 2021, to January 15th, 2022
2	Courier picks up SPRTs from INEN and delivers SPRTs to CEM	Planned May 1st to June 15th, 2022
3	CEM completes measurements.	Planned February 1st to March 20th, 2022
4	Courier picks up SPRTs from CEM and delivers SPRTs to INEN	Planned June 15th to July 15th , 2022
5	INEN completes measurements.	Planned July 15th to August 31st, 2022
6	Draft A Report submitted to INEN	Planned by September 15 th, 2022
7	Draft A observations submitted to CEM	Planned by September 30th, 2022
8	Draft B submitted to CCT WG KC	Planned by October 15th, 2022
9	Draft B CCT WG KC observations submitted to CEM	Planned by October 30th, 2022
10	Draft B Report submitted to INEN	Planned by November 15th, 2022
11	Draft B observations submitted to CEM	Planned by November 30th, 2022
12	Draft B submitted to CCT WG KC	Planned by December 15th, 2022
13	Report approved by CCT WG KC. Report publication	Planned by January 15th, 2023

3. Travelling standards

The transfer standards for the bilateral comparison are two SPRTs Pt25 (25 Ω nominal value at 0 ºC) with four terminals, as described in the Table 3. These two standards are supplied by INEN. The SPRT-1 will be measured at Hg, Ga, In, Sn and Zn fixed points. The SPRT-2 will be measured at Al fixed point.

Table 3. Travelling standards for bilateral comparison (CEM-INEN)

Category	**SPRT-1 (25 Ω nominal value at 0 ºC)**	**SPRT-2 (25 Ω nominal value at 0 ºC)**
Manufacturer	FLUKE HART SCIENTIFIC	ISOTECH
Model	5681	909-Q
Serial number:	1502	1773
Length / mm	520	480
Diameter / mm	7	7.5

Transportation of travelling standard

The travelling standards are packed in a carton box of size (100 cm x 20 cm x 20 cm) and total weight of 3 kg. The transport box can be easily opened for customs inspection.

INEN is responsible for delivering SPRT-1 and SPRT-2s between the NMIs by using a courier; INEN will be responsible for the transportation of the SPRT-1 and SPRT-2.

Test upon receipt and final test at INEN

The SPRT-1 and SPRT-2 will be calibrated by INEN and CEM by the fixed point method. This includes initial and a final stability tests of the resistances of the SPRTs at the triple point of water before the transportation to CEM from INEN and after arrival at INEN.

Failure of travelling standard

In case of loss or damage to any of the SPRTs, due to transport, the laboratory will inform to the other one: if they are both SPRT-1 and SPRT-2, the bicomparison will end; if it is only one of the two SPRT-1 and SPRT-2, the comparison will continue with that only SPRT.

Organizational aspects

INEN will cover the costs of sending the thermometers between INEN and CEM by using a courier, including associated taxes. SPRT-1 and SPRT-2 will be covered by an insurance policy, paid by INEN, in case of loss or damage due to transportation.

Communication flows

INEN will inform to CEM of the arrival of the travelling standards.

INEN will inform to CEM the initial and a final stability test of the resistances of the SPRTs at the triple point of water before the transportation to CEM and after arrival at INEN.

INEN will inform to CEM if there are any measurement delays.

After INEN has completed the measurements, INEN will send a measurement report to the pilot laboratory.

CEM will ask to INEN to check their data values in case their shows an apparently anomalous result before issuing the draft A.

CEM will write the Draft A of the final report. This Draft will be sent to INEN. INEN will have two weeks to send their feedback. No reply will be interpreted as a tacit approval.

Draft A will be corrected accordingly up to consensus is reached and afterwards submitted to CCT-WG-KC for review.

CEM will prepare Draft B and it will be submitted to the CCT-WG-KC.

4. Measurement instructions and procedures

Measurand

The measurand for this comparison will be defined through $W(t_{90})$ values, being

$$W(t_{90}) = \frac{R(t_{90})}{R_{TPW}} \quad (1)$$

where $R(t_{90})$: is the value of the SPRT resistance at temperature t_{90} and R_{TPW}: is the resistance value of the SPRT at the triple point of water measured after the measurement at the fixed point.

Preliminary check

When travelling standards are received at INEN, they will be inspected to check if they seem to have external and visible damage due to transportation. In case they seem to be with no damage, the participant laboratory will proceed to measure their R_{TPW} values; INEN will send the corresponding report to the pilot (Annex 1).

Measurements

INEN will inform about their standards and equipment used (Annex 2).

The electrical resistance measurement will be carried out with two currents: 1 mA and √2 mA by using four wires for electrical connections, and extrapolated to 0 mA. If different current values are used it should be notified to the pilot laboratory.

The measurement current used must be such that the generated power does not exceed 250 µW. All the measurements should also be corrected for the hydrostatic head to obtain the resistance values.

SPRT-1 (fixed points Hg, Ga, In, Sn, Zn)

5.4.1. Stabilization procedure

Before starting measurements at the fixed points, the SPRT-1(see table 3 and table 4) should follow a stabilization procedure passing through the following sequence:

1. Measurement at the triple point of water (TPW)
2. Carefully insert the SPRT into a furnace at 480 °C.
3. Anneal the SPRT for two hours at 480 °C
4. Carefully remove the SPRT from the furnace directly to the room environment.
5. Re-determine the value of resistance at the TPW.

If the change of the resistance value at TPW is equivalent to 0.3 mK or greater repeat steps 2 to 5.

If the change of the resistance value at TPW is less than 0.3 mK the calibration can be performed.

5.4.2. Measurement procedure

Measurements at the fixed points should be performed in order of decreasing temperatures alternating with a measurement at the triple point of water:

$$TPW \rightarrow Zn \rightarrow TPW \rightarrow Sn \rightarrow TPW \rightarrow In \rightarrow TPW \rightarrow Ga \rightarrow TPW \rightarrow Hg \rightarrow TPW$$

Both laboratories have to follow their normal practice when realizing the ITS-90. For each fixed point the measurand for this comparison will be defined through $W(t_{90})$ values:

$$W(t_{90}) = \frac{R(t_{90})}{R_{TPW}} \qquad (2)$$

where

$R(t_{90})$: is the value of the SPRT resistance at temperature t_{90}

R_{TPW}: is the resistance value of the SPRT at the triple point of water, measured after the measurement at the fixed point.

$R(t_{90})$ and R_{TPW} should have been corrected for self-heating, hydrostatic head and if any the pressure effect. At least 2 different phase transitions (2 freezing for Zn, Sn, In, 2 melting for Ga, 2 triple points for Hg) will be performed. The different values will be delivered together with the calculated mean.

For each fixed point cell used in the comparison, it has to be determined (using the circulating SPRT) the change of phase transition temperature, dT, versus immersion depth, dh. These measurements will be reported in a graph where the theoretical dT/dh curve, using the hydrostatic pressure coefficients (mK/m of liquid) given in the ITS-90 text, and the measured dT/dh curve will be plotted.

SPRT-2 (fixed point of Al)

In order to avoid any damage of the SPRT-2 (see table 3) it has to be cleaned carefully prior any insertion at a temperature above 500 ºC. Nitric acid, acetone or ethanol can be used to perform the cleaning following a several times rinsing with distilled water.

5.5.1. Stabilization procedure

Before starting measurements at the fixed point of aluminum, the SPRT should follow a stabilization procedure passing through the following sequence:

1. Measurement at the triple point of water (TPW)
2. Insert slowly the transfer SPRT into an annealing furnace which is preheated to 500 °C, and then increase the temperature of the annealing furnace to 675 °C over approximately 1 hour. Maintain the temperature at that point for 30 minutes, and then reduce it to 500 °C over approximately 1.5 to 4 hours.
3. When the temperature has reached 500 °C, remove slowly the SPRT from the furnace directly to the room environment.
4. Re-determine the value of resistance at the TPW.
    - If the change of the resistance value at TPW is equivalent to 0.5 mK or greater repeat steps 2 to 4.
    - If the change of the resistance value at TPW is less than 0.5 mK the calibration can be performed.

5.5.2. Measurement procedure

Measurement at the fixed point should be performed with measurements at the triple point of water just before and other after of that of the Al:

$$TPW \rightarrow Al \rightarrow TPW$$

Both laboratories (CEM and INEN) have to follow their normal practice when realizing the ITS-90. For each fixed point the measurand for this comparison will be defined through $W(t_{90})$ values:

$$W(t_{90}) = \frac{R(t_{90})}{R_{TPW}} \qquad (3)$$

where

$R(t_{90})$: is the value of the SPRT resistance at temperature t_{90}

R_{TPW}: is the resistance value of the SPRT at the triple point of water, measured after the measurement at the fixed point.

$R(t_{90})$ and R_{TPW} should have been corrected for self-heating, hydrostatic head and if any the pressure effect. At least 2 different phase transitions (2 freezing for Al) will be performed. The different values will be delivered together with the calculated mean.

The sequence of measurement for each plateau in the fixed point of the aluminum should be as follows:

1. The SPRT must be preheated in an annealing furnace which is preheated to 500 °C, and then the temperature is increased up to a value between 600 °C and 660 °C over approximately 1 hour. The transfer SPRT should be removed then from the annealing

furnace, and inserted into the well of the aluminum freezing point cell and calibrated in the stable plateau of the freezing curve of aluminum.

2. Once the thermometer has been measured at the Al fixed point, the SPRT should be removed and inserted into the annealing furnace whose temperature is maintained at a temperature between 600 °C and 660 °C, annealed for 30 minutes and then cooled down to 450 °C within approximately 1.5 to 4 hours.
3. When the temperature of the annealing furnace (along with the SPRT) has been dropped to 450 °C, wait for approximately 30 minutes and then remove slowly the SPRT from the furnace directly to the room environment.
4. After the SPRT has cooled down to room temperature, measure its resistance at the TPW (RTPW).

For each fixed point cell used in the comparison, it has to be determined (using the circulating SPRT) the change of phase transition temperature, dT, versus immersion depth, dh. These measurements will be reported in a graph where the theoretical dT/dh curve, using the hydrostatic pressure coefficients (mK/m of liquid) given in the ITS-90 text, and the measured dT/dh curve will be plotted.

5. Evaluation of the results of the thermometers

Once the SPRT-1 and SPRT-2 are back to INEN it is necessary to check their stability. To do so, INEN will carry out the ensuing actions:

- Stabilization of the 25 Ω SPRT used for the fixed points of Hg, Ga, In, Sn , Zn as in 5.4.1
- Measurement *W* at the freezing points of zinc, tin and indium, the melting point of gallium and the triple point for mercury, following 5.4.2.
- Stabilization of the 25 Ω SPRT used for the fixed points of Al as in 5.5.1
- Measurement of the *W* at the freezing points of aluminum, following 5.5.2.

The result of these measurements, with their associated uncertainties, will be reported to CEM to be included in the final report.

6. Uncertainties

Uncertainty analysis according to the "Guide to the Expression of Uncertainty in Measurement", JCGM 100:2008, GUM 1995 with minor corrections, First edition September 2008 must been performed for both laboratories. The uncertainty analysis must include the following terms and other items that the participating laboratory wants to include:

- Phase transition repeatability
- Chemical impurities and isotopic composition for the TPW
- Hydrostatic-head errors
- Bridge measurement errors:
    - effects of changes in reference resistors
    - non-linearity of bridge
    - quadrature effects in ac measurements
    - resolution
    - repeatability of the measurements at each fixed point
- Uncertainty propagated from the TPW
- Heat flux-immersion errors

- Errors in gas pressure
- Errors in the choice of freezing point value from plateau of the freezing curve
- SPRT internal Insulation leakage (if any)
- High-temperature insulation degradation of the transfer SPRT (only in the case of the Al freezing point)

7. Report of Results

The reports with the results provided by the participants will include the information requested in annex 3 and annex 4.

For each fixed point cell used in the comparison, a graph where is plotted the measured and theoretical dT/dh curve using the hydrostatic pressure coefficients (mK/m of liquid) given in the ITS-90 text will be also provided, together with examples of their phase transition curves.

INEN shall fill out the annex 3 and annex 4 spreadsheet and send it to CEM.

Method of analysis of the results

CEM will collect the results of INEN. The results to be reported are temperature differences and measurement uncertainty.

For each fixed point the fixed-point realization temperature differences between CEM and the participant laboratory (INEN) (before and after CEM measurements) will be calculated according to:

$$\Delta T_{XX,INEN-CEM} = \left(T_{XX,INEN} - T_{XX,CEM}\right) \cdot s \qquad (4)$$

Where XX represents the different fixed points: Al, Zn, Sn, In, Ga and Hg and s is the first derivative of the inverse reference function.

The uncertainty of this difference will be calculated based on the uncertainties reported by CEM and INEN and the drift of the travelling standards, calculated based on the measurements performed by INEN at the beginning and end of the comparison:

$$u^2\left(\Delta T_{XX,INEN-CEM}\right) = u^2\left(T_{XX,INEN}\right) + u^2\left(T_{XX,CEM}\right) + u^2\left(T_{XX,drift}\right) \qquad (5)$$

The $\Delta T_{XX,}$ values will be used to link the results of this comparison to the CCT-K9 through the CEM differences with respect the CCT-K9 KCRV obtained in the EURAMET.T-K9 when their results are available according to the following equation:

$$\Delta T_{XX,INEN-EURAMET.T-K9} = \Delta T_{XX,INEN-CEM} - \Delta T_{XX,CCT-K9-CEM} \qquad (6)$$

With the corresponding uncertainty:

$$u^2\left(\Delta T_{XX,INEN-CCT-K9}\right) = u^2\left(\Delta T_{XX,INEN-CEM}\right) + u^2\left(\Delta T_{XX,CCT-K9-CEM}\right) \qquad (7)$$

Where:

$$\Delta T_{XX,CCT-K9-CEM} = \Delta T_{XX,CCT-K9} - \Delta T_{XX,CEM} \qquad (8)$$

Being $T_{XX,EURAMET.T-K9}$ the EURAMET.T-K9 reference value for the XX fixed point.

The ΔT_{Al} values for the Al will be used to link the results of this comparison to the CCT-K3 (via the EURAMET.T-K4 and the ARV-K3) and to the CCT-K4 (via EURAMET.T-K4 and the KCRV) as detailed in [2].

Comparison report

CEM will ask to INEN to check their data values in case their show an apparently anomalous result before issuing the draft A.

CEM will write the Draft A of the final report. This Draft will be sent to INEN. INEN will have two weeks to send their feedback. No reply will be interpreted as a tacit approval.

Draft A will be corrected accordingly up to consensus is reached and afterwards submitted to CCT-WG-KC for review, becoming Draft B.

If the CCT-WG-KC has any observation on this Draft B, CEM will prepare a new Draft B attending these observations and will it send it to INEN for consensus. INEN will have two weeks to send their feedback. No reply will be interpreted as a tacit approval.

Once this Draft B is corrected accordingly up to consensus is reached and the observations of the CCT-WG-KC attended, this Draft will be submitted to the CCT-WG-KC for its approval as a final report.

Reference

1. MRA Document (January 2021): *Measurement comparisons in the CIPM MRA*. CIPM MRA-G-11D05, Version 1.1- https://www.bipm.org/documents/20126/43742162/CIPM-MRA-G-11.pdf/9fe6fb9a-500c-9995-2911-342f8126226c?version=1.9&download=

2. D. del Campo, C. García and A. Solano, "Bilateral Comparison Between CEM and LACOMET in the Range from 83.8058 K to 933.473 K, Linking to CTT Comparisons", *Int. J. Thermophys (2011), 32: 120-126.*

Annex 1

Format of Reception

In order to have information about the comparison development and, if it is necessary to take adequate corrective actions, the participant laboratory must send by e-mail to the pilot laboratory this format after the first measurement at 0.01 °C.

Laboratory and contact person: ____

____

Travelers standards arrived on the day: ________________

Are there some damage signals due to transport? Yes/Not_________

Description, in case of damage: ____

____

Measurement at 0.01 ºC:

- Resistance (1 mA): _____________ Ω
- Resistance ($\sqrt{2}$ mA): _____________ Ω
- Resistance (0 mA): _____________ Ω
- Immersion correction: _____________ Ω
- Expanded Uncertainty: _____________ °C

The participant laboratories could also declare the first (upon receipt) and last resistance values at the TPW. This might be helpful for keeping track of the SPRTs in the case there are drifts.

Comments:

Annex 2

Instrumentation Details

Bi-Lateral comparison CEM-INEN

Laboratory name	
Bridge	
Manufactured	
Type	
AC or DC	
If AC, give Frequency	
If DC, give Period of reversal	
Normal measurement current	
Self-heating current	
Evaluation of linearity of resistance	
Bridge (yes or not)	
If yes, How?	
Reference resistor	
Manufactured / type	
Reference resistor temperature control (yes or not)	
If yes, How?	
TPW Cell	
Manufactured / model	
Serial number	
Is it a primary reference? (if not explain its traceability)	
Immersion depth of middle of the SPRT sensitive element/cm	
How are mantles maintained (ice, bath, …)	
Al Cell	
Manufactured / model	
Serial number	
Is it a primary reference? (if not explain its traceability)	
Closed cell or open	
Nominal purity	
Immersion depth of middle of the SPRT sensitive element/cm	
Al Furnace	
Type (1 zone, 3 zones, heat pipe, …)	
Typical duration of the melting / freezing plateaux	
Zn Cell	
Manufactured / model	
Is it a primary reference? (if not explain its traceability)	
Immersion depth of middle of the SPRT sensitive element/cm	
Closed cell or open	
Nominal purity	
Immersion depth of middle of the SPRT sensitive element/cm	
Zn Furnace	
Type (1 zone, 3 zones, heat pipe, …)	
Typical duration of the melting / freezing plateaux	
Sn Cell	
Manufactured / model	
Is it a primary reference? (if not explain its traceability)	
Immersion depth of middle of the SPRT sensitive element/cm	
Closed cell or open	
Nominal purity	
Immersion depth of middle of the SPRT sensitive element/cm	
Sn Furnace	
Type (1 zone, 3 zones, heat pipe, …)	

Typical duration of the melting / freezing plateaux	
In Cell	
Manufactured / model	
Is it a primary reference? (if not explain its traceability)	
Immersion depth of middle of the SPRT sensitive element/cm	
Closed cell or open	
Nominal purity	
Immersion depth of middle of the SPRT sensitive element/cm	
In Furnace	
Type (1 zone, 3 zones, heat pipe, …)	
Typical duration of the melting / freezing plateaux	
Ga Cell	
Manufactured / model	
Is it a primary reference? (if not explain its traceability)	
Immersion depth of middle of the SPRT sensitive element/cm	
Closed cell or open	
Nominal purity	
Immersion depth of middle of the SPRT sensitive element/cm	
Ga Furnace	
Type (1 zone, 3 zones, heat pipe, …)	
Typical duration of the melting / freezing plateaux	
Hg Cell	
Manufactured / model	
Is it a primary reference? (if not explain its traceability)	
Immersion depth of middle of the SPRT sensitive element/cm	
Closed cell or open	
Nominal purity	
Immersion depth of middle of the SPRT sensitive element/cm	
Hg cryostat	
Type (cryocooler, bath,…)	
Typical duration of the melting plateaux	

Annex 3
RESULTS

Bi-Lateral comparison CEM-INEN

Laboratory name:

SPRT Manufacturer:
Model:
serial/number:

Date	Point	R measured Ω	Selft heating Ω	Hydrostatic Ω	Pressure Ω	R corrected Ω	W
	Zn						
	TPW						
	Zn						
	TPW						
	Average of W for Zn						
	Sn						
	TPW						
	Sn						
	TPW						
	Average of W for Sn						
	In						
	TPW						
	In						
	TPW						
	Average of W for In						
	Ga						
	TPW						
	Ga						
	TPW						
	Average of W for Ga						
	Hg						
	TPW						
	Hg						
	TPW						
	Average of W for Hg						

RESULTS

Bi-Lateral comparison CEM-INEN

Laboratory name:

SPRT Manufacturer:
Model:
serial/number:

Date	Point	R measured Ω	Selft heating Ω	Hydrostatic Ω	Pressure Ω	R corrected Ω	W
	Al						
	TPW						
	Al						
	TPW						
	Average of W for Al						

Annex 4

Uncertainty analysis

Bi-lateral comparison CEM-INEN

Laboratory name:
Fixed point:
Ser.-No. of SPRT:

Quantity	Components	Standard uncertainty $U_{(Q_i)}$	Degrees of freedom components evaluated by a	Sensitivity coefficient	Uncertainty contribution u_i in mK
Combined uncertainty					
Effective degrees of freedom					
Expanded uncertainty					

Annex 5

DOCUMENT REVISION HISTORY

Version	Date	Description	Change
01	June 15th, 2021	Initial	No apply.
02	September 28th, 2021	Corrections following comments of the reviewers of the CCT-WG-KC before the approval of the protocol	Attending comments of the reviewers of the CCT-WG-KC
03	October 5th, 2021	Incorporation of CESMEC to the comparison	New participant. Two bi-lateral comparisons in parallel
04	February 22nd, 2022	Withdraw of CESMEC and update of the timetable	Withdraw all the mentions to CESMEC and update of the timetable of section 3

Appendix II. Detailed uncertainty tables

The following tables contain detailed uncertainties supplied by INEN, along with the uncertainties determined at the pilot laboratory (CEM).

The columns named “Type” provide information to indicate whether an uncertainty component is related to either a random (R) or a systematic effect (S).

II.1. CEM uncertainty

Uncertainty component	Zn	Sn	In	Ga	Hg	Type
	mK					
Phase Transition Realization Repeatability	0.007	0.090	0.091	0.097	0.163	R
Bridge (repeatability, non-linearity, AC quadrature)	0.095	0.039	0.058	0.026	0.033	S
Reference resistor stability	0.002	0.001	0.002	0.002	0.002	S
Chemical impurities	0.326	0.171	0.270	0.039	0.057	S
Hydrostatic-head	0.031	0.025	0.029	0.014	0.082	S
Propagated TPW	0.149	0.097	0.170	0.056	0.042	S
SPRT self-heating	0.045	0.044	0.046	0.032	0.026	R
Heat Flux	0.029	0.058	0.100	0.012	0.014	S
Insulation leakage	0.000	0.000	0.002	0.002	0.002	S
SPRT Pt Oxydation	0.242	0.080	0.179	0.030	0.074	S
Gas pressure	0.025	0.019	0.028	0.001	0.003	S
Combined uncertainty	0.45	0.25	0.40	0.13	0.21	
Expanded uncertainty (*k*=2) (coverage probability ~95 %)	0.90	0.49	0,80	0.26	0.43	

Table 11. Annex 2. CEM’s uncertainty budget for Zn, Sn, In, Ga and Hg, all values are expressed in mK.

II.2. INEN uncertainty

Laboratory name: SERVICIO ECUATORIANO DE NORMALIZACIÓN INEN - TEMPERATURE LABORATORY.

Fixed point: Freezing point of zinc

Ser.-No. of SPRT: 1502

Quantity Q_i	**Components**	**Uncertainty type**	**Uncertainty contribution u_i in mK**
X(PTA)	Repeatibility of readings	Random	0.013
$C_{0.01°C/1}$	Relative temperature variation standard resistor	Systematic	0.11
$C_{0.01°C/2}$	Relative drift standard resistor	Systematic	0.14
$C_{0.01°C/3}$	Standard resistor calibration	Systematic	0.15
$C_{0.01°C/4}$	Uncertainty linked of hydrostatic pressure correction	Systematic	0.0021
$C_{0.01°C/5}$	Uncertainty linked with self-heating correction	Systematic	0.0069
$C_{0.01°C/6}$	Uncertainty linked with gas pressure	Systematic	0.15
$C_{0.01°C/7}$	Repeatibility of temperature realized by cell	Random	0.24
$C_{0.01°C/8}$	Uncertainty linked perturbing heat exchanges	Systematic	0.040
$C_{0.01°C/9}$	Uncertainty linked lack of linearity of the bridge	Systematic	0.040
$C_{0.01°C/10}$	Uncertainty linked SPRT internal insulation leakage insulation	Systematic	0.099
$C_{0.01°C/11}$	Uncertainty linked change of the X(TPW) of SPRT between before and after the FP	Random	0.099
$C_{0.01°C/12}$	Uncertainty linked with purity	Systematic	0.10
$C_{0.01°C/13}$	Absolute value of direct comparison difference	Systematic	0.050
$C_{0.01°C/14}$	Direct comparison measurement	Systematic	0.070
X(Zn)	Repeatibility of readings	Random	0.030
$CZn_{419.527°C/1}$	Relative temperature variation standard resistor	Systematic	0.13
$CZn_{419.527°C/2}$	Relative drift standard resistor	Systematic	0.16
$CZn_{419.527°C/3}$	Standard resistor calibration	Systematic	0.17
$CZn_{419.527°C/4}$	Uncertainty linked of hydrostatic pressure correction	Systematic	0.0078
$CZn_{419.527°C/5}$	Uncertainty linked with self-heating correction	Systematic	0.013
$CZn_{419.527°C/6}$	Uncertainty linked with gas pressure	Systematic	1.7
$CZn_{419.527°C/7}$	Repeatibility of temperature realized by cell	Random	1.3
$CZn_{419.527°C/8}$	Uncertainty linked perturbing heat exchanges	Systematic	0.052
$CZn_{419.527°C/9}$	Uncertainty linked lack of linearity of the bridge	Systematic	0.045
$CZn_{419.527°C/10}$	Uncertainty linked with purity and isotopic composition	Systematic	0.71
$CZn_{419.527°C/11}$	Absolute value of direct comparison difference	Systematic	0.41
$CZn_{419.527°C/12}$	Direct comparison measurement	Systematic	0.20
Combined standard uncertainty, u_c			2.4
***k*, coverage factor, estimated according the effective degrees of freedom. The assigned expanded uncertainty corresponds to a coverage probability of approximately 95 %.**			2.01
Expanded uncertainty, $U = k \cdot u_c$			**4.77**

Table 12. INEN's uncertainty budget for zinc, all values are expressed in mK.

Laboratory name: SERVICIO ECUATORIANO DE NORMALIZACIÓN INEN - TEMPERATURE LABORATORY.

Fixed point: Freezing point of tin

Ser.-No. of SPRT: 1502

Quantity Q_i	**Components**	**Uncertainty type**	**Uncertainty contribution u_i in mK**
X(PTA)	Repeatability of readings	Random	0.013
$C_{0.01°C/1}$	Relative temperature variation standard resistor	Systematic	0.11
$C_{0.01°C/2}$	Relative drift standard resistor	Systematic	0.14
$C_{0.01°C/3}$	Standard resistor calibration	Systematic	0.15
$C_{0.01°C/4}$	Uncertainty linked of hydrostatic pressure correction	Systematic	0.0021
$C_{0.01°C/5}$	Uncertainty linked with self-heating correction	Systematic	0.0069
$C_{0.01°C/6}$	Uncertainty linked with gas pressure	Systematic	0.15
$C_{0.01°C/7}$	Repeatibility of temperature realized by cell	Random	0.24
$C_{0.01°C/8}$	Uncertainty linked perturbing heat exchanges	Systematic	0.040
$C_{0.01°C/9}$	Uncertainty linked lack of linearity of the bridge	Systematic	0.040
$C_{0.01°C/10}$	Uncertainty linked SPRT internal insulation leakage insulation	Systematic	0.099
$C_{0.01°C/11}$	Uncertainty linked change of the X(TPW) of SPRT between before and after the FP	Random	0.099
$C_{0.01°C/12}$	Uncertainty linked with purity	Systematic	0.10
$C_{0.01°C/13}$	Absolute value of direct comparison difference	Systematic	0.050
$C_{0.01°C/14}$	Direct comparison measurement	Systematic	0.070
X(Sn)	Repeatibility of readings	Random	0.024
$CSn_{231.928°C/1}$	Relative temperature variation standard resistor	Systematic	0.12
$CSn_{231.928°C/2}$	Relative drift standard resistor	Systematic	0.15
$CSn_{231.928°C/3}$	Standard resistor calibration	Systematic	0.16
$CSn_{231.928°C/4}$	Uncertainty linked of hydrostatic pressure correction	Systematic	0.0064
$CSn_{231.928°C/5}$	Uncertainty linked with self-heating correction	Systematic	0.011
$CSn_{231.928°C/6}$	Uncertainty linked with gas pressure	Systematic	0.70
$CSn_{231.928°C/7}$	Repeatibility of temperature realized by cell	Random	0.74
$CSn_{231.928°C/8}$	Uncertainty linked perturbing heat exchanges	Systematic	0.034
$CSn_{231.928°C/9}$	Uncertainty linked lack of linearity of the bridge	Systematic	0.043
$CSn_{231.928°C/10}$	Uncertainty linked with purity and isotopic composition	Systematic	0.52
$CSn_{231.928°C/11}$	Absolute value of direct comparison difference	Systematic	0.20
$CSn_{231.928°C/12}$	Direct comparison measurement	Systematic	0.15
		Combined standard uncertainty, u_c	1.3
		***k*, coverage factor, estimated according the effective degrees of freedom. The assigned expanded uncertainty corresponds to a coverage probability of approximately 95 %.**	2.02
		Expanded uncertainty, $U = k \cdot u_c$	**2.57**

Table 13. INEN's uncertainty budget for tinc, all values are expressed in mK.

Laboratory name: SERVICIO ECUATORIANO DE NORMALIZACIÓN INEN - TEMPERATURE LABORATORY.

Fixed point: Freezing point of indium

Ser.-No. of SPRT: 1502

Quantity Q_i	Components	Uncertainty type	Uncertainty contribution u_i in mK
$X(PTA)$	Repeatibility of readings	Random	0.016
$C_{0.01°C/1}$	Relative temperature variation standard resistor	Systematic	0.11
$C_{0.01°C/2}$	Relative drift standard resistor	Systematic	0.14
$C_{0.01°C/3}$	Standard resistor calibration	Systematic	0.15
$C_{0.01°C/4}$	Uncertainty linked of hydrostatic pressure correction	Systematic	0.0021
$C_{0.01°C/5}$	Uncertainty linked with self-heating correction	Systematic	0.0069
$C_{0.01°C/6}$	Uncertainty linked with gas pressure	Systematic	0.15
$C_{0.01°C/7}$	Repeatibility of temperature realized by cell	Random	0.24
$C_{0.01°C/8}$	Uncertainty linked perturbing heat exchanges	Systematic	0.040
$C_{0.01°C/9}$	Uncertainty linked lack of linearity of the bridge	Systematic	0.040
$C_{0.01°C/10}$	Uncertainty linked SPRT internal insulation leakage insulation	Systematic	0.099
$C_{0.01°C/11}$	Uncertainty linked change of the X(TPW) of SPRT between before and after the FP	Random	0.099
$C_{0.01°C/12}$	Uncertainty linked with purity	Systematic	0.10
$C_{0.01°C/13}$	Absolute value of direct comparison difference	Systematic	0.050
$C_{0.01°C/14}$	Direct comparison measurement	Systematic	0.070
$X(In)$	Repeatibility of readings	Random	0.023
$CIn_{156.5985°C/1}$	Relative temperature variation standard resistor	Systematic	0.12
$CIn_{156.5985°C/2}$	Relative drift standard resistor	Systematic	0.14
$CIn_{156.5985°C/3}$	Standard resistor calibration	Systematic	0.16
$CIn_{156.5985°C/4}$	Uncertainty linked of hydrostatic pressure correction	Systematic	0.0095
$CIn_{156.5985°C/5}$	Uncertainty linked with self-heating correction	Systematic	0.011
$CIn_{156.5985°C/6}$	Uncertainty linked with gas pressure	Systematic	0.63
$CIn_{156.5985°C/7}$	Repeatibility of temperature realized by cell	Random	0.27
$CIn_{156.5985°C/8}$	Uncertainty linked perturbing heat exchanges	Systematic	0.10
$CIn_{156.5985°C/9}$	Uncertainty linked lack of linearity of the bridge	Systematic	0.042
$CIn_{156.5985°C/10}$	Uncertainty linked with purity and isotopic composition	Systematic	0.50
$CIn_{156.5985°C/11}$	Absolute value of direct comparison difference	Systematic	0.27
$CIn_{156.5985°C/12}$	Direct comparison measurement	Systematic	0.47
		Combined standard uncertainty, u_c	1.1
		k, coverage factor, estimated according the effective degrees of freedom. The assigned expanded uncertainty corresponds to a coverage probability of approximately 95 %.	2.01
		Expanded uncertainty, $U = k \cdot u_c$	**2.25**

Table 14. INEN's uncertainty budget for indium, all values are expressed in mK

Laboratory name: SERVICIO ECUATORIANO DE NORMALIZACIÓN INEN - TEMPERATURE LABORATORY.

Fixed point: Melting point of gallium

Ser.-No. of SPRT: 1502

Quantity Q_i	**Components**	**Uncertainty type**	**Uncertainty contribution u_i in mK**
X(PTA)	Repeatibility of readings	Random	0.014
$C_{0.01°C/1}$	Relative temperature variation standard resistor	Systematic	0.11
$C_{0.01°C/2}$	Relative drift standard resistor	Systematic	0.14
$C_{0.01°C/3}$	Standard resistor calibration	Systematic	0.15
$C_{0.01°C/4}$	Uncertainty linked of hydrostatic pressure correction	Systematic	0.0021
$C_{0.01°C/5}$	Uncertainty linked with self-heating correction	Systematic	0.0069
$C_{0.01°C/6}$	Uncertainty linked with gas pressure	Systematic	0.15
$C_{0.01°C/7}$	Repeatibility of temperature realized by cell	Random	0.24
$C_{0.01°C/8}$	Uncertainty linked perturbing heat exchanges	Systematic	0.040
$C_{0.01°C/9}$	Uncertainty linked lack of linearity of the bridge	Systematic	0.040
$C_{0.01°C/10}$	Uncertainty linked SPRT internal insulation leakage insulation	Systematic	0.099
$C_{0.01°C/11}$	Uncertainty linked change of the X(TPW) of SPRT between before and after the FP	Random	0.099
$C_{0.01°C/12}$	Uncertainty linked with purity	Systematic	0.10
$C_{0.01°C/13}$	Absolute value of direct comparison difference	Systematic	0.050
$C_{0.01°C/14}$	Direct comparison measurement	Systematic	0.070
X(Ga)	Repeatibility of readings	Random	0.014
$CGa_{29.7646°C/1}$	Relative temperature variation standard resistor	Systematic	0.12
$CGa_{29.7646°C/2}$	Relative drift standard resistor	Systematic	0.14
$CGa_{29.7646°C/3}$	Standard resistor calibration	Systematic	0.15
$CGa_{29.7646°C/4}$	Uncertainty linked of hydrostatic pressure correction	Systematic	0.0035
$CGa_{29.7646°C/5}$	Uncertainty linked with self-heating correction	Systematic	0.008
$CGa_{29.7646°C/6}$	Uncertainty linked with gas pressure	Systematic	0.01
$CGa_{29.7646°C/7}$	Repeatibility of temperature realized by cell	Random	0.05
$CGa_{29.7646°C/8}$	Uncertainty linked perturbing heat exchanges	Systematic	0.02
$CGa_{29.7646°C/9}$	Uncertainty linked lack of linearity of the bridge	Systematic	0.040
$CGa_{29.7646°C/10}$	Uncertainty linked with purity and isotopic composition	Systematic	0.20
$CGa_{29.7646°C/11}$	Absolute value of direct comparison difference	Systematic	0.10
$CGa_{29.7646°C/12}$	Direct comparison measurement	Systematic	0.050
Combined standard uncertainty, u_c			0.53
k, coverage factor, estimated according the effective degrees of freedom. The assigned expanded uncertainty corresponds to a coverage probability of approximately 95 %.			2.03
Expanded uncertainty, $U = k \cdot u_c$			**1.08**

Table 15. INEN's uncertainty budget for gallium, all values are expressed in mK

Laboratory name: SERVICIO ECUATORIANO DE NORMALIZACIÓN INEN - TEMPERATURE LABORATORY.

Fixed point: Triple point of mercury

Ser.-No. of SPRT: 1502

Quantity Q_i	**Components**	**Uncertainty type**	**Uncertainty contribution u_i in mK**
$X(PTA)$	Repeatibility of readings	Random	0.014
$C_{0.01°C/1}$	Relative temperature variation standard resistor	Systematic	0.11
$C_{0.01°C/2}$	Relative drift standard resistor	Systematic	0.14
$C_{0.01°C/3}$	Standard resistor calibration	Systematic	0.15
$C_{0.01°C/4}$	Uncertainty linked of hydrostatic pressure correction	Systematic	0.0021
$C_{0.01°C/5}$	Uncertainty linked with self-heating correction	Systematic	0.0069
$C_{0.01°C/6}$	Uncertainty linked with gas pressure	Systematic	0.15
$C_{0.01°C/7}$	Repeatibility of temperature realized by cell	Random	0.24
$C_{0.01°C/8}$	Uncertainty linked perturbing heat exchanges	Systematic	0.040
$C_{0.01°C/9}$	Uncertainty linked lack of linearity of the bridge	Systematic	0.040
$C_{0.01°C/10}$	Uncertainty linked SPRT internal insulation leakage insulation	Systematic	0.099
$C_{0.01°C/11}$	Uncertainty linked change of the X(TPW) of SPRT between before and after the FP	Random	0.099
$C_{0.01°C/12}$	Uncertainty linked with purity	Systematic	0.10
$C_{0.01°C/13}$	Absolute value of direct comparison difference	Systematic	0.050
$C_{0.01°C/14}$	Direct comparison measurement	Systematic	0.070
$X(Hg)$	Repeatibility of readings	Random	0.012
$CHg_{-38.8344°C/1}$	Relative temperature variation standard resistor	Systematic	0.11
$CHg_{-38.8344°C/2}$	Relative drift standard resistor	Systematic	0.14
$CHg_{-38.8344°C/3}$	Standard resistor calibration	Systematic	0.15
$CHg_{-38.8344°C/4}$	Uncertainty linked of hydrostatic pressure correction	Systematic	0.020
$CHg_{-38.8344°C/5}$	Uncertainty linked with self-heating correction	Systematic	0.0067
$CHg_{-38.8344°C/6}$	Uncertainty linked with gas pressure	Systematic	0.010
$CHg_{-38.8344°C/7}$	Repeatibility of temperature realized by cell	Random	0.46
$CHg_{-38.8344°C/8}$	Uncertainty linked perturbing heat exchanges	Systematic	0.0085
$CHg_{-38.8344°C/9}$	Uncertainty linked lack of linearity of the bridge	Systematic	0.039
$CHg_{-38.8344°C/10}$	Uncertainty linked with purity and isotopic composition	Systematic	0.25
$CHg_{-38.8344°C/11}$	Absolute value of direct comparison difference	Systematic	0.51
$CHg_{-38.8344°C/12}$	Direct comparison measurement	Systematic	0.040
Combined standard uncertainty, u_c			0.87
k, coverage factor, estimated according the effective degrees of freedom. The assigned expanded uncertainty corresponds to a coverage probability of approximately 95 %.			2.0
Expanded uncertainty, $U = k \cdot u_c$			**1.79**

Table 16. INEN's uncertainty budget for mercury, all values are expressed in mK

9. Appendix III. IMMERSION PROFILES

The immersion profiles included in the following plots correspond to the results obtained with the travelling thermometer in the fixed point cells used by INEN and CEM in this comparison. Figures 7 to 11 correspond to the INEN measurements and figures 12 to 16 to the CEM measurements. In the INEN figures the straight line corresponds to the ITS-90 theoretical slope. In the CEM graphs are also include the corrected values. This corrected values are the differences between the measured values are those of the theoretical slope predicted by the ITS-90.

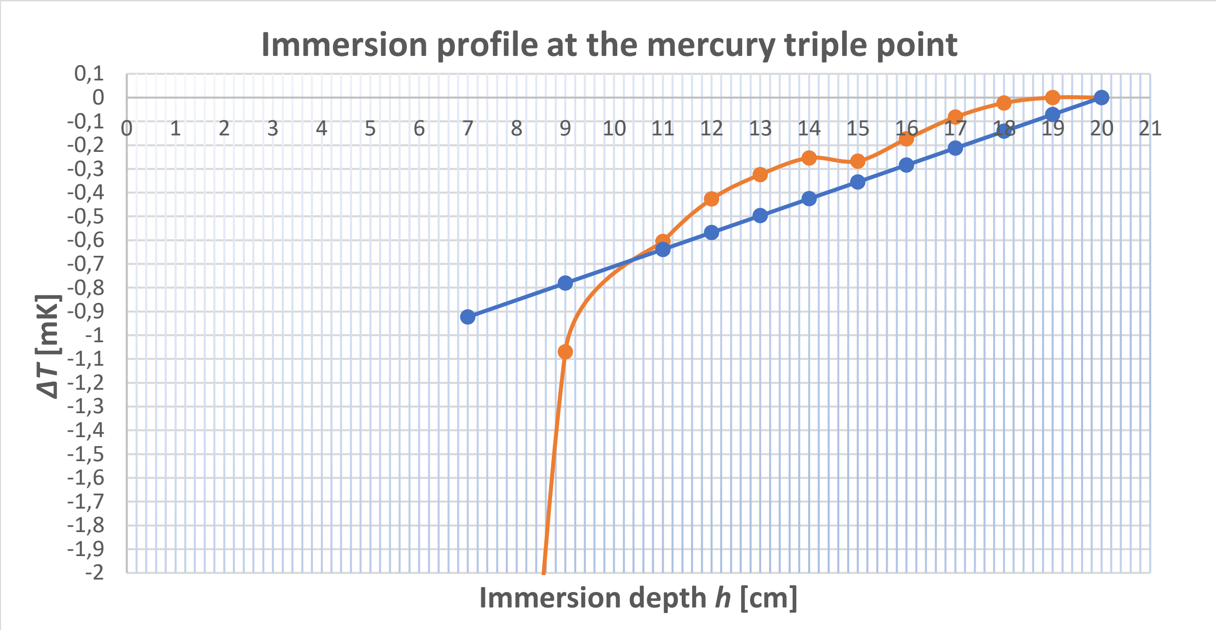

Figure 7. INEN immersion profile at the mercury triple point.

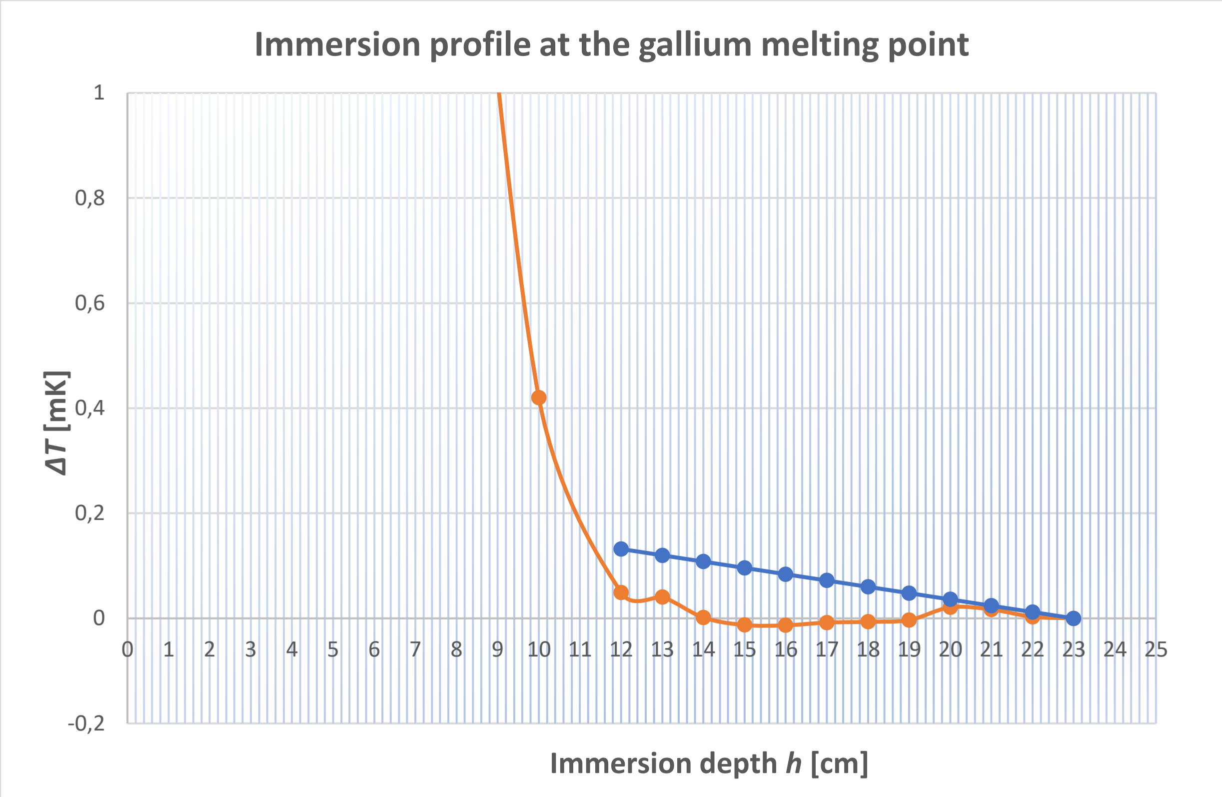

Figure 8. INEN immersion profile at the gallium melting point.

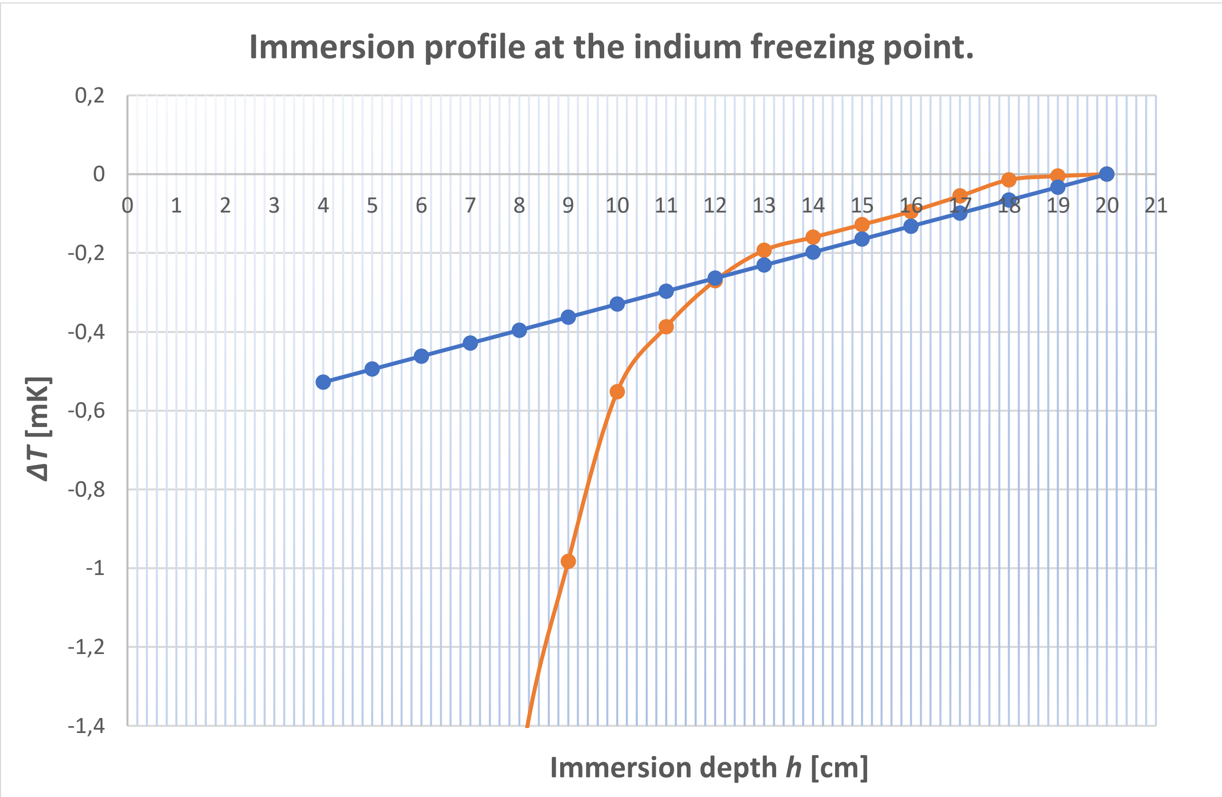

Figure 9. INEN immersion profile at the indium freezing point.

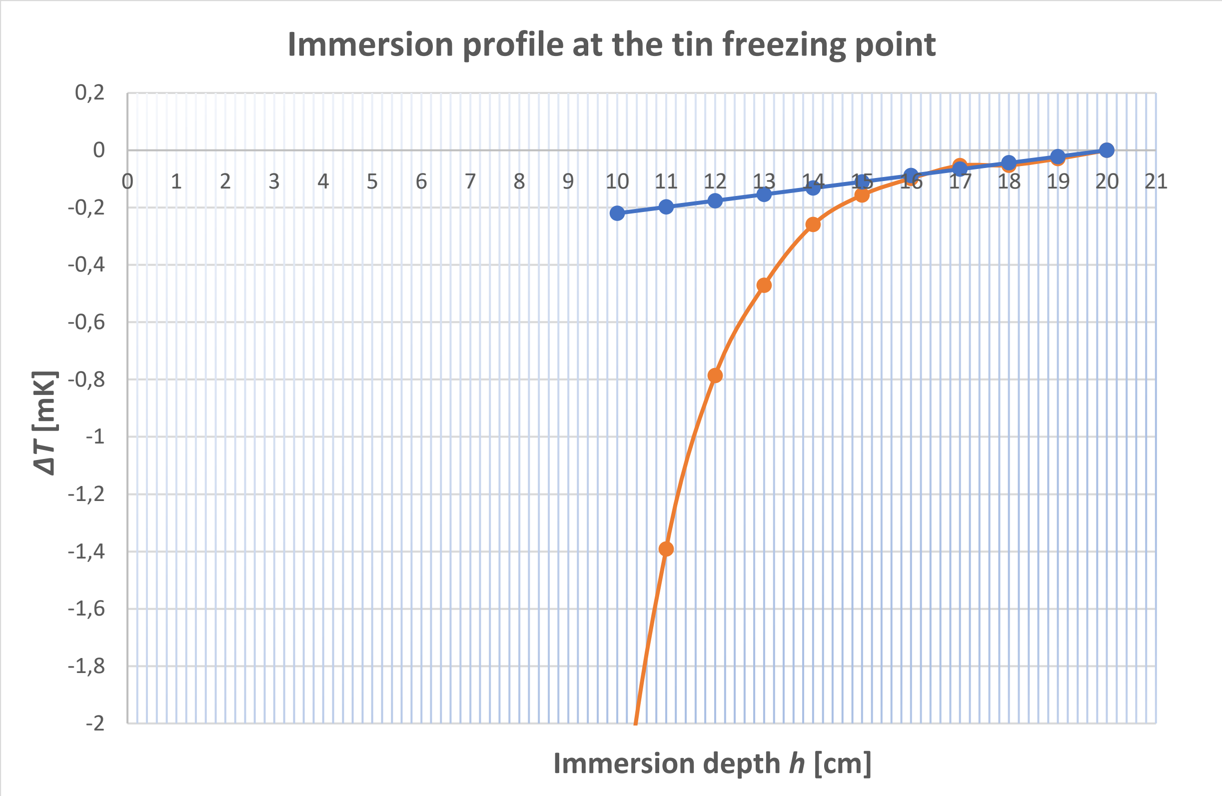

Figure 10. INEN immersion profile at the tin freezing point.

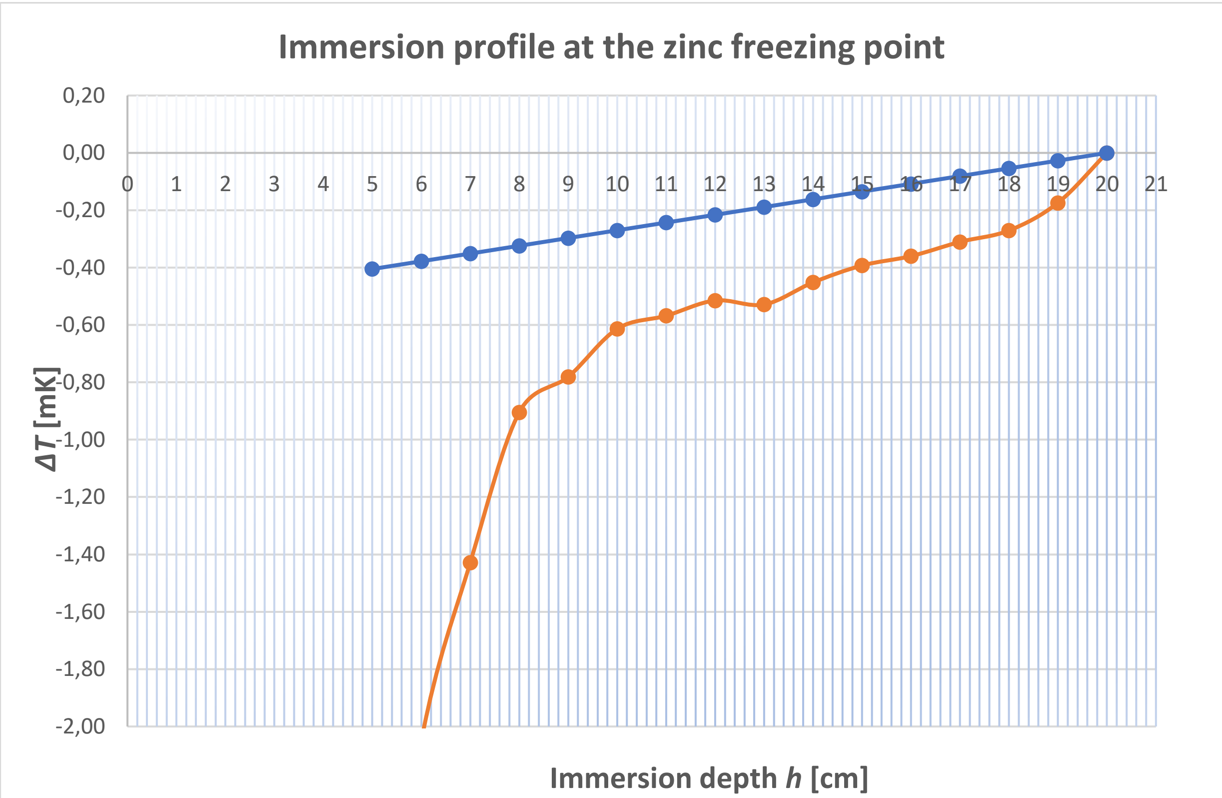

Figure 11. INEN immersion profile at the zinc freezing point.

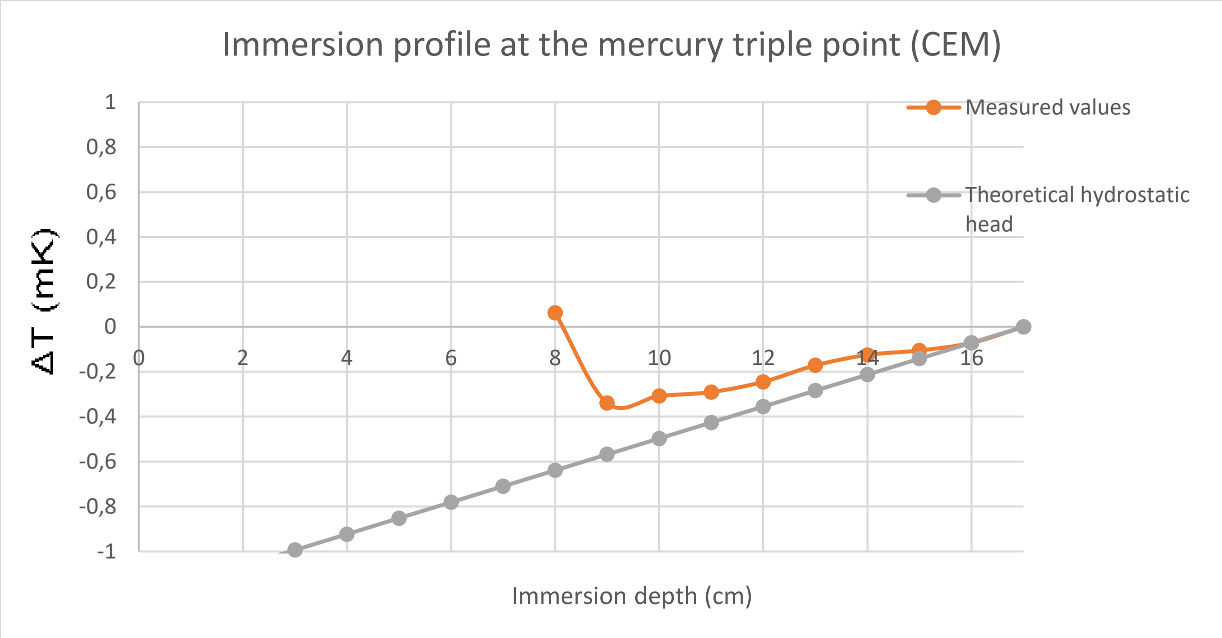

Figure 12. CEM immersion profile at the mercury triple point.

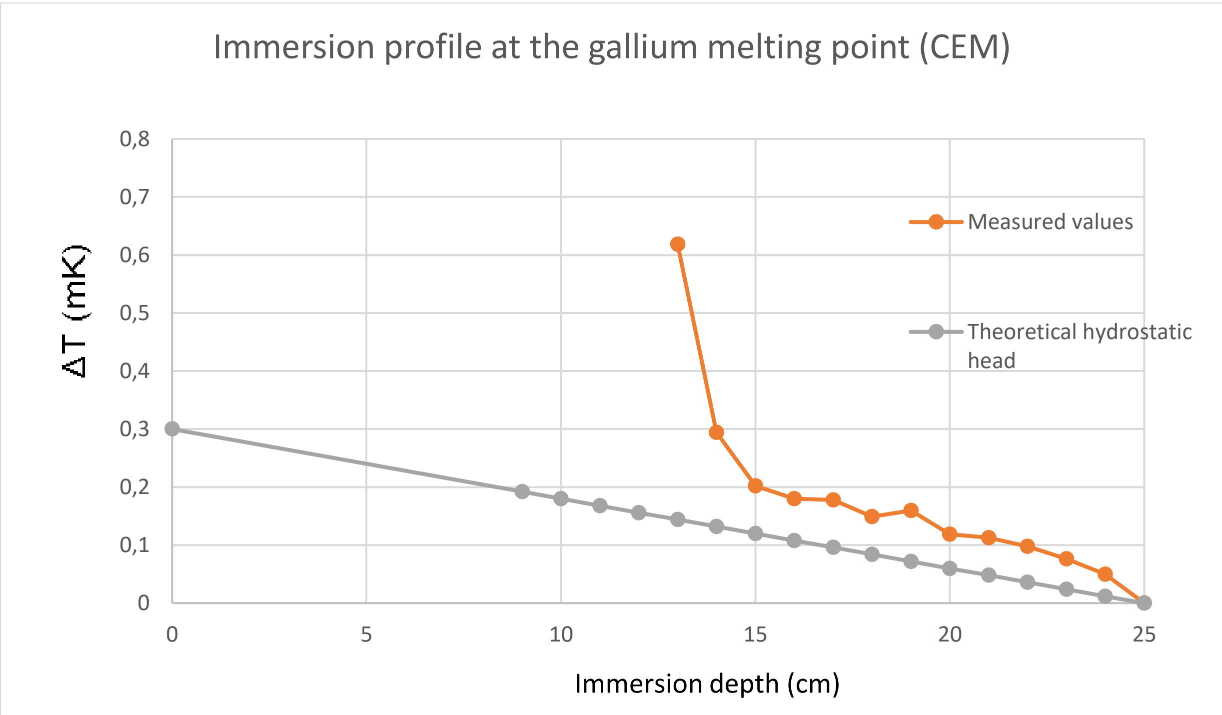

Figure 13. CEM immersion profile at the gallium melting point.

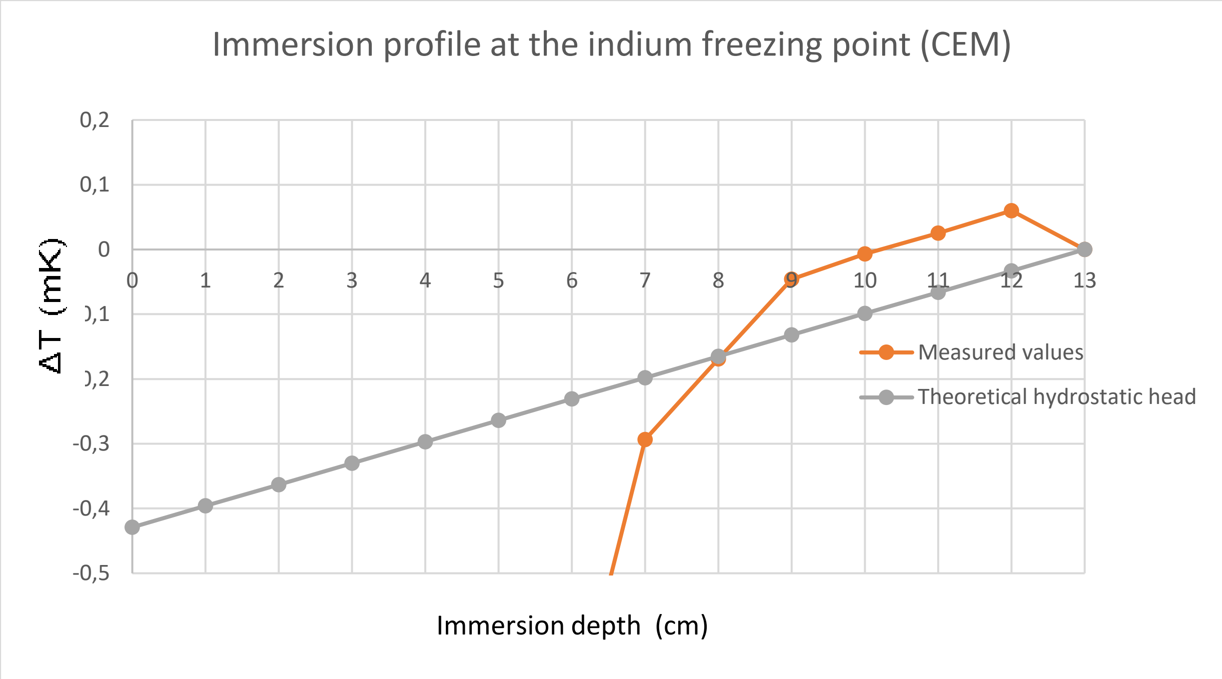

Figure 14. CEM immersion profile at the indium freezing point.

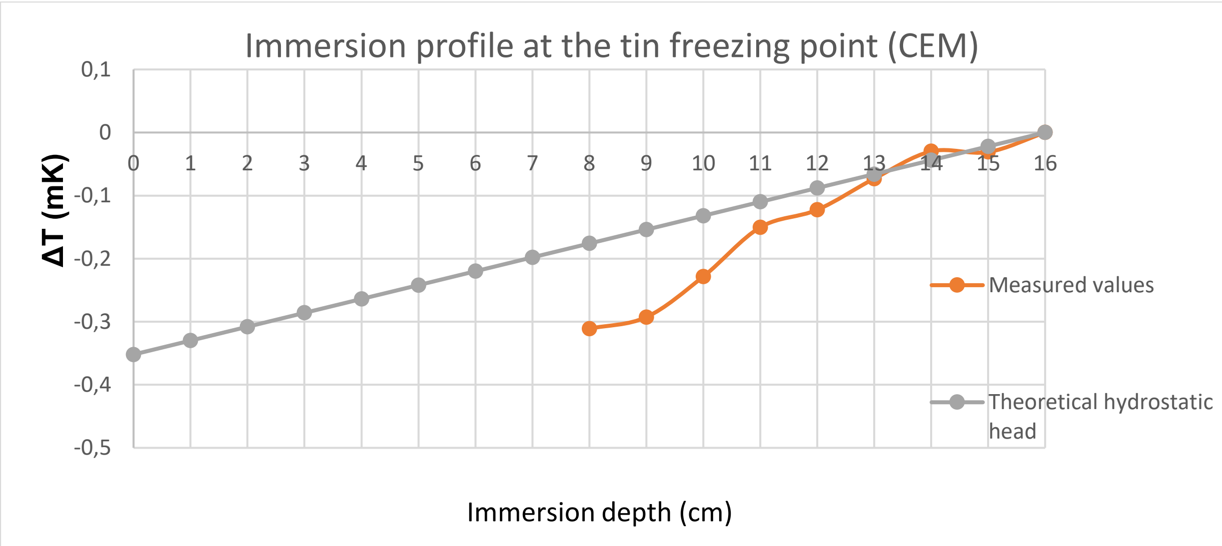

Figure 15. CEM immersion profile at the tin freezing point.

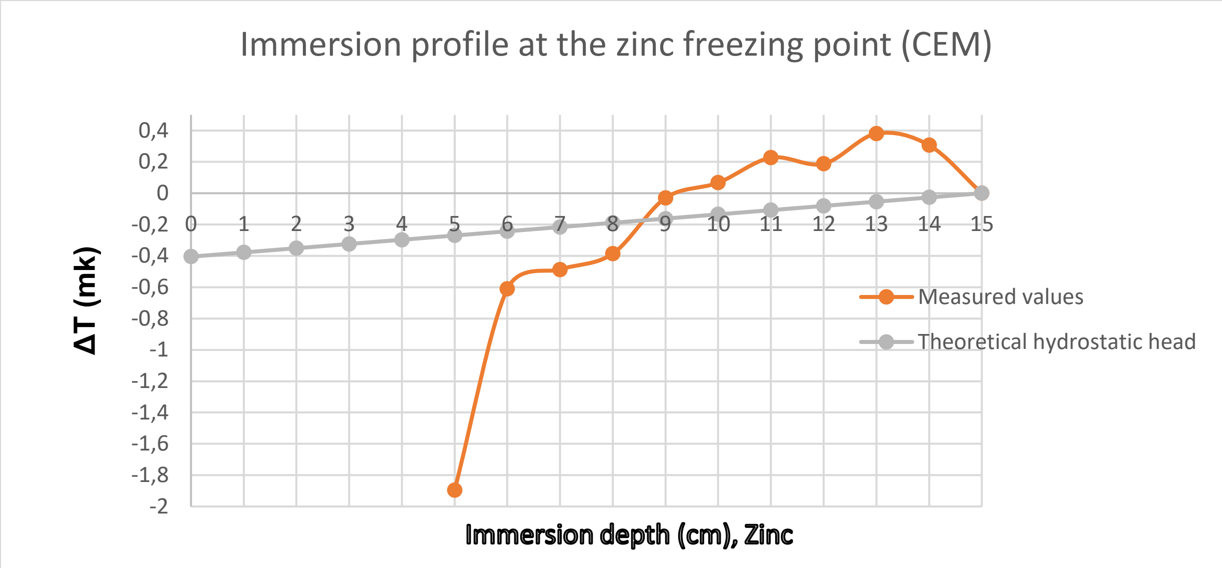

Figure 16. CEM immersion profile at the zinc freezing point.

Appendix IV. PHASE TRANSITION CURVES

The graphs included below correspond to examples of the phase transition curves of the fixed point cells used by INEN and CEM during the comparison in the same or similar furnaces (in the case of CEM) used during the comparison. The results are not obtained with the travelling SPRTs.

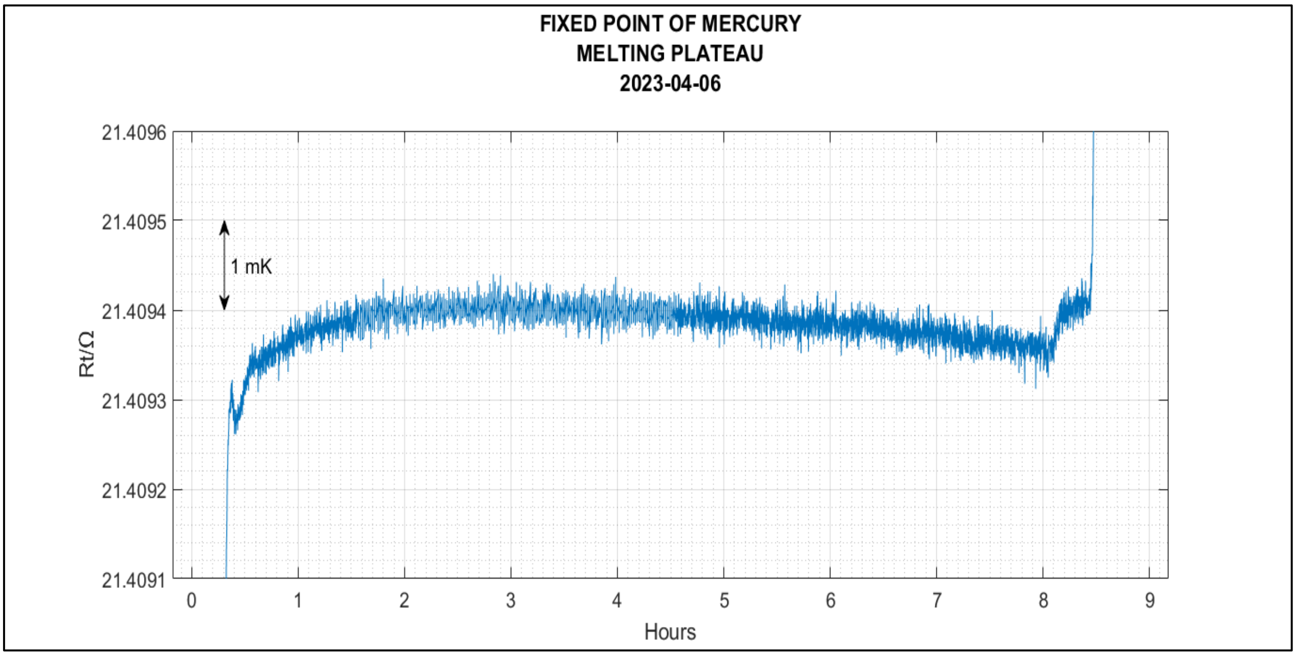

Figure 17. INEN mercury phase transition curve

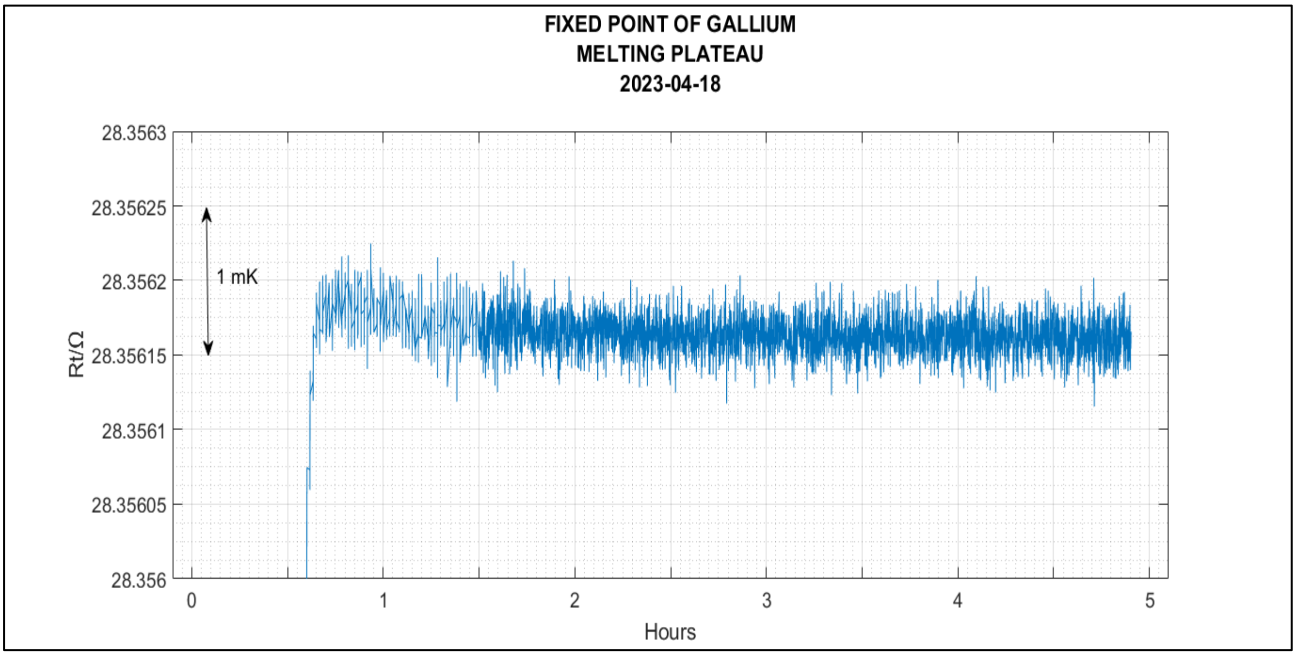

Figure 18. INEN gallium phase transition curve

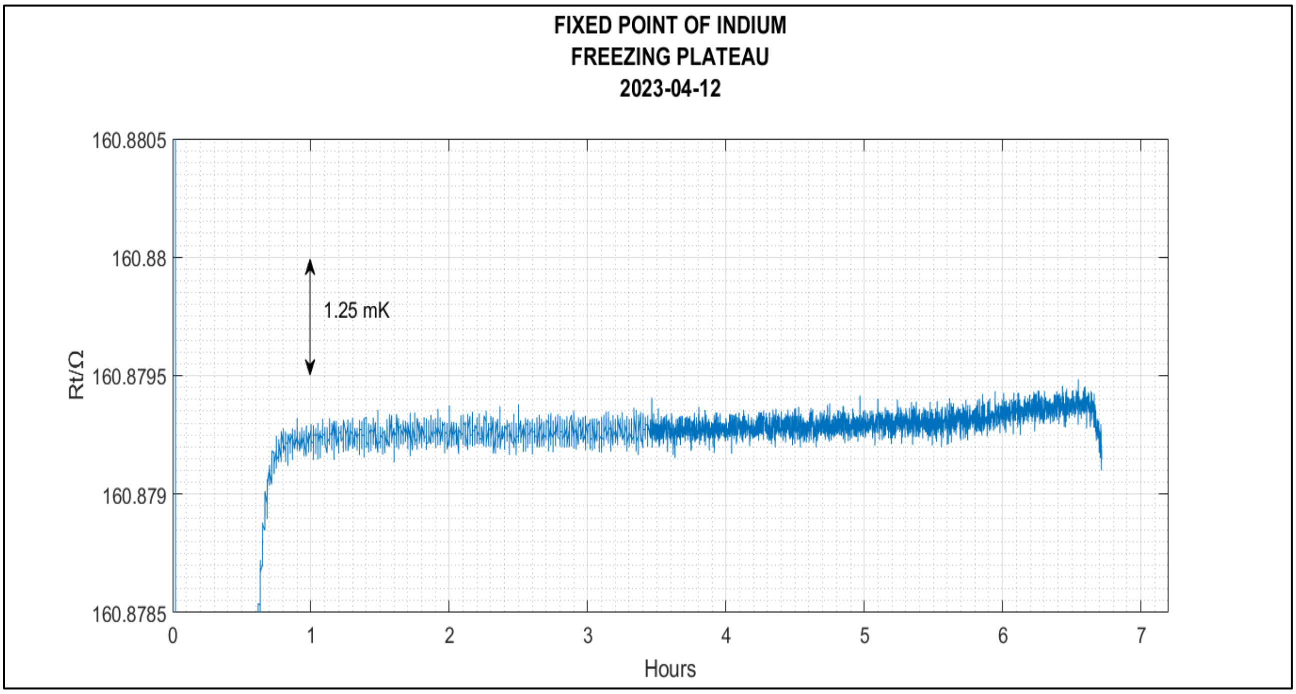

Figure 19. INEN indium phase transition curve

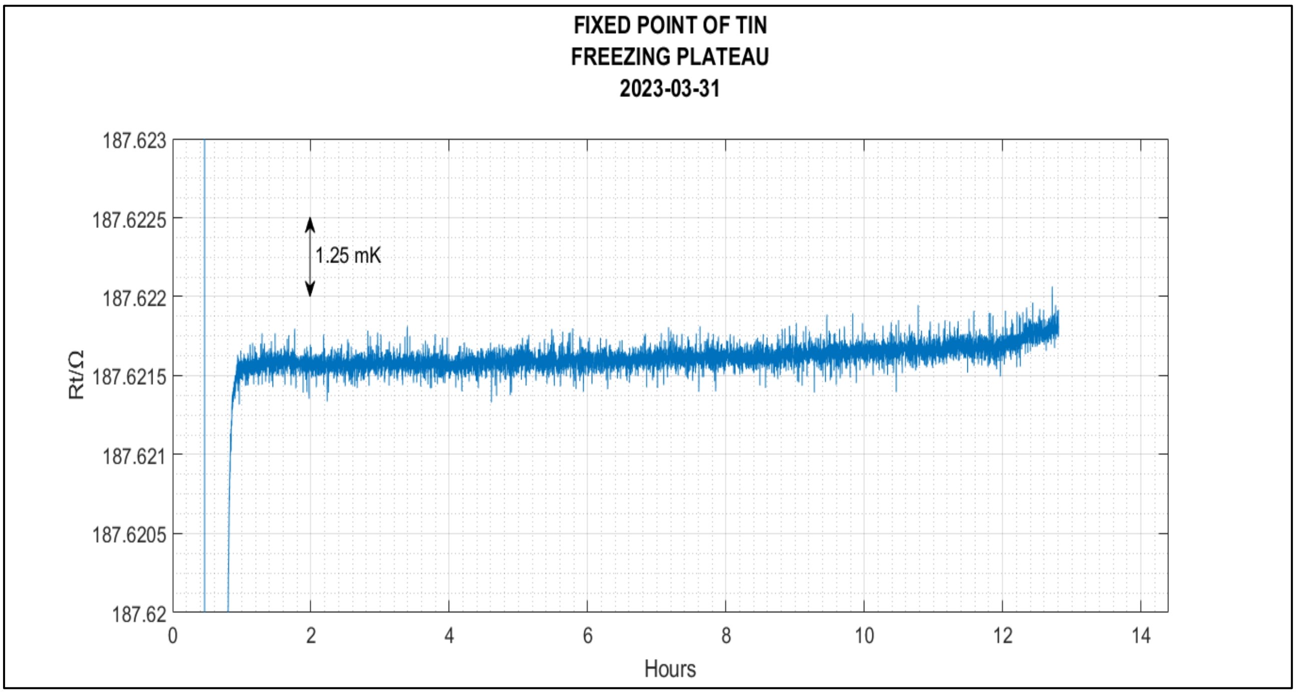

Figure 20. INEN tin phase transition curves

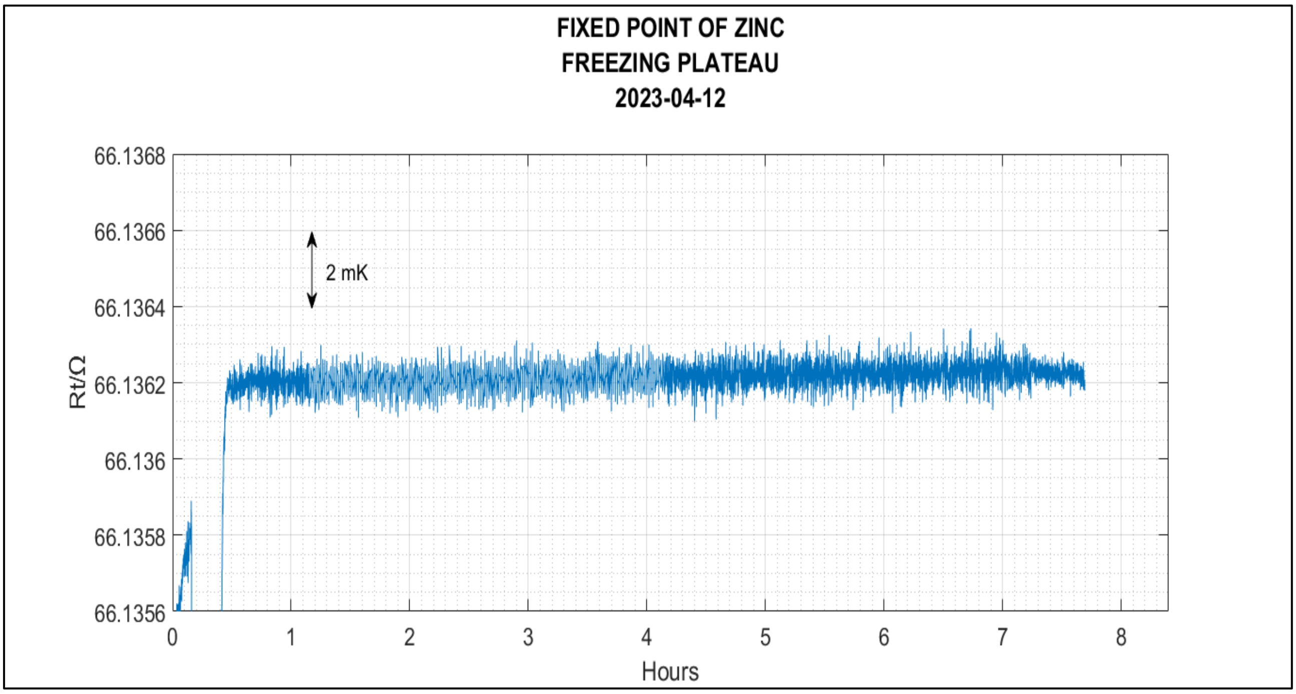

Figure 21. INEN zinc phase transition curve

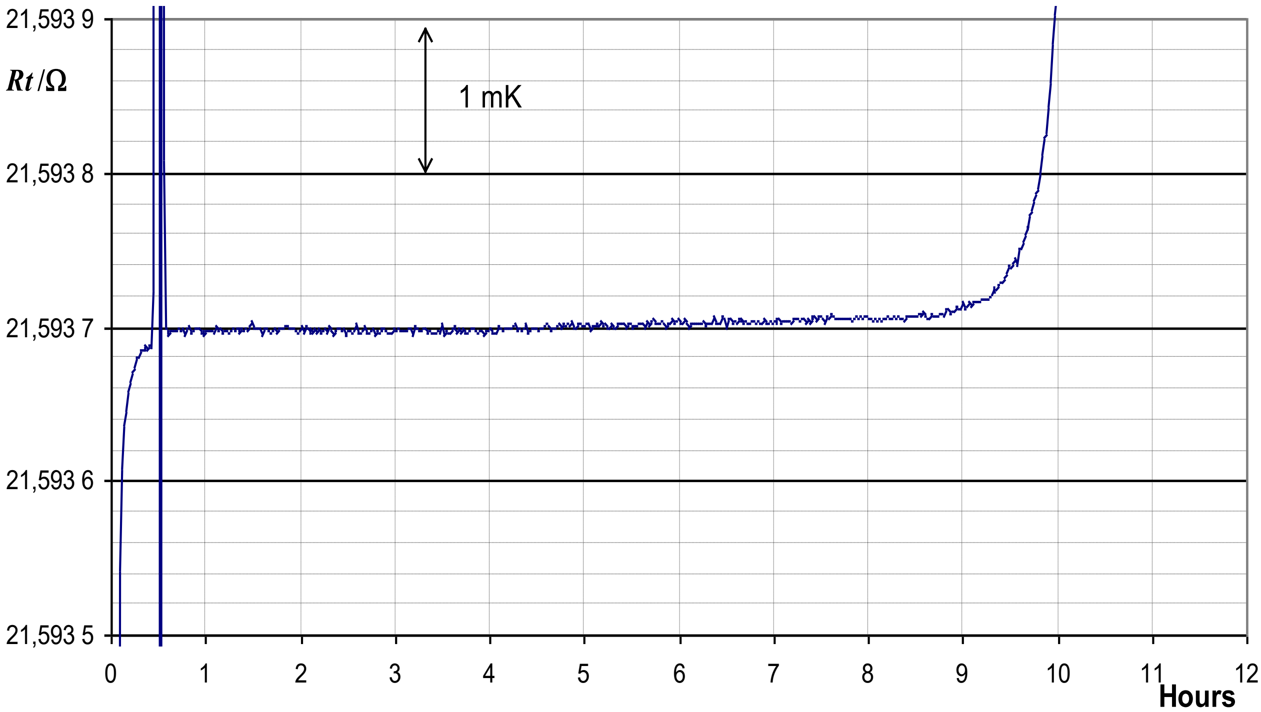

Figure 22. CEM mercury phase transition curve

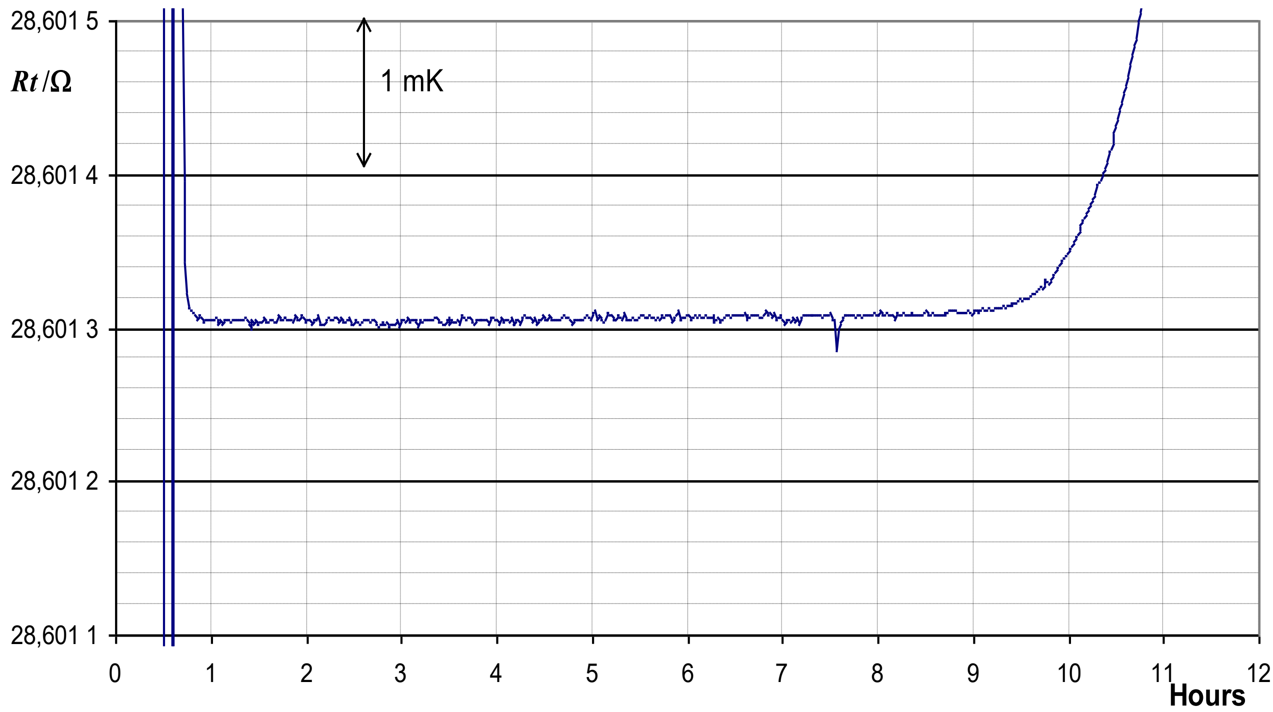

Figure 23. CEM gallium phase transition curve

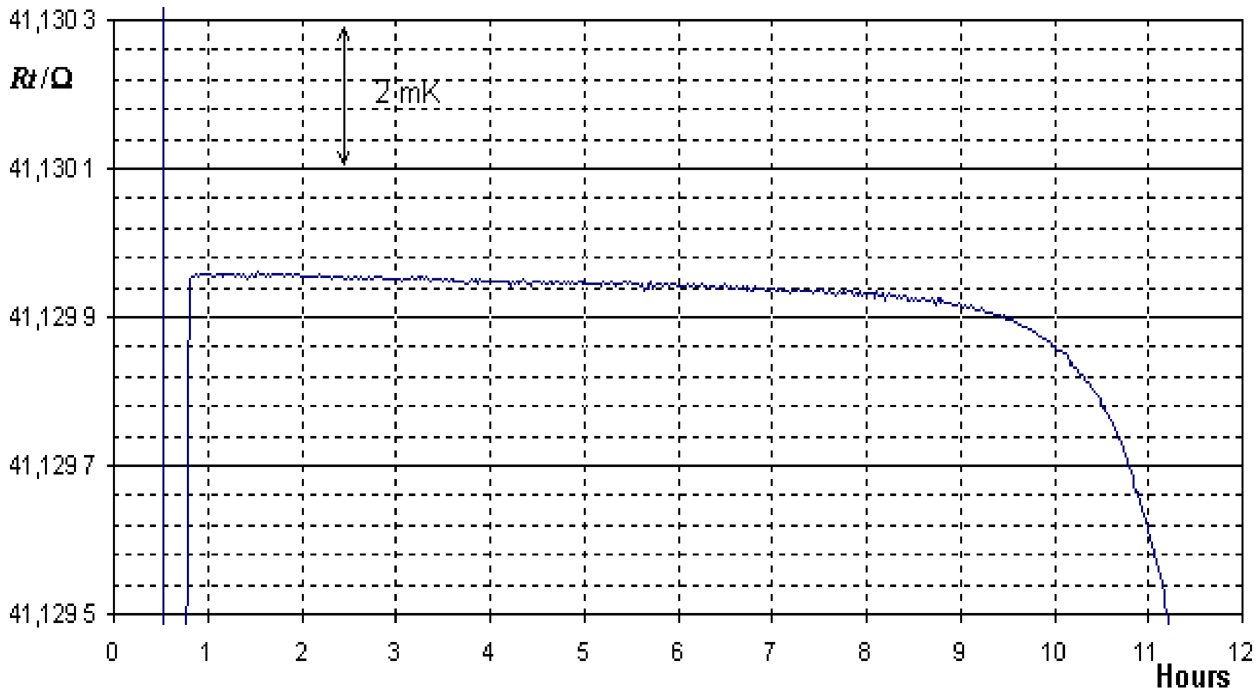

Figure 24. CEM indium phase transition curve

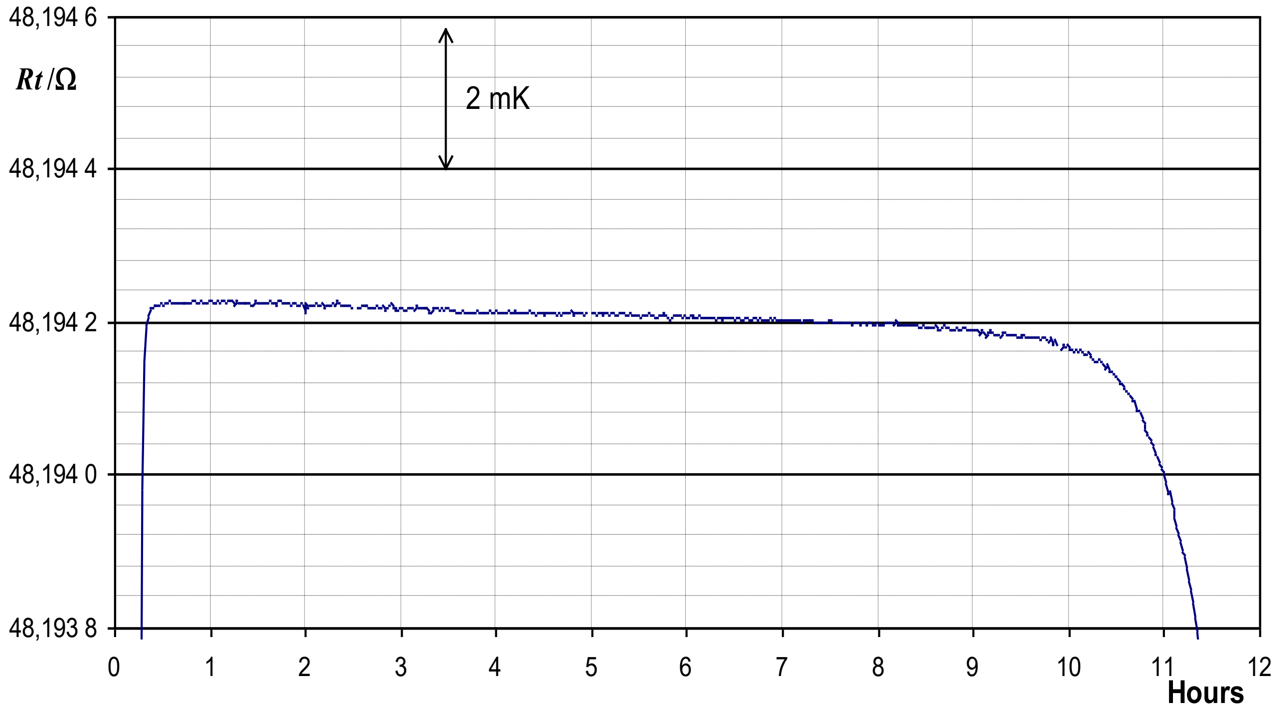

Figure 25. CEM tin phase transition curve

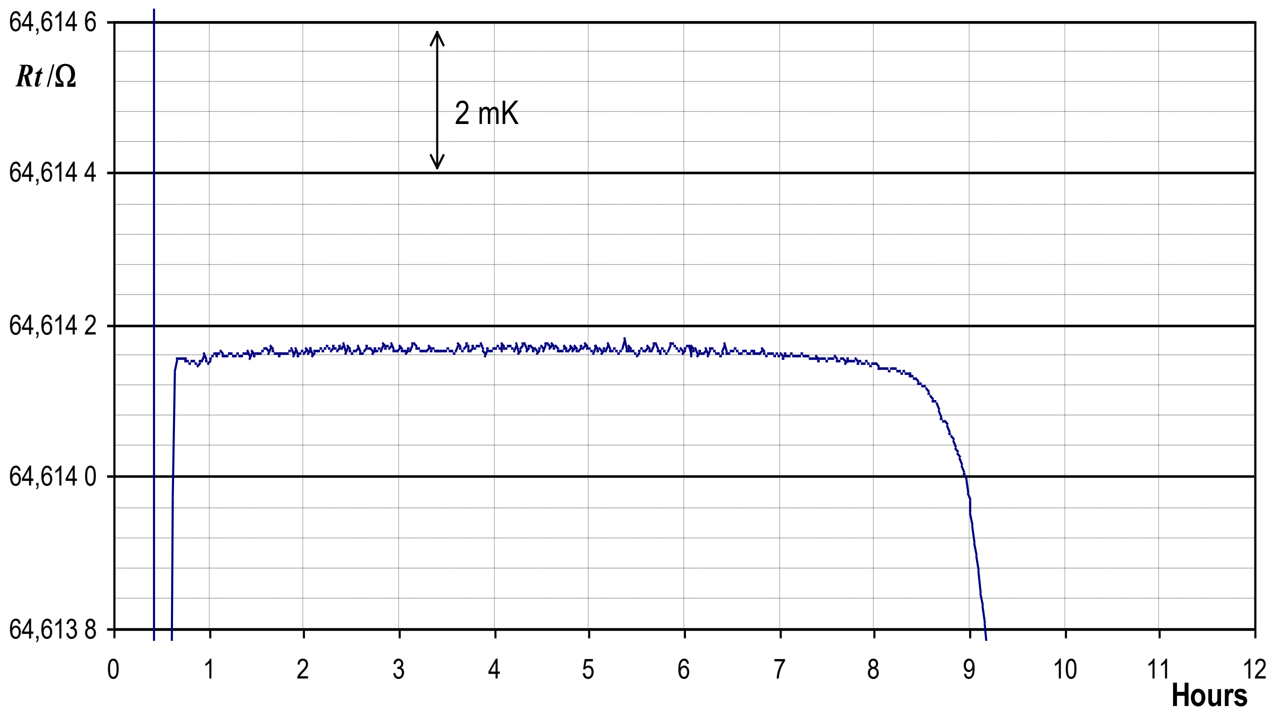

Figure 26. CEM zinc phase transition curve