FINAL REPORT

EURAMET.T-K3.3

Report on the comparison of the realisations of the ITS-90 over the range 83,805 8 K to 933,473 K between Centro Español de Metrología (CEM) and Laboratorio Costarricense de Metrología (LACOMET)

21st August 2012

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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 993,473 K (freezing point of aluminium) in the National Metrology Institutes of Spain (Centro Español de Metrología - CEM) and Costa Rica (Laboratorio Costarricense de Metrología - LACOMET) has been organized with the aim to provide support to the Calibration Measurement Capabilities claimed by LACOMET in this range. Due to the participation of CEM in the regional comparisons EUROMET.T-K3 and EUROMET.T-K4, the linkage with the corresponding key comparisons is possible.

There was an additional comparison point close to the triple point of argon (83,805 8 K) that CEM realized using an Argon Triple Point Apparatus, and LACOMET using a Nitrogen Boiling Point Apparatus to allow LACOMET to support their calibration by comparison capabilities at this temperature. The LACOMET apparatus consists of an equilibration block in a LN_2-cooled cryostat used to transfer a calibration from a reference SPRT to the transfer SPRT.

The measurements of this comparison were performed during the months of August to December of 2009.

1.1 Participants

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2. Protocol

The protocol of this comparison (see annex 3) was agreed between both participants and approved on July 2009 by the CCT-WG7 with few comments that were taken into account in the final performance of the comparison and the data analysis.

Additional measurements to the ones included in the protocol were performed by LACOMET when the standard platinum resistance thermometers (SPRT) came back to Costa Rica: the thermometer measured in the fixed points from argon to zinc was not only checked at the zinc freezing point but in all the fixed points.

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

Date	**Laboratory**	**Action**
26th August 2009	LACOMET	Start of measurements
25th September 2009	LACOMET	End of measurements
28th September 2009	LACOMET	SPRTs transported to CEM
1st October 2009	CEM	Start of measurements
20th November 2009	CEM	End of measurements
28th November	CEM/LACOMET	SPRTs transported to LACOMET
30th November – 18th December 2009	LACOMET	Checking of the SPRT's stability

Table 1. Schedule of the comparison

Table 2 summarizes the equipment used for both laboratories during the comparison.

Laboratory name	CEM	LACOMET
Bridge		
Manufacturer	A.S.L.	MEASUREMENTS INTERNATIONAL
Type	F18	6010 / 990108
AC or DC	AC	DC
If AC, give Frequency	75 Hz	-
If DC, give Period of reversal	-	8 s
Normal measurement current	1 mA	1 mA
Self-heating current	√2 mA	√2 mA
Evaluation of linearity of resistance	yes	yes
bridge (yes or not)	With calibrated inductive divider (RTU)	Double set of resistance (1, 10, 25, 100 and 1000) Ω
If yes, How?		
Reference resistor		
Manufacturer / type	Tinsley / Wilkins (model 5685 A)	TINSLEY / 5685A
Reference resistor temperature control (yes or not)	yes	yes
If yes, How?	Oil bath: (23 ± 0,01) °C	Oil bath: (20 ± 0,01) ºC
TPW Cell		
Manufacturer / model / sn	Jarret / A13 / 1179	Isotech-Jarret / B11-50 /435
Is it a primary reference? (if not explain its traceability)	Yes	NPTL, Isotech
Immersion depth of middle of the SPRT sensitive element/cm	25	23,5
How are mantles maintained (ice, bath,….)	stirred water bath	Water bath
Al Cell		
Manufacturer / model / sn	Isotech/ ITLM-17672 / Al 63	National Resear Council of Canada / NRC-1
Is it a primary reference? (if not explain its traceability)	Yes	NRC (Report - CCT/01-24)
Closed cell or open	open	Close
Nominal purity	99,999 9 %	6N
Immersion depth of middle of the SPRT sensitive element/cm	14	16
Al Furnace		
Type (1 zone, 3 zones, heat pipe, …….)	heat pipe	Heat pipe
Typical duration of the melting / freezing plateaux	8 h / 8 h	>7 h
Zn Cell		
Manufacturer / model /sn	Isotech / ITLM17671 / Zn 11	Isotech / ITL-M-17671/ Zn 136
Is it a primary reference? (if not explain its traceability)	yes	NPTL-Isotech-99-03-65, NRC (Report - CCT/01 24) and PTB-Nr-8392
Immersion depth of middle of the SPRT sensitive element/cm	12	16
Closed cell or open	open	Close
Nominal purity	99,999 9 %	6N
Zn Furnace		
Type (1 zone, 3 zones, heat pipe, …….)	3 zones	3 zone
Typical duration of the melting / freezing plateaux	9 h / 8,5 h	> 9 h
Sn Cell		
Manufacturer / model	L&N / 8411 / 742876	Isotech / ITL-M-17669 /Sn88
Is it a primary reference? (if not explain its traceability)	Yes	NPTL-Isotech-99-03-64, NRC (Report - CCT/01 24) and PTB-Nr-8392
Immersion depth of middle of the SPRT sensitive element/cm	15	16
Closed cell or open	closed	Close
Nominal purity	99,999 9 %	6N
Sn Furnace		
Type (1 zone, 3 zones, heat pipe, …….)	3 zones	1 zone
Typical duration of the melting / freezing plateaux	13 h /9,5 h	> 9 h

Laboratory name	CEM	LACOMET
In Cell		
Manufacturer / model /sn	isotech / ITL-M-17668-O / In 97	Isotech / ITL-M-17668 /In 63
Is it a primary reference? (if not explain its traceability)	Yes	NPTL-Isotech-99-03-63, NRC (Report - CCT/01 24) and PTB-Nr-8392
Immersion depth of middle of the SPRT sensitive element/cm	13	16
Closed cell or open	open	Close
Nominal purity	99,999 9 %	6N
In Furnace		
Type (1 zone, 3 zones, heat pipe, …….)	3 zones	1 zone
Typical duration of the melting / freezing plateaux	8,5 / 8,5	> 9 h
Ga Cell		
Manufacturer / model /sn	YSI / 17401 / L8256	Isotech / ITL-M-17401 /Ga 240
Is it a primary reference? (if not explain its traceability)	Yes	NPTL-Isotech-99-04-05, NRC (Report - CCT/01 24) and PTB-Nr-8392
Immersion depth of middle of the SPRT sensitive element/cm	25	21,5
Closed cell or open	closed	Close
Nominal purity	99,999 9+ %	7N
Ga Furnace		
Type (1 zone, 3 zones, heat pipe, …….)	1 zone	1 zone
Typical duration of the melting / freezing plateaux	9 h / 8 h	> 12 h
Hg Cell		
Manufacturer / model	Isotech / ITLM-17924 / Hg 62	Isotech / 17924 / Hg 129
Is it a primary reference? (if not explain its traceability)	yes	NPTL-Isotech-15-03-2000, NRC (Report - CCT/01-24) and PTB-Nr-8392
Immersion depth of middle of the SPRT sensitive element/cm	17	14,5
Closed cell or open	closed	Close
Nominal purity	99,999 95%	7N
Hg cryostat		
Type (cryostat, bath, …….)	alcohol stirred bath	Cryostat
Typical duration of the melting / freezing plateaux	9 h / 8,5 h	>9 h
Ar Cell		
Manufacturer / model /sn	INM / - / Ar 29	-
Is it a primary reference? (if not explain its traceability)	yes	-
Immersion depth of middle of the SPRT sensitive element/cm	7	-
Closed cell or open	closed	-
Nominal purity	99,999 9 %	-
Ar cryostat		
Type (cryocooler, bath,..)	liquid nitrogen bath	-
Typical duration of the melting plateau	9 h	-
N Boliling Point Apparatus		
Manufacturer / model	-	Isotech / ITL-M-18205
Typical duration of the melting plateau	-	CF-4515-255
SPRT used as reference manufacturer/model	-	Isotech / 670-082
Calibrated at (Laboratory name)	-	NORTHERN TEMPERATURE PRIMARY LABORATORY
Calibration date	-	36323

Table 2. Summary of the equipment used

CEM maintains their fixed points by means of a group of cells. Periodically, comparisons are performed to assure their integrity. In addition control SPRT is assigned to each fixed point and all the plateaux performed are initiated and finalised using them.

3. Transfer standards

The transfer standards were two 25 Ω SPRTs one for the fixed points of Hg, Ga, In, Sn and Zn and the additional Ar point and another for the freezing point of aluminium. The thermometers had proven stability and were provided by LACOMET.

Manufacturer	Model	Serial number	Calibration points
Isotech	670	054	Zn, Sn, In, Ga, Hg, Ar
Isotech	670	244	Al

Table 3. Transfer standards

The resistance of the travelling SPRTs 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

The results obtained by the participants in the different fixed points are included below from tables 4 to 10, where the provided resistance ratios for each laboratory are shown. Figures 1 to 7 plot the results with their assigned uncertainties; the solid lines represent the corresponding values of the related key comparisons. The procedure for linking the values to the key comparisons and the uncertainty calculation is explained in section 6 of this report.

The immersion profiles of the fixed points of CEM and LACOMET are in annex 1 and annex 2 respectively. In addition, examples of the phase transition curves for each fixed point are included in annex 3 and 4.

	ALUMINIUM	
	W	*U*/mK
LACOMET	3,375 380 9	4,6
CEM	3,375 383 7	2,9
LACOMET	3,375 391 6	4,6

Table 4. Results for aluminium

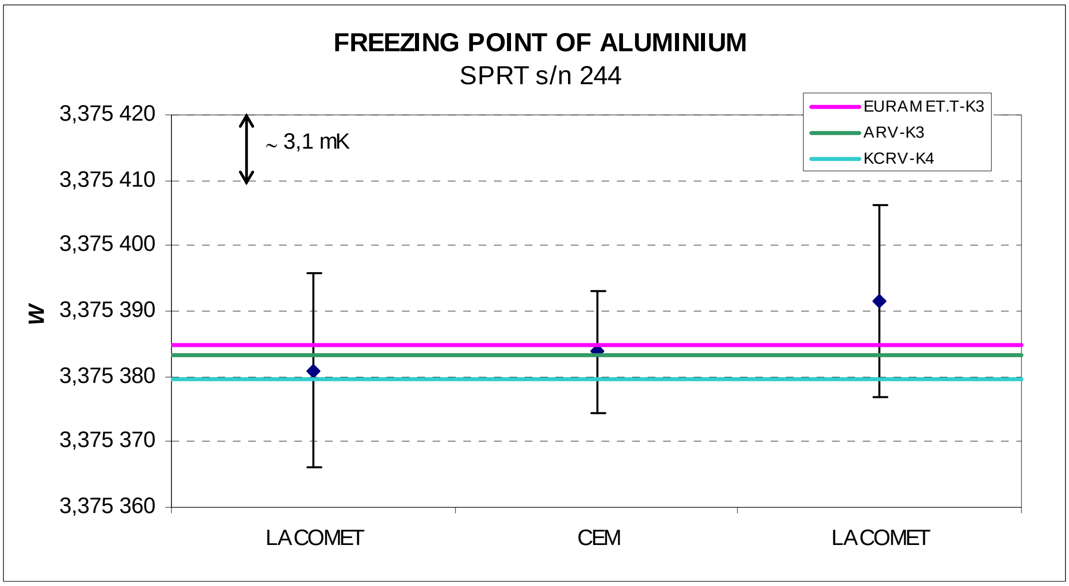

Figure 1. Results for aluminium

	ZINC	
	W	*U*/mK
LACOMET	2,568 603 4	1,9
CEM	2,568 611 2	0,92
LACOMET	2,568 601 9	1,9

Table 5. Results for zinc

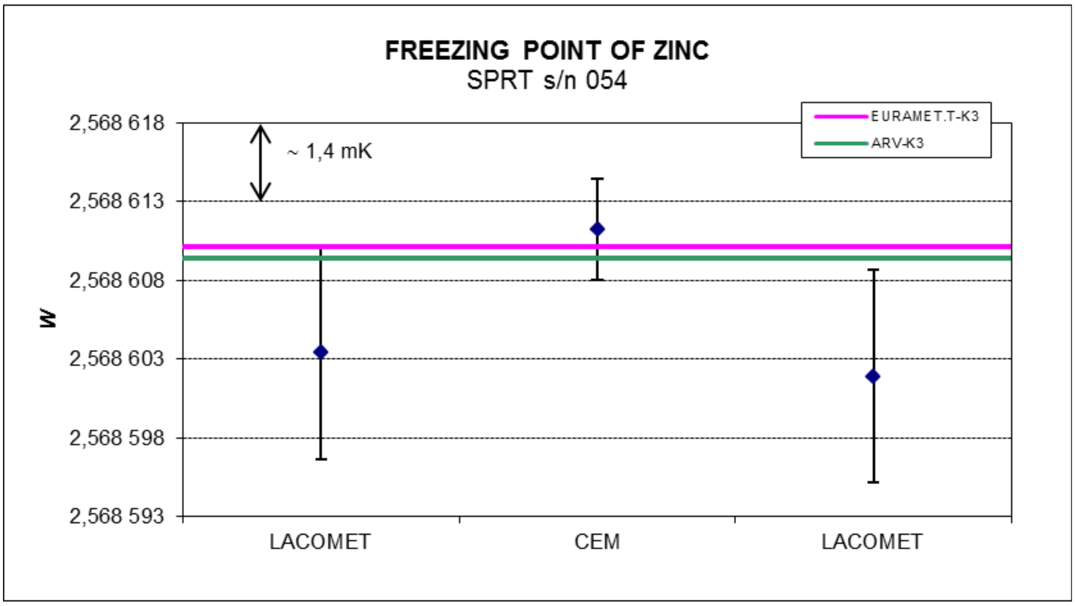

Figure 2. Results for zinc

	TIN	
	W	*U*/mK
LACOMET	1,892 626 2	1,5
CEM	1,892 631 8	0,80
LACOMET	1,892 627 6	1,5

Table 6. Results for tin

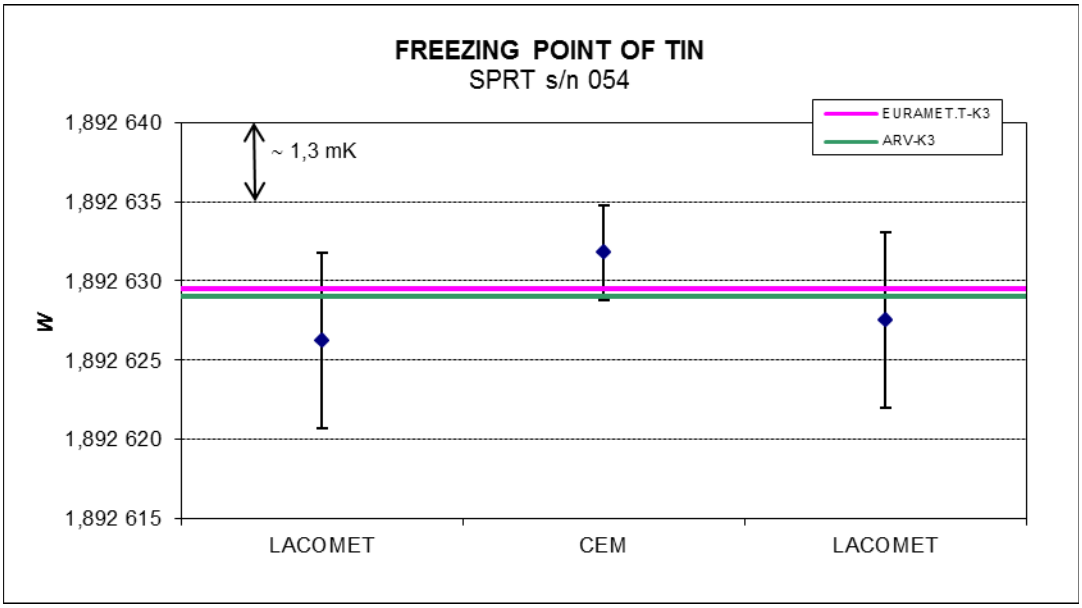

Figure 3. Results for tin

	INDIUM	
	W	*U*/mK
LACOMET	1,609 686 9	1,5
CEM	1,609 685 6	0,78
LACOMET	1,609 687 6	1,5

Table 7. Results for indium

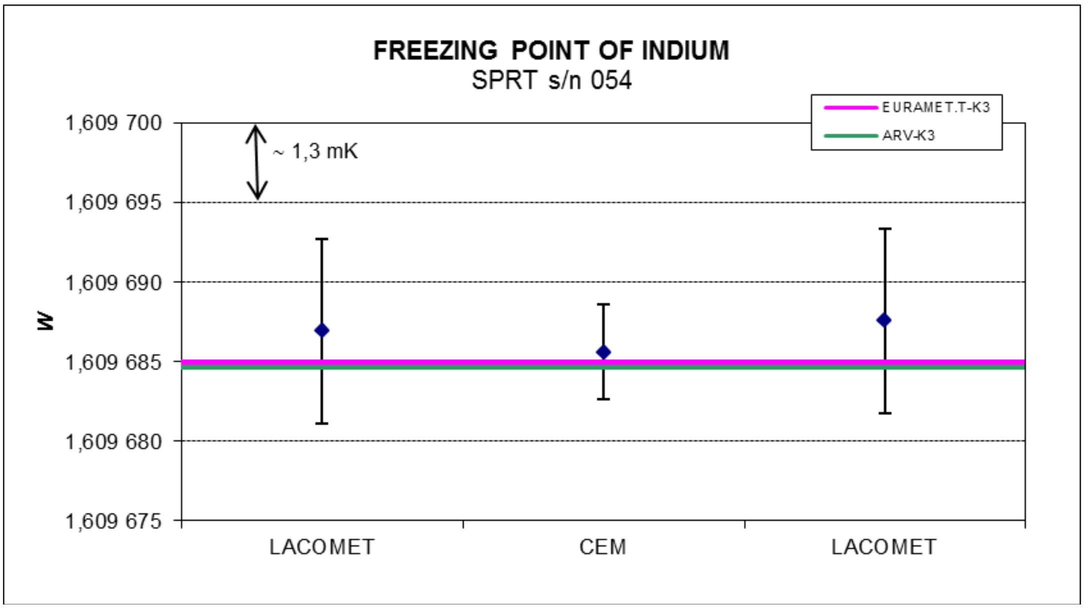

Figure 4. Results for indium

	GALLIUM	
	W	*U*/mK
LACOMET	1,118 116 9	0,48
CEM	1,118 115 7	0,39
LACOMET	1,118 116 6	0,48

Table 8. Results for gallium

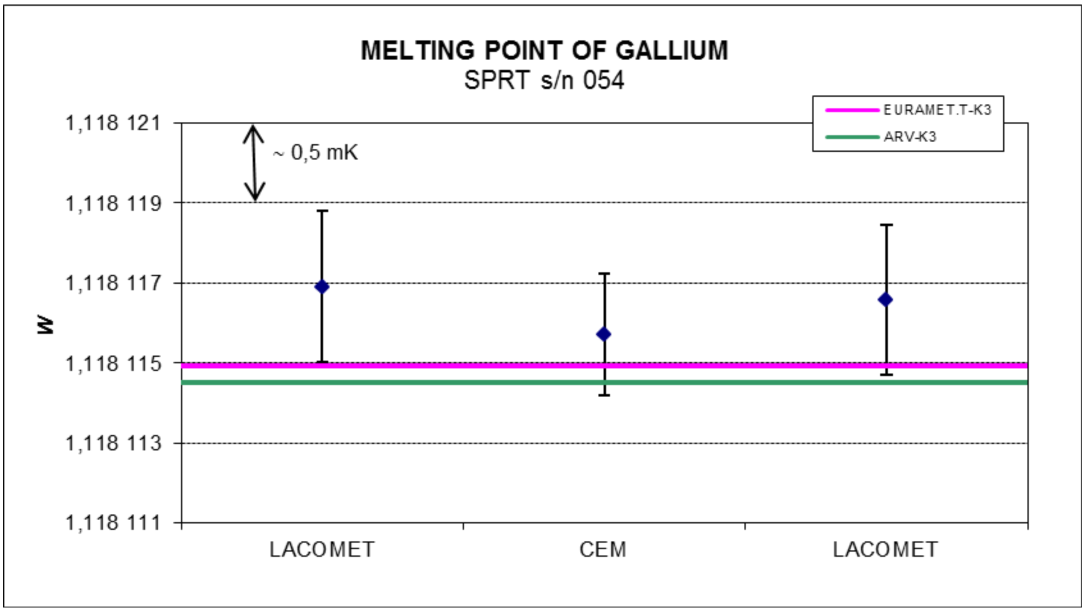

Figure 5. Results for gallium

	MERCURY	
	W	***U*/mK**
LACOMET	0,844 166 3	0,49
CEM	0,844 166 2	0,40
LACOMET	0,844 165 3	0,49

Table 9. Results for mercury

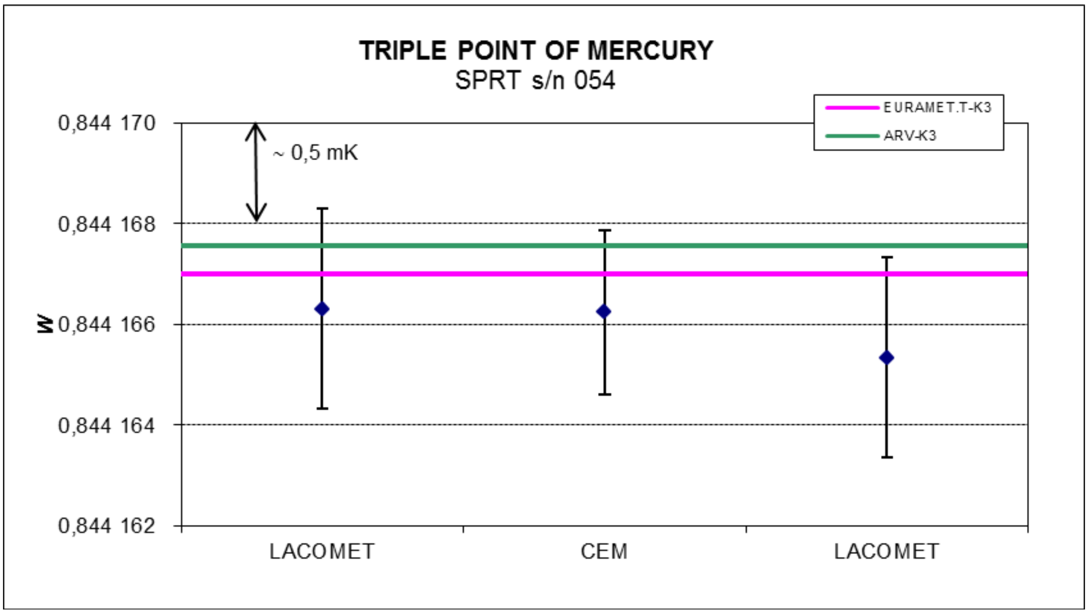

Figure 6. Results for mercury

	ARGON	
	W	***U*/mK**
LACOMET	0.215 986 0	11
CEM	0.215 984 2	1,2
LACOMET	0.215 985 8	11

Table 10. Results for argon

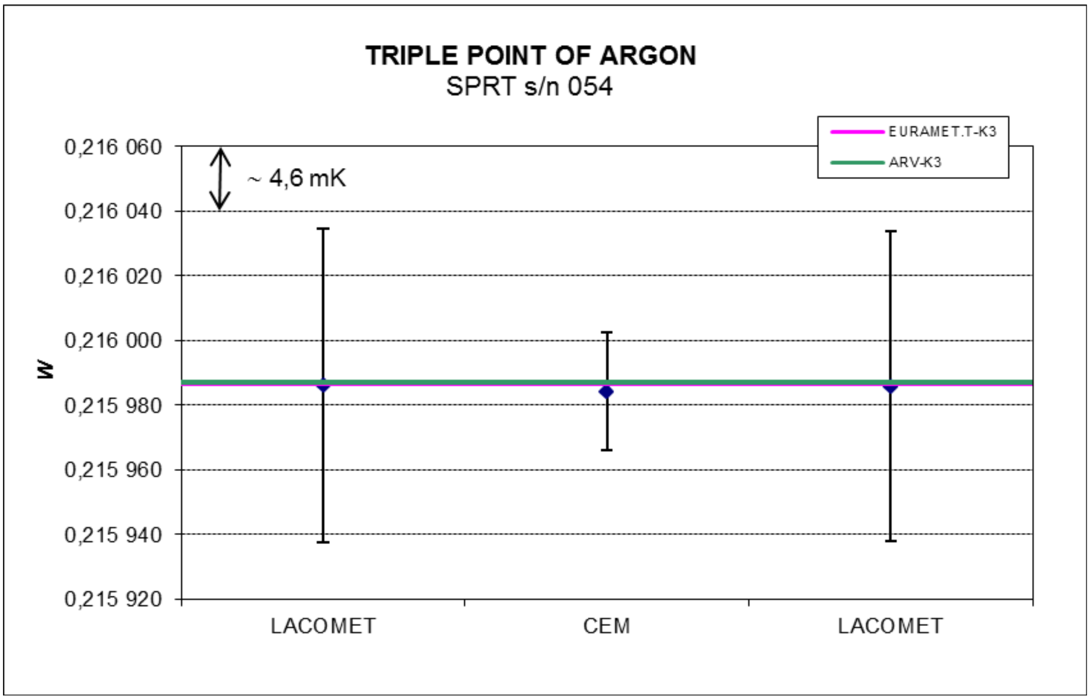

Figure 7. Results for argon

In this comparison, the LACOMET Zn cell has realized a Zn fixed point temperature lower that the one realized by the CEM Zn fixed point. An overpressure in the cell can not be the reason because it would cause the Zn cell to be hotter. A previous comparison [3], [4], had shown similar differences in the past, in consequence impurities are the most probable cause of the lower temperature realized by the LACOMET Zn cell. LACOMET has taken this into account in its uncertainties estimation but no correction has been applied for this comparison.

5. Uncertainties

The participants were requested to supply the uncertainty budget associated with the calibration at the different fixed points. It was asked to the laboratories to fill the agreed uncertainty format included in the protocol. The uncertainty budgets can be found in table 11.

Table 11. Summary of uncertainties

Components	ALUMINIUM		ZINC		TIN		INDIUM		GALLIUM		MERCURY		ARGON	
	Uncertainty contribution u_i / mK		Uncertainty contribution u_i / mK		Uncertainty contribution u_i / mK		Uncertainty contribution u_i / mK		Uncertainty contribution u_i / mK		Uncertainty contribution u_i / mK		Uncertainty contribution u_i / mK	
Q_i	**CEM**	**LACOMET**	**CEM**	**LACOMET**	**CEM**	**LACOMET**	**CEM**	**LACOMET**	**CEM**	**LACOMET**	**CEM**	**LACOMET**	**CEM**	**LACOMET**
SPRT repeatability	0,063	1,50	0,057	0,30	0,054	0,20	0,053	0,20	0,051	0,08	0,050	0,15	0,046	0,5
SPRT oxidations	-	0,20	-	0,10	-	0,10	-	0,10	-	0,05	-	0,05	-	0,05
SPRT Drift at high temperature	-	0,90	-	0,60	-	0,50	-	0,50	-	0,10	-	0,10	-	0,1
Choice of the freezing point value	0,577	0,60	0,058	0,20	0,162	0,20	0,058	0,20	0,058	0,10	0,040	0,10	0,520	-
Purity of the fixed point cell	0,387	-	0,326	-	0,250	-	0,270	-	0,039	-	0,057	-	0,028	-
Hydrostatic pressure correction (*)	0,016	0,10	0,027	0,10	0,022	0,08	0,033	0,08	0,012	0,04	0,071	0,04	0,033	-
Pperturbing heat exchanges (*)	0,346	0,40	0,029	0,15	0,115	0,15	0,173	0,15	0,012	0,02	0,058	0,02	0,140	0,1
Self-heating correction (*)	0,092	0,30	0,092	0,16	0,092	0,15	0,092	0,13	0,092	0,05	0,092	0,05	0,092	0,1
Bridge linearity (*)	0,083	0,15	0,076	0,13	0,072	0,10	0,070	0,07	0,067	0,03	0,066	0,03	0,061	0,03
AC/DC differences including quadrature effects	0,087	-	0,029	-	0,023	-	0,017	-	0,017	-	0,017	-	0,017	-
Gas pressure in the fixed point cell	0,040	0,30	0,025	0,20	0,019	0,20	0,028	0,15	0,001	0,10	0,003	0,01	0,014	-
SPRT heat conduction	0,866	-	0,000	-	0,000	-	0,000	-	0,000	-	0,000	-	0,231	-
High -temperature insulation degradation	0,577	0,05	0,000	0,00	0,000	0,00	0,000	0,00	0,000	0,00	0,000	0,00	0,000	-
Uncertainties coming from the SPRT used as reference	-	-	-	-	-	-	-	-	-	-	-	-	-	5
Uncertainties due to the interpolation at the Ar fixed point	-	-	-	-	-	-	-	-	-	-	-	-	-	0,8
Uncertainties coming from the isothermal enclosure	-	-	-	-	-	-	-	-	-	-	-	-	-	2,3
Phase transition repeatability	0,382	0,95	0,044	0,20	0,017	0,20	0,020	0,05	0,053	0,05	0,058	0,05	0,058	
Effects of changes in the reference resistor	0,002	0,01	0,001	0,01	0,001	0,01	0,001	0,01	0,001	0,01	0,001	0,01	0,001	0,01
UNCERTAINTIES LINKED WITH THE PROPAGATION FROM THE TRIPLE POINT OF WATER														
Repeatability of readings	0,025	-	0,023	-	0,022	-	0,021	-	0,020	-	0,020	-	0,018	-
Repeatability of temperature realized by cell	0,004	-	0,004	-	0,004	-	0,004	-	0,004	-	0,004	-	0,004	-
Aging of the cell	-	0,01	-	0,01	-	0,01	-	0,01	-	0,01	-	0,01	-	0,01
Short repeatability of calibrated SPRT	0,249	-	0,018	-	0,050	-	0,024	-	0,012	-	0,015	-	0,032	-
Purity and isotopic composition	0,034	0,70	0,034	0,50	0,034	0,30	0,034	0,40	0,034	0,06	0,034	0,06	0,034	0,00
Hydrostatic pressure correction	0,008	-	0,006	-	0,005	-	0,004	-	0,003	-	0,002	-	0,001	-
Perturbing heat exchanges	0,034	-	0,026	-	0,019	-	0,016	-	0,011	-	0,008	-	0,002	-
Self-heating correction	0,389	-	0,272	-	0,188	-	0,156	-	0,105	-	0,077	-	0,018	-
Bridge linearity	0,070	-	0,049	-	0,034	-	0,028	-	0,019	-	0,014	-	0,003	-
AC/DC differences including quadrature effects	0,087	-	0,029	-	0,023	-	0,017	-	0,017	-	0,017	-	0,017	-
Internal insulation leakage	0,000	0,05	0,000	0,03	0,000	0,02	0,000	0,02	0,000	0,01	0,000	0,01	0,000	-
Uncertainty propagated from the triple point of water	-	0,28	-	0,17	-	0,15	-	0,10	-	0,08	-	0,06	-	0,15
Combined uncertainty	1,445	2,304	0,460	0,967	0,402	0,771	0,391	0,766	0,193	0,239	0,201	0,246	0,605	5,589
Expanded uncertainty	**2,89**	**4,61**	**0,92**	**1,93**	**0,80**	**1,54**	**0,78**	**1,53**	**0,39**	**0,48**	**0,40**	**0,49**	**1,21**	**11,2**

(*) This source of uncertainty for LACOMET includes the hydrostatic head errors in the triple point of water and in the corresponding fixed point

The combined uncertainties were computed by root-sum-of-squares of the contributions. Whatever the fixed point considered, LACOMET evaluated all their uncertainties using type B method and estimated as infinite their degrees of freedom so a coverage factor k=2 was used in order to calculate the expanded uncertainties to approximately 95 % probability. 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-Satterthwaite formula.

In 2007, after the CIPM 2005 clarification of the isotopic composition defining the value of the triple point of water, CEM decided to change the value maintained in +0,14 mK. During this comparison, the value used for the triple point of water was the value corrected for isotopic composition what consequently differs in +0,14 mK with respect the reference value used for the triple point of water during the EURAMET.T-K3 comparison. The uncertainty due to the isotopic composition has been taken into account in the uncertainty calculations,

6. Linkage to CCT comparisons

The linkage to the CCT comparisons has been made from the differences obtained between CEM and LACOMET and the differences that CEM obtained in the regional comparisons EUROMET.T-K3 and EUROMET.T-K4 that provided linking to the CCT-K3 and CCT-K4 comparisons.

6.1. Differences between CEM and LACOMET

The differences in temperature $T_{\mathrm{LACOMET}} - T_{\mathrm{CEM}}$ are calculated using the W values provided for each fixed point and the sensitivity coefficient of the ITS-90 reference function $\mathrm{d}T/\mathrm{d}W_{\mathrm{r}}$. The values for LACOMET are calculated using the mean of the W measured values before and after CEM measurements:

$$T_{\mathrm{LACOMET}} - T_{\mathrm{CEM}} = \left(\frac{W_{\mathrm{LACOMET\text{-}before}} + W_{\mathrm{LACOMET\text{-}after}}}{2} - W_{\mathrm{CEM}}\right) \cdot \frac{\mathrm{d}T}{\mathrm{d}W_{\mathrm{r}}} \qquad (1)$$

The uncertainty of this difference is estimated using the uncertainties calculated by the laboratories (see table 11). An additional uncertainty due to the SPRT drift is taken into account, it is estimated from the differences measured by LACOMET for the SPRT in each fixed point before and after CEM measurements (see table 12). These differences are consistent within the calculated uncertainties.

FIXED POINT	***Differences*** $T_{LACOMET\text{-}before} - T_{LACOMET\text{-}after}$ mK	$T_{LACOMET} - T_{CEM}$ mK	$U(T_{LACOMET} - T_{CEM})$ mK
Al	-3,33	0,8	5,4
Zn	0,43	-2,4	2,1
Sn	-0,36	-1,3	1,7
In	-0,17	0,4	1,7
Ga	0,08	0,26	0,61
Hg	0,24	-0,10	0,64
Ar	-	0,4	11

Table 12. Differences in mK between the measurements performed by LACOMET before and after CEM measurements

A rectangular probability distribution is assigned to calculate the standard uncertainty of the comparison.

$$u^2(T_{LACOMET} - T_{CEM}) = u^2\,(W_{LACOMET}) + u^2\,(W_{CEM}) + u^2\,(T_{drift}) \quad (2)$$

A coverage factor k = 2 is considered to calculate the expanded uncertainty to approximately 95 % probability. Table 12 summarizes the differences for all the fixed points with their corresponding uncertainties.

6.2. Differences between LACOMET and EUROMET.T-K3

The differences in temperature $T_{LACOMET} - T_{EUROMET.T\text{-}K3}$ are evaluated using the $T_{LACOMET} - T_{CEM}$ differences calculated in the previous paragraph and the hypothesis that the CEM value has no change since the EUROMET.T-K3 comparison was performed. It is important to highlight that CEM employed in this comparison the same reference cells used in the EUROMET comparison. The differences $T_{CEM} - T_{EUROMET.T\text{-}K3}$ are obtained from the tables 16 to 21 of the final report to the CCT on Key Comparison EUROMET. T-K3 [1]:

$$T_{LACOMET} - T_{EUROMET.T\text{-}K3} = (T_{LACOMET} - T_{CEM}) + (T_{CEM} - T_{EUROMET.T\text{-}K3}) \quad (3)$$

Using the law of propagation of uncertainties in (3) and the uncertainties provided in tables 16 to 21 in [1] it is possible to estimate the uncertainty of the differences $T_{\mathrm{LACOMET}} - T_{\mathrm{EUROMET.T\text{-}K3}}$:

$$u^2(T_{\mathrm{LACOMET}} - T_{\mathrm{EUROMET.T\text{-}K3}}) = u^2(T_{\mathrm{LACOMET}} - T_{\mathrm{CEM}}) + u^2(T_{\mathrm{CEM}} - T_{\mathrm{EUROMET.T\text{-}K3}}) \quad (4)$$

A coverage factor $k = 2$ is considered to calculate the expanded uncertainty to approximately 95 % probability. Table 13 summarizes the differences for all the fixed points with their corresponding uncertainties.

6.3. Differences between LACOMET, CCT-K3 and CCT-K4

The differences in temperature $T_{\mathrm{LACOMET}} - T_{\mathrm{ARV\text{-}K3}}$ are calculated using the $T_{\mathrm{LACOMET}} - T_{\mathrm{EUROMET.T\text{-}K3}}$ differences obtained in the previous paragraph and the same hypothesis proposed in [1] that is: the mean temperature of the pilot and co-pilot laboratories is the same in EUROMET.T-K3 as it was in CCT-K3. Tables 22 to 27 in [1] show the differences between the mean of the pilot and co-pilot laboratories in the CCT-K3 ($T_{\mathrm{ARV\text{-}K3}} - T_{\mathrm{P\&CPmean}}$) and the differences between the EUROMET.T-K3 reference value and the mean of the pilot and co-pilot laboratories ($T_{\mathrm{EUROMET.T\text{-}K3}} - T_{\mathrm{P\&CPmean}}$). With this information it is possible to link the LACOMET results to the CCT-K3 comparison:

$$T_{\mathrm{LACOMET}} - T_{\mathrm{ARV\text{-}K3}} = (T_{\mathrm{LACOMET}} - T_{\mathrm{EUROMET.T\text{-}K3}}) + (T_{\mathrm{EUROMET.T\text{-}K3}} - T_{\mathrm{P\&CPmean}}) - (T_{\mathrm{ARV\text{-}K3}} - T_{\mathrm{P\&CPmean}}) \quad (5)$$

To estimate the uncertainty of $T_{\mathrm{LACOMET}} - T_{\mathrm{ARV\text{-}K3}}$ we have taken into account the uncertainties of $T_{\mathrm{LACOMET}} - T_{\mathrm{EUROMET.T\text{-}K3}}$ differences and the standard deviation of the mean of the EUROMET.T-K3 pilot and co-pilot laboratories differences to the CCT-K3 average reference value (ARV) and to the EUROMET.T-K3 reference value:

$$u^2(T_{\mathrm{LACOMET}} - T_{\mathrm{ARV\text{-}K3}}) = u^2(T_{\mathrm{LACOMET}} - T_{\mathrm{EUROMET.T\text{-}K3}}) + u^2(T_{\mathrm{P\&CPmean\_CCT\text{-}K3}}) + u^2(T_{\mathrm{P\&CPmean\_EUROMET.T\text{-}K3}}) \quad (6)$$

In the case of the freezing point of aluminium the Report to the CCT on Key Comparison EUROMET.T-K4 [2] provides in its 17th page information related to the linkage to the CCT-K3 and CCT-K4 comparison for the aluminium freezing point with their corresponding uncertainties that can be used to link the LACOMET results to the corresponding CCT comparisons:

$$T_{\mathrm{LACOMET}} - T_{\mathrm{ARV\text{-}K3}} = (T_{\mathrm{LACOMET}} - T_{\mathrm{EUROMET.T\text{-}K4}}) + (T_{\mathrm{EUROMET.T\text{-}K4}} - T_{\mathrm{ARV\text{-}K3}}) \quad (7)$$

$$T_{\mathrm{LACOMET}} - T_{\mathrm{KCRV\text{-}K4}} = (T_{\mathrm{LACOMET}} - T_{\mathrm{EUROMET.T\text{-}K4}}) + (T_{\mathrm{EUROMET.T\text{-}K4}} - T_{\mathrm{KCV\text{-}K4}}) \quad (8)$$

Using the law of propagation of uncertainties in (5) and (6) together with the information provided in [2] it is possible to evaluate the uncertainty of these differences:

$$u^2(T_{\mathrm{LACOMET}} - T_{\mathrm{ARV\text{-}K3}}) = u^2(T_{\mathrm{LACOMET}} - T_{\mathrm{EUROMET.T\text{-}K4}}) + u^2(T_{\mathrm{EUROMET.T\text{-}K4}} - T_{\mathrm{ARV\text{-}K3}}) \quad (9)$$

$$u^2(T_{\mathrm{LACOMET}} - T_{\mathrm{KCRV\text{-}K4}}) = u^2(T_{\mathrm{LACOMET}} - T_{\mathrm{EUROMET.T\text{-}K4}}) + u^2(T_{\mathrm{EUROMET.T\text{-}K4}} - T_{\mathrm{KCV\text{-}K4}}) \quad (10)$$

A coverage factor k = 2 is considered to calculate the expanded uncertainty to approximately 95 % probability. Tables 13 and 14 summarize the differences for all the fixed points with their corresponding uncertainties.

FIXED POINT	$T_{CEM} – T_{EUROMET.T-K3}$ mK	$U(T_{CEM} – T_{EUROMET.T-K3})$ mK	$T_{LACOMET} – T_{EUROMET.T-K3}$ mK	$U(T_{LACOMET} – T_{EUROMET.T-K3})$ mK	$T_{CEM} – T_{EUROMET.T-K4}$ mK	$U(T_{CEM} – T_{EUROMET.T-K4})$ mK	$T_{LACOMET} – T_{EUROMET.T-K4}$ mK	$U(T_{LACOMET} – T_{EUROMET.T-K3})$ mK
Al	-	-	-	-	-0,3	7,4	0,4	9,2
Zn	0,3	2,4	-2,1	3,2	-	-	-	-
Sn	0,6	1,4	-0,7	2,2	-	-	-	-
In	0,2	1,2	0,6	2,1	-	-	-	-
Ga	0,20	0,55	0,46	0,82	-	-	-	-
Hg	-0,19	0,57	-0,29	0,85	-	-	-	-
Ar	-0,6	1,1	-0,1	12	-	-	-	-

Table 13. Linkage to the EURAMET regional comparisons

FIXED POINT	$T_{CEM} – T_{ARV-K3}$ mK	$U(T_{CEM} – T_{ARV-K3})$ mK	$T_{LACOMET} – T_{ARV-K3}$ mK	$U(T_{LACOMET} – T_{ARV-K3})$ mK	$T_{CEM} – T_{KCRV-K4}$ mK	$U(T_{CEM} – T_{KCRV-K4})$ mK	$T_{LACOMET} – T_{KCRV-K4}$ mK	$U(T_{LACOMET} – T_{KCRVK4})$ mK
Al	0,2	7,7	1,0	9,4	-1,3	7,7	2,1	9,7
Zn	0,5	2,5	-2,0	3,3	-	-	-	-
Sn	0,7	1,5	-0,6	2,2	-	-	-	-
In	0,2	1,3	0,7	2,2	-	-	-	-
Ga	0,30	0,57	0,56	0,84	-	-	-	-
Hg	-0,33	0,61	-0,43	0,88	-	-	-	-
Ar	-0,6	1,2	-0,2	12	-	-	-	-

Table 14. Linkage to the CCT key comparison.

7. References

[1] Report to the CCT on Key Comparison EUROMET. T-K3. http://kcdb.bipm.org/appendixB/appbresults/euromet.t-k3/euromet.t-k3_final_report.pdf.

[2] Report to the CCT on Key Comparison EUROMET. T-K4. http://kcdb.bipm.org/appendixB/appbresults/cct-k4/euromet.t-k4_final_report.pdf.

[3] TEST MEASUREMENTS REPORTPN 1997.2263.8 / PTB-Nr. 8392.

[4] Certificate: NPTL 99-03-65; NAMAS N°0175

ANNEX 1. CEM FIXED POINT CELLS IMMERSION PROFILES

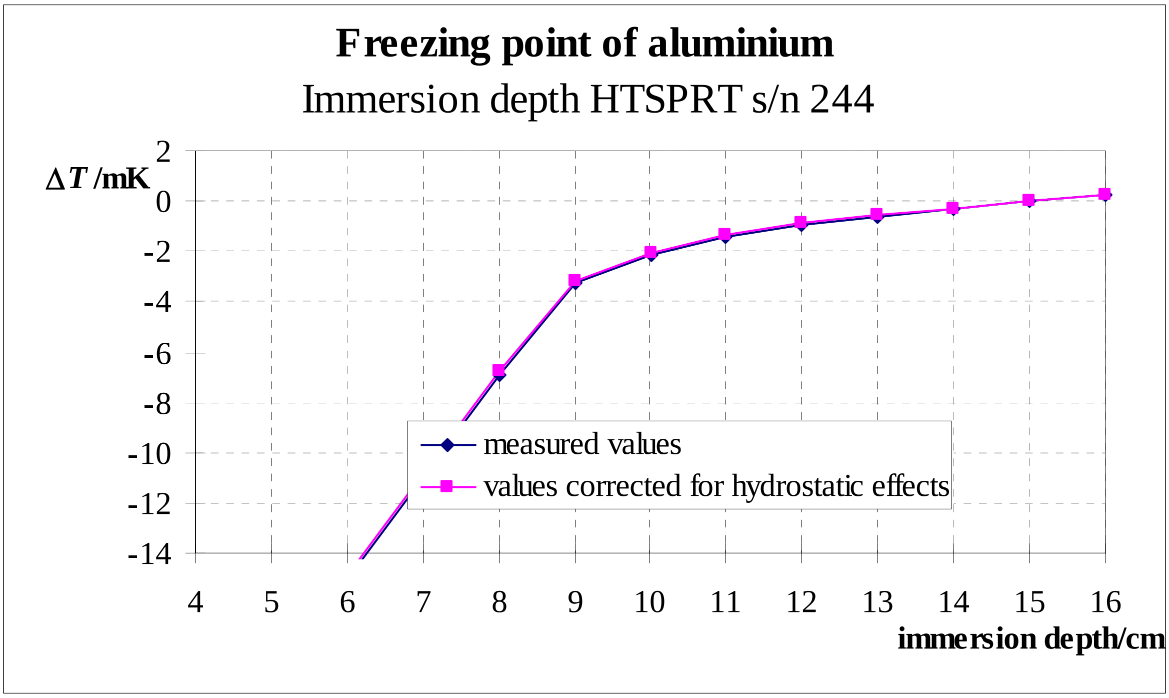

Figure 8. HTSPRT s/n 224, immersion profile in the aluminium freezing point

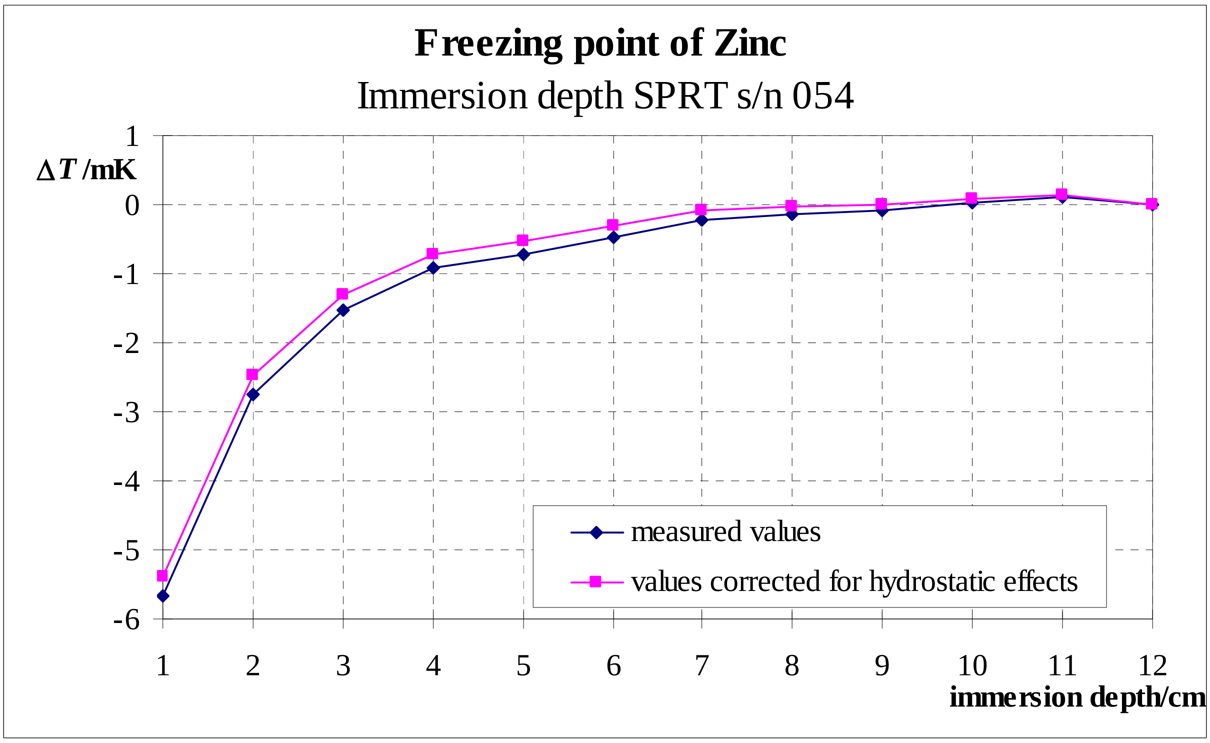

Figure 9. SPRT s/n 054, immersion profile in the zinc freezing point

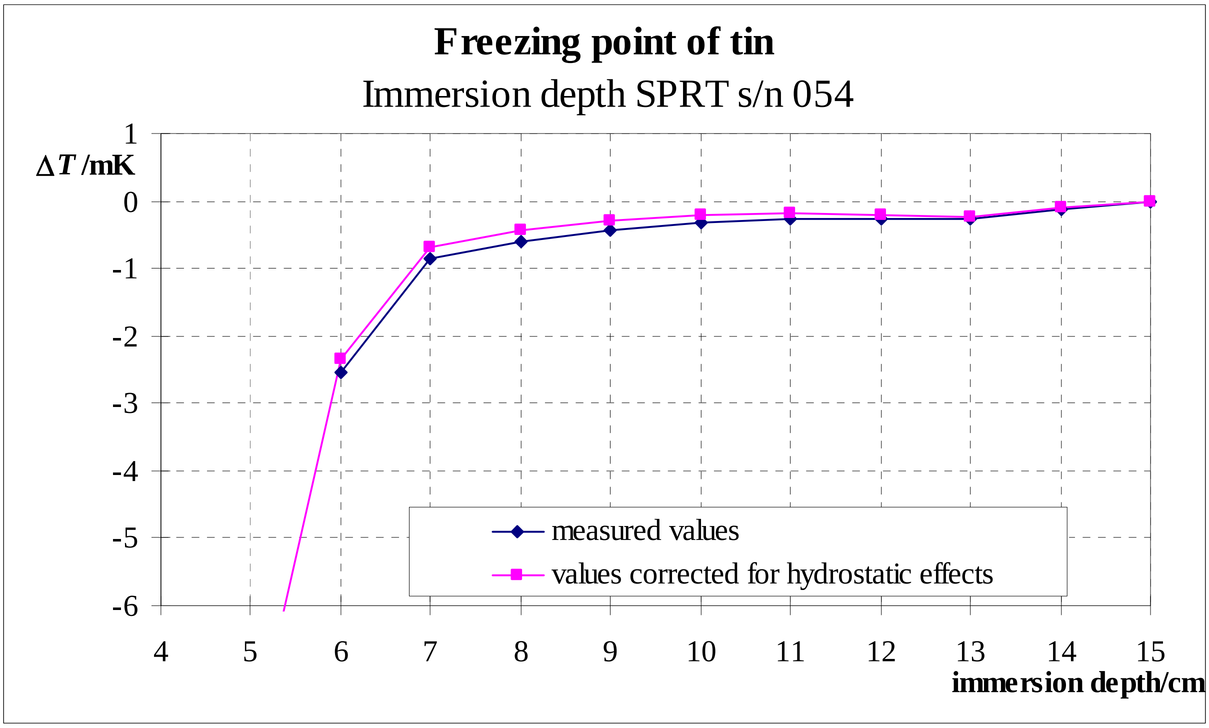

Figure 10. SPRT s/n 054, immersion profile in the tin freezing point

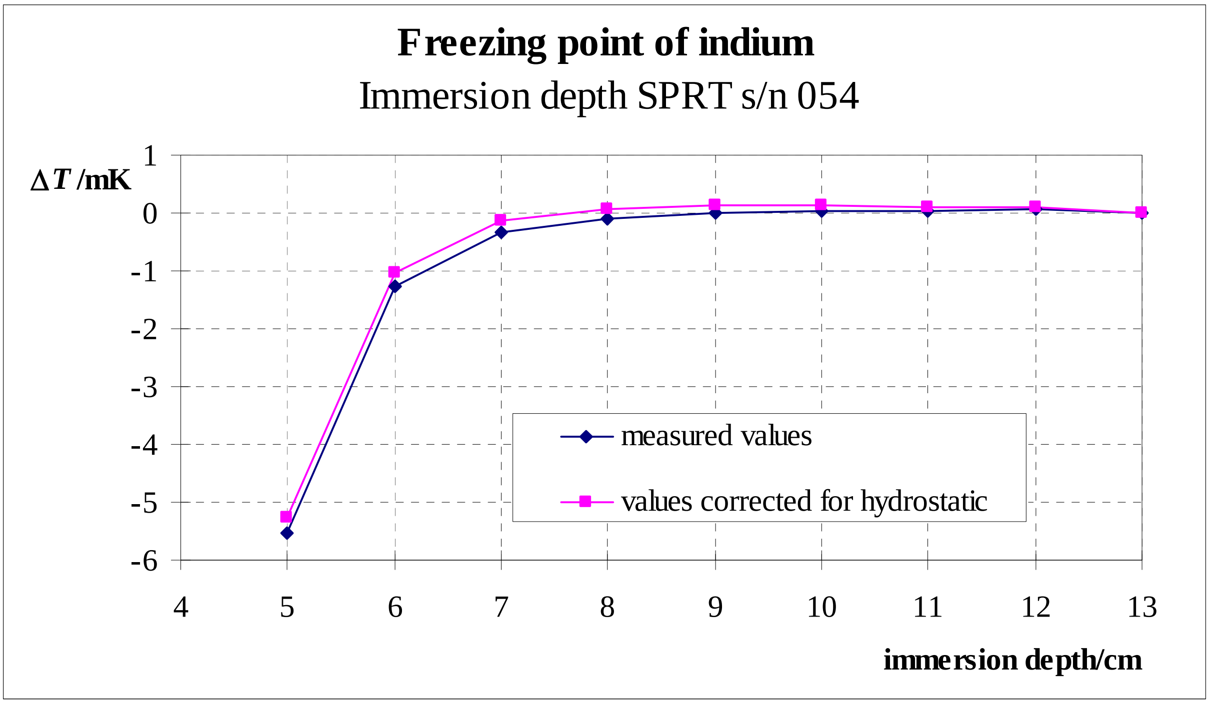

Figure 11. SPRT s/n 054, immersion profile in the indium freezing point

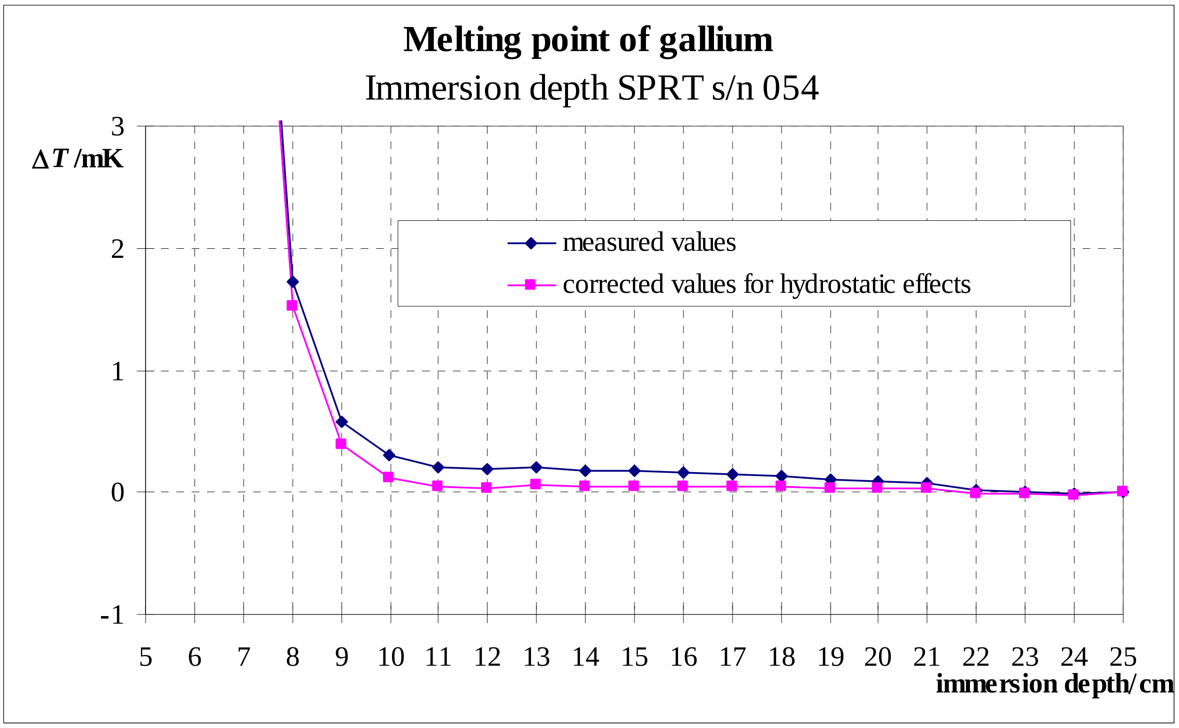

Figure 12. SPRT s/n 054, immersion profile in the gallium melting point

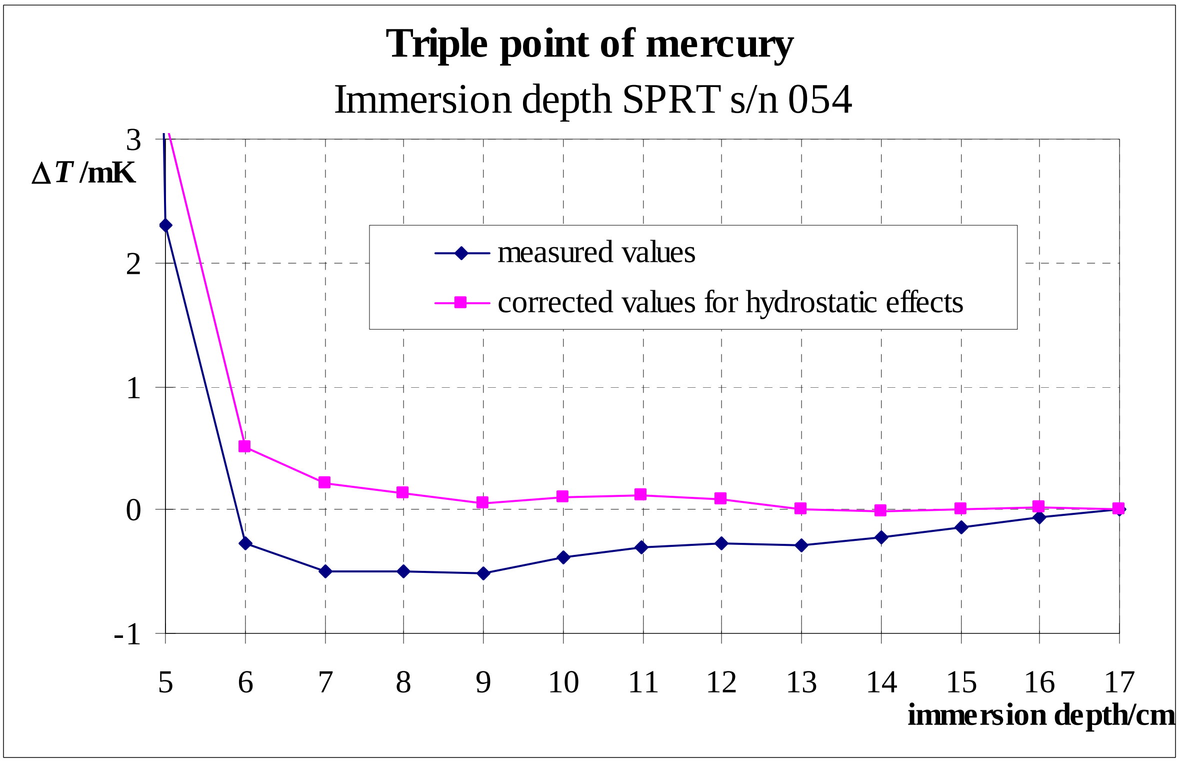

Figure 13. SPRT s/n 054, immersion profile in the mercury freezing point

ANNEX 2. LACOMET FIXED POINT CELLS IMMERSION PROFILES

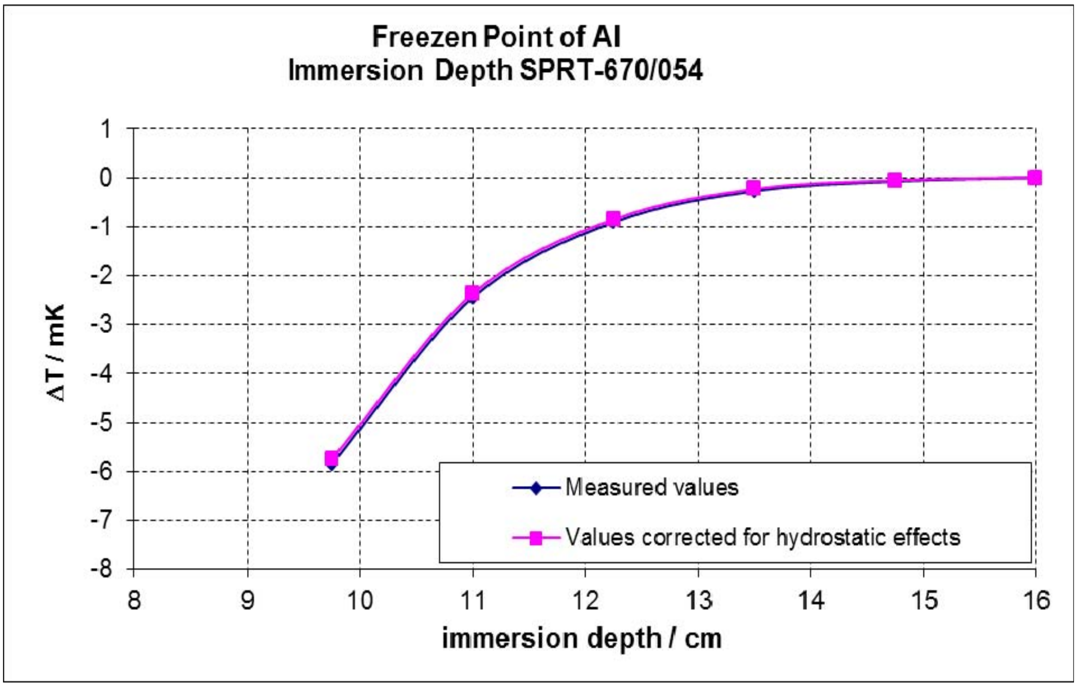

Figure 14. SPRT s/n 244 immersion profile in the aluminium freezing point

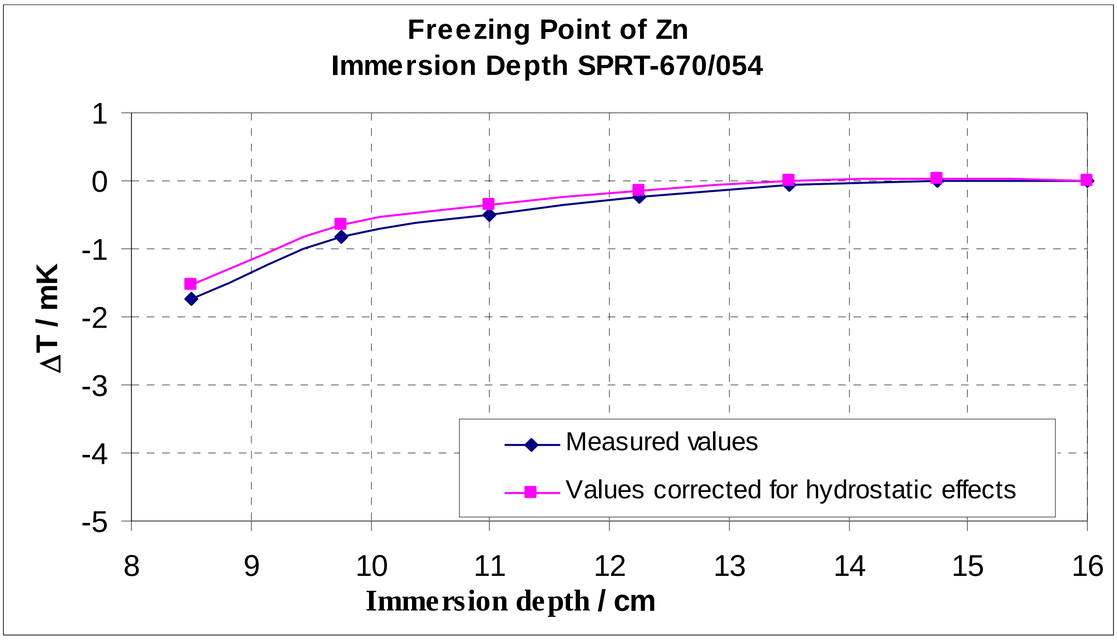

Figure 15. SPRT s/n 054, immersion profile in the zinc freezing point

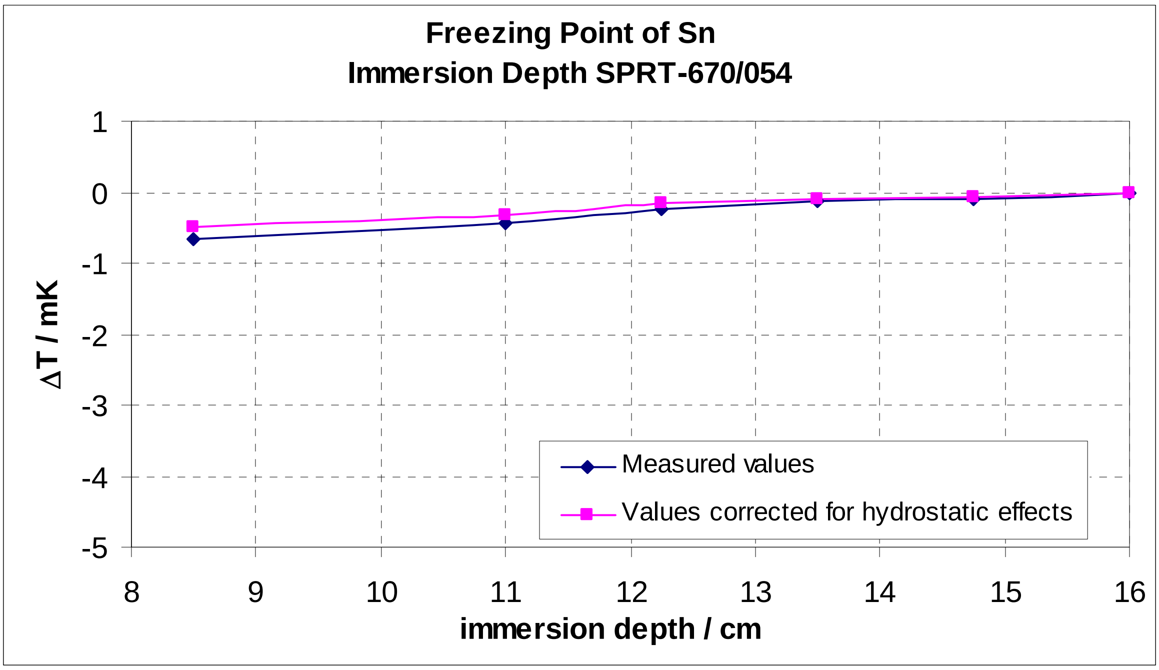

Figure 16. SPRT s/n 054, immersion profile in the tin freezing point

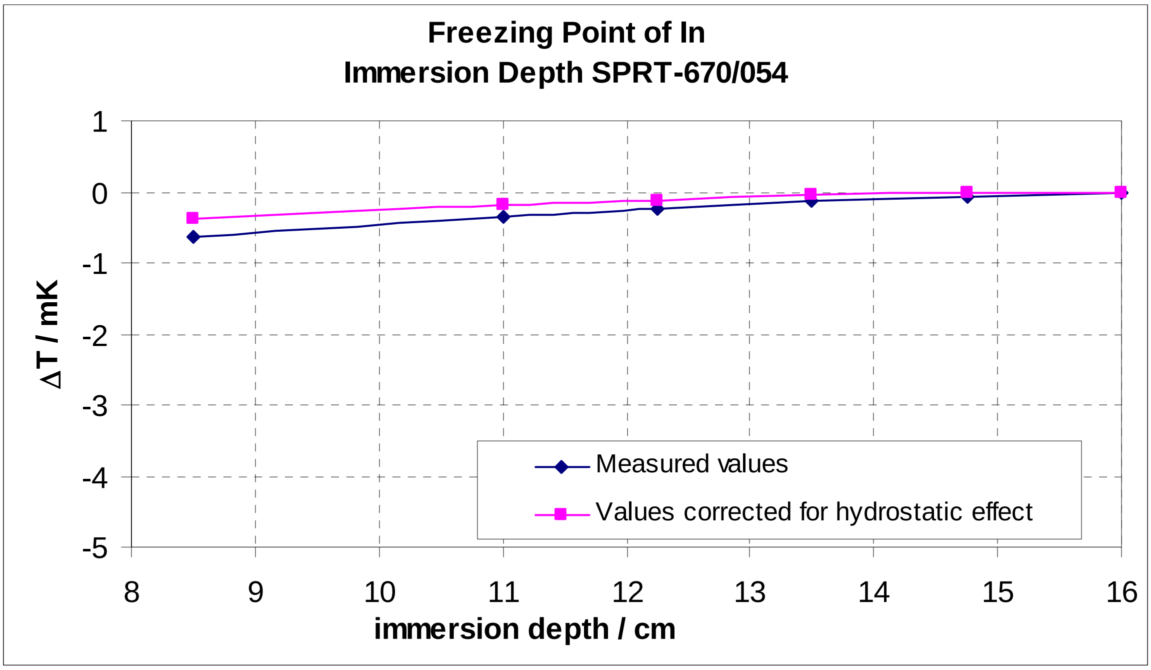

Figure 17. SPRT s/n 054, immersion profile in the indium freezing point

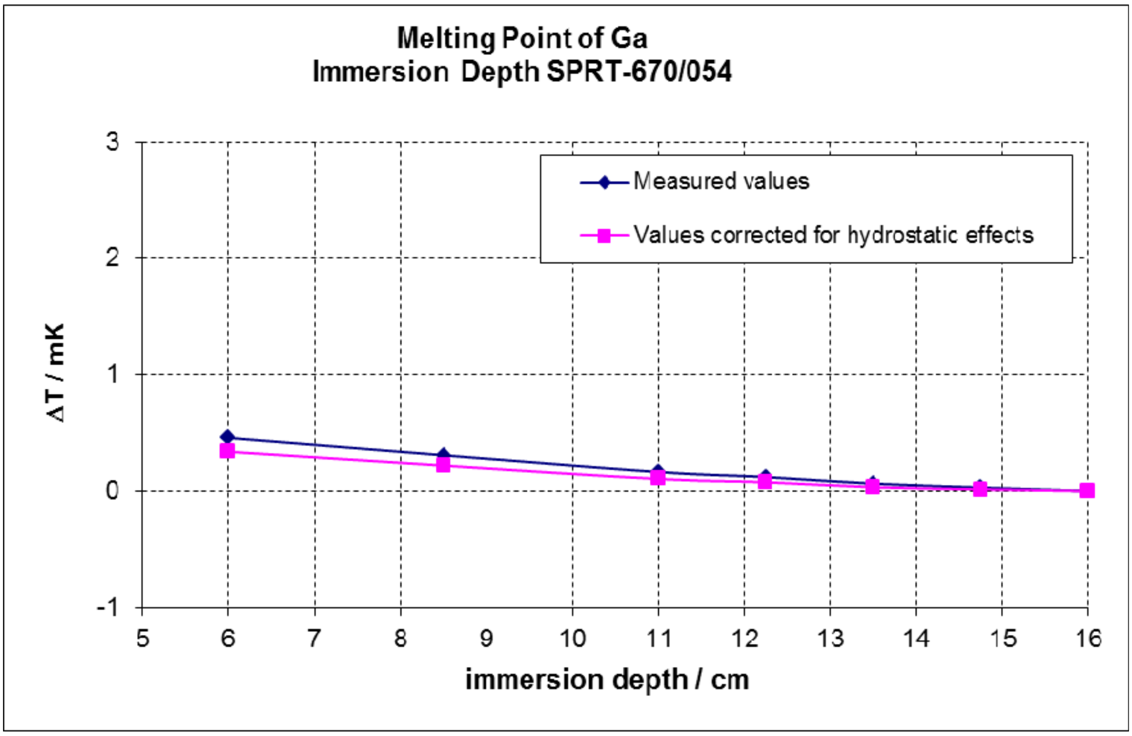

Figure 18. SPRT s/n 054, immersion profile in the gallium melting point

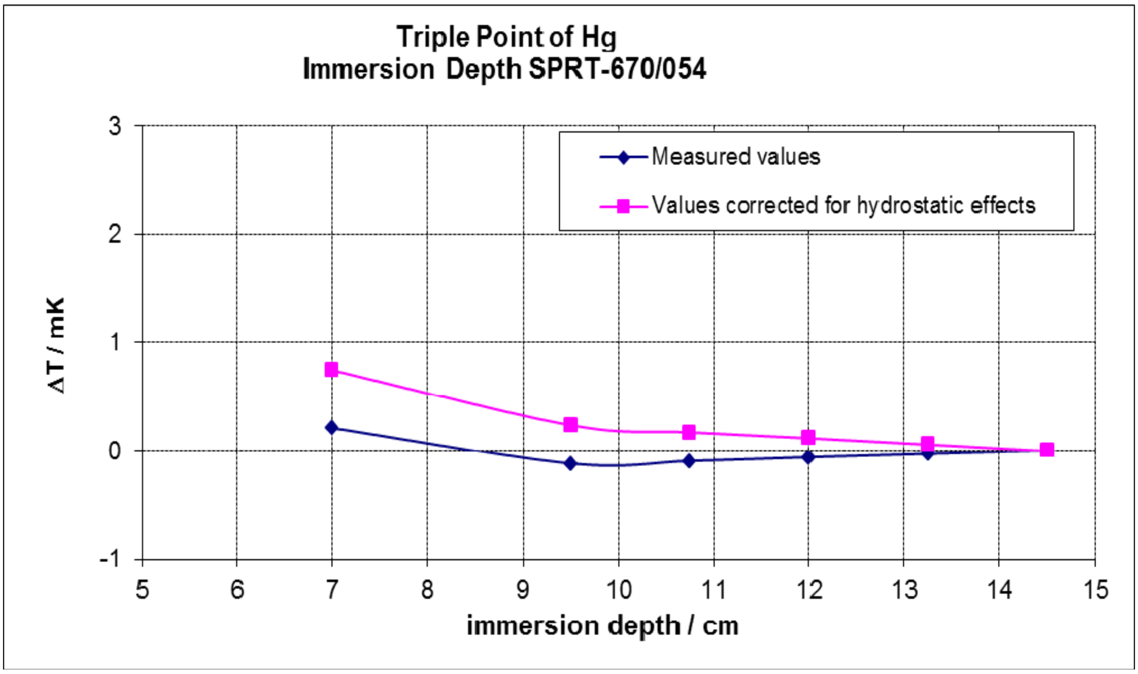

Figure 19. SPRT s/n 054, immersion profile in the mercury freezing point

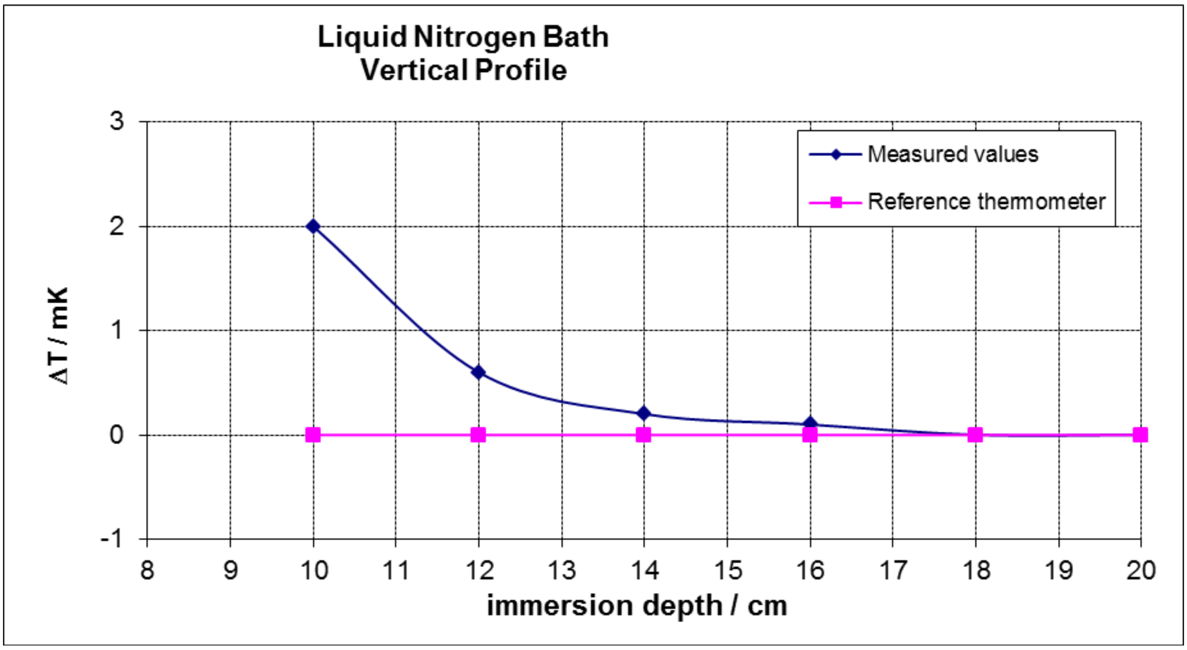

Figure 20. Immersion profile of liquid nitrogen bath

ANNEX 3. CEM FIXED POINT CELLS PHASE TRANSITION CURVES

In order to not disturb the SPRT used as comparison standard, all the fixed point plateaus were recorded using our check SPRT for the corresponding fixed point. The procedure used for the realisation of every fixed point was the same as had been used to make the measurements with the travelling SPRT.

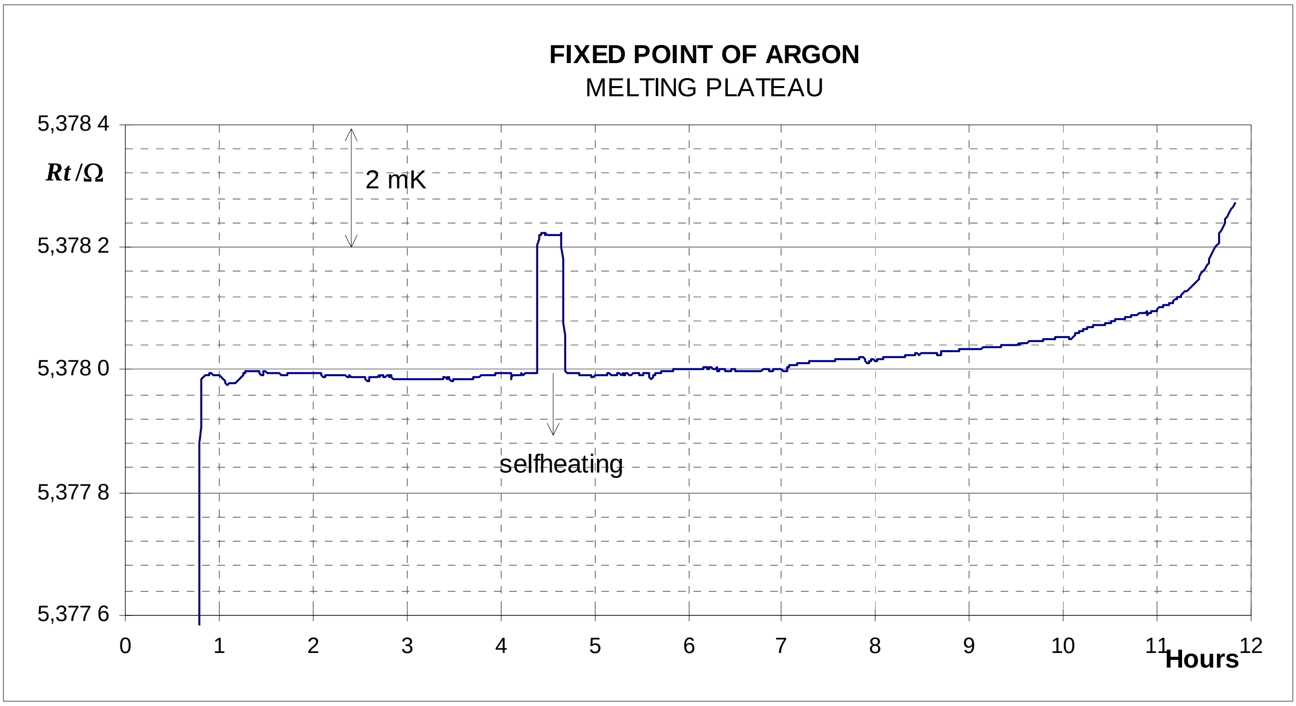

Figure 21. Argon melting plateau

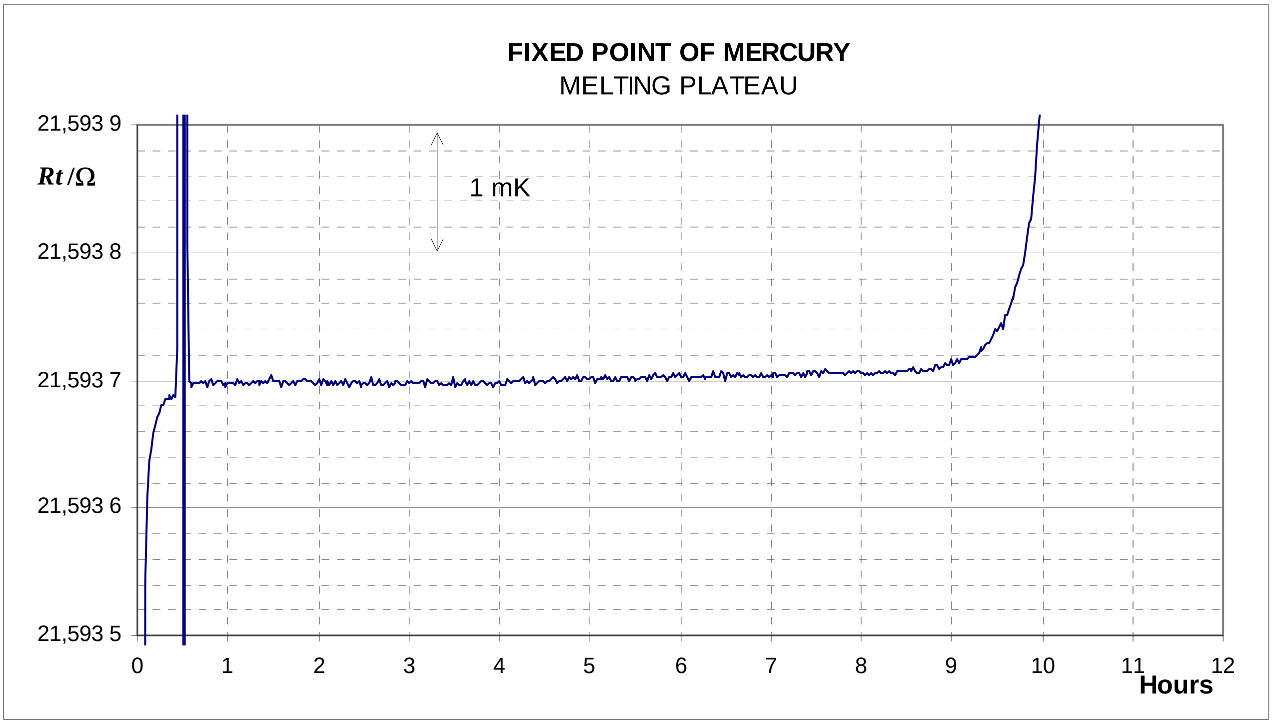

Figure 22. Mercury melting plateau

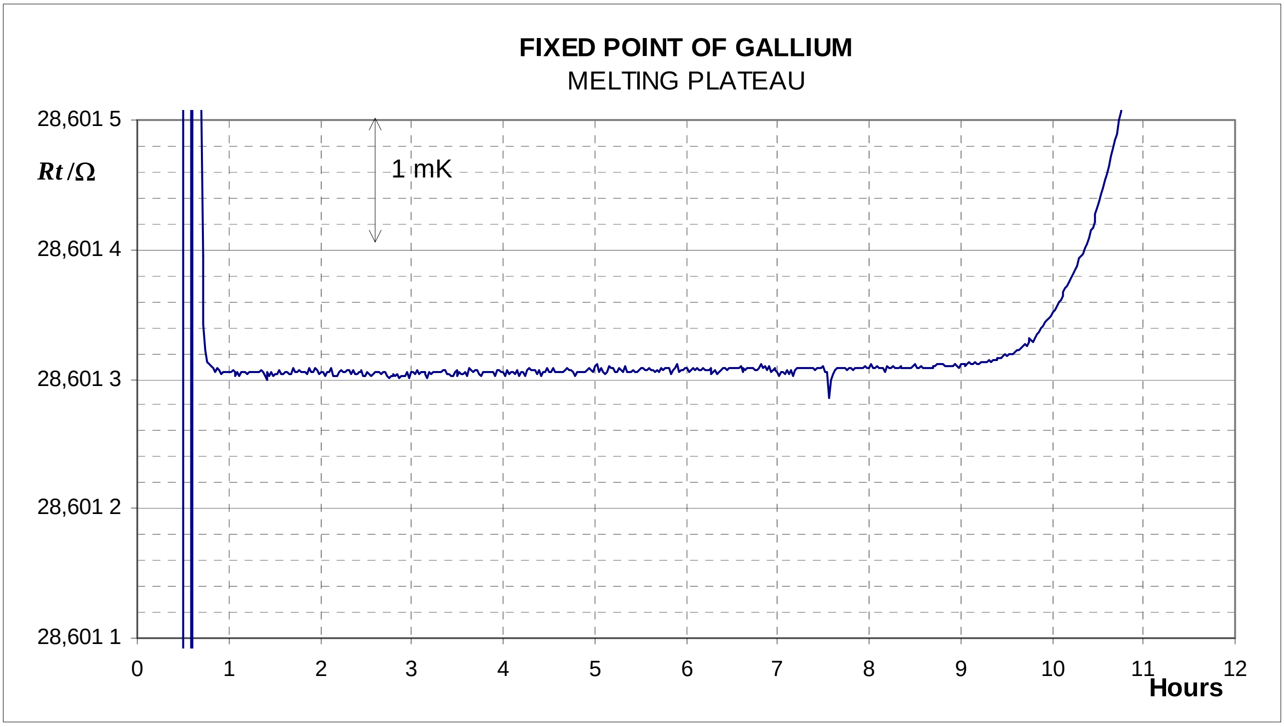

Figure 23. Gallium melting plateau

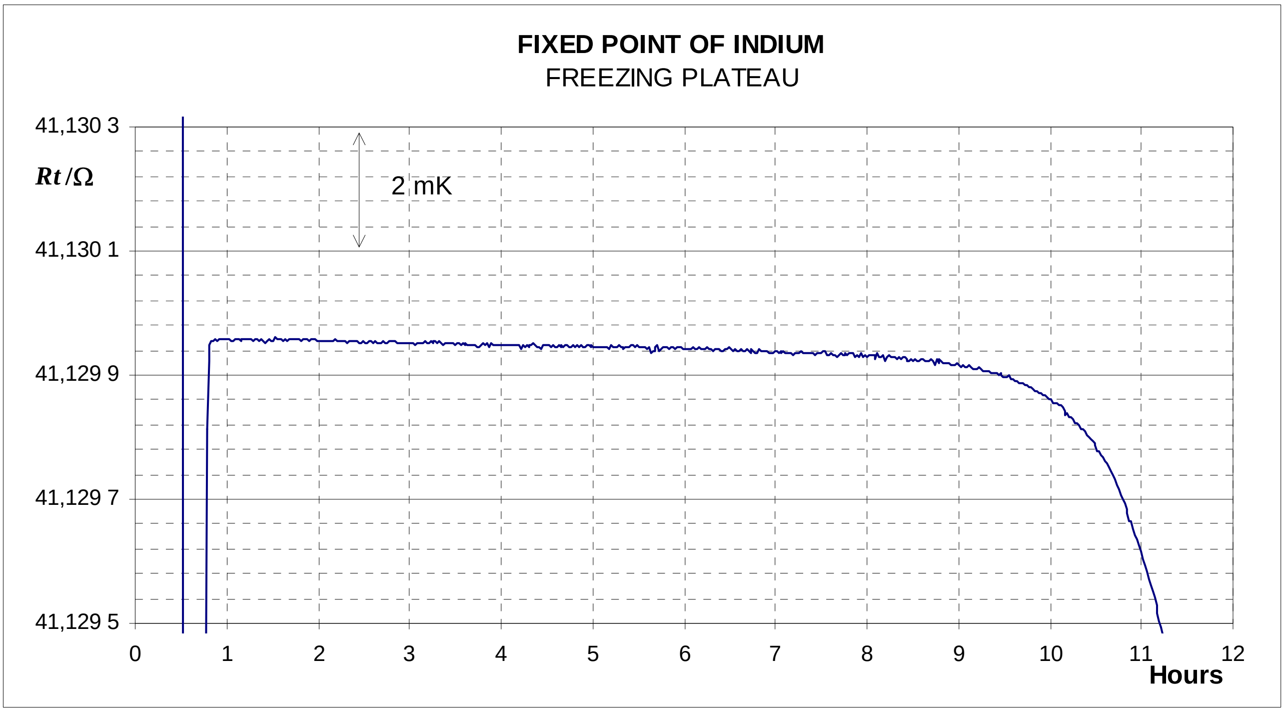

Figure 24. Indium freezing plateau

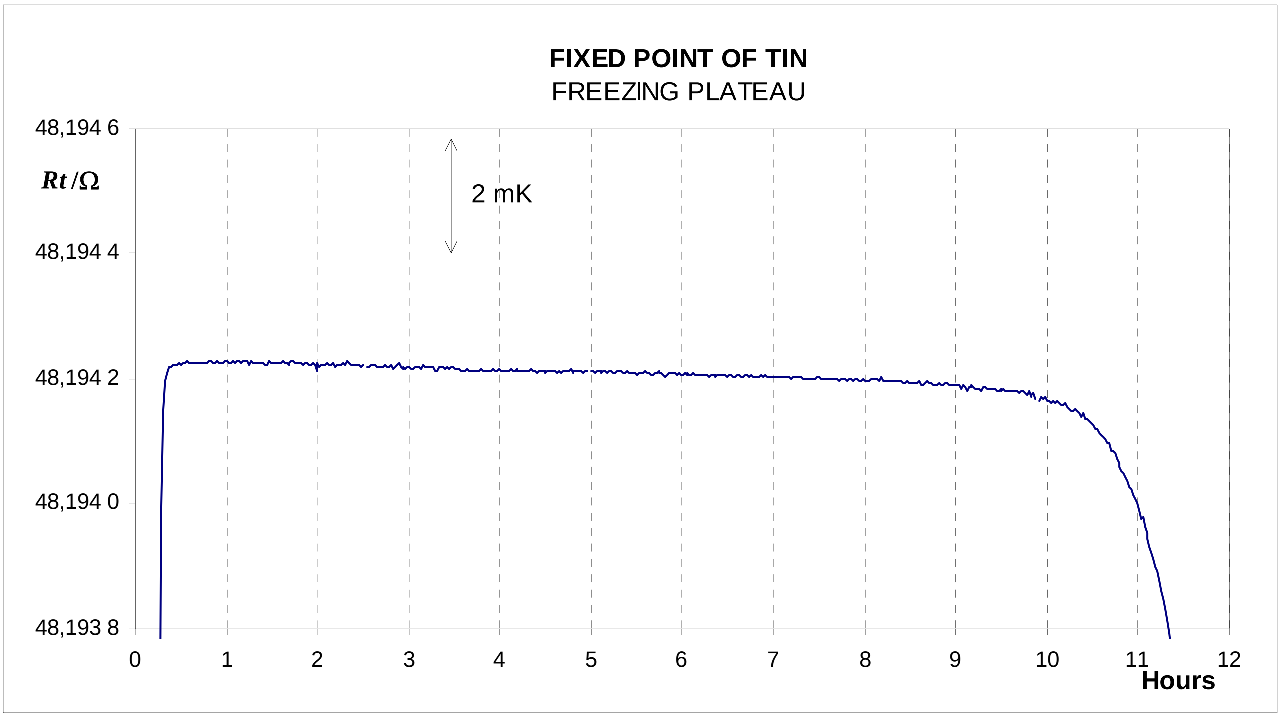

Figure 25. Tin freezing plateau

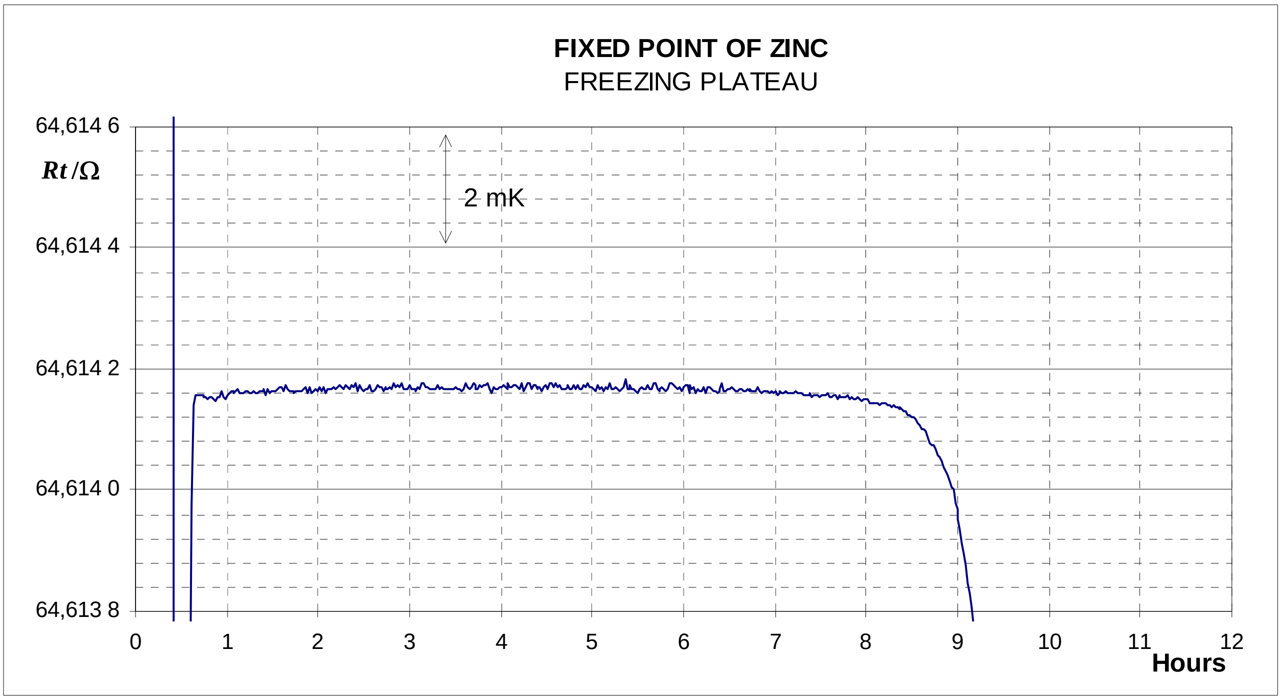

Figure 26. Zinc freezing plateau

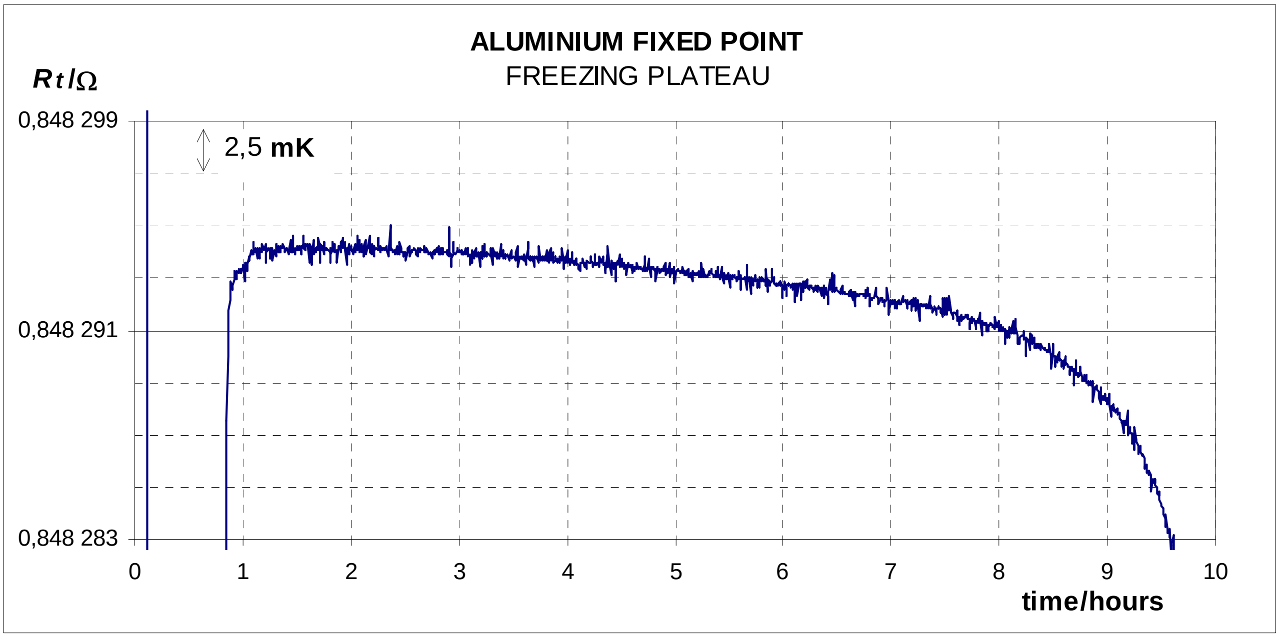

Figure 27. Aluminium freezing plateau

ANNEX 4. LACOMET FIXED POINT CELLS PHASE TRANSITION CURVES

All the fixed point plateaus were recorded using our check SPRT for the corresponding fixed point.

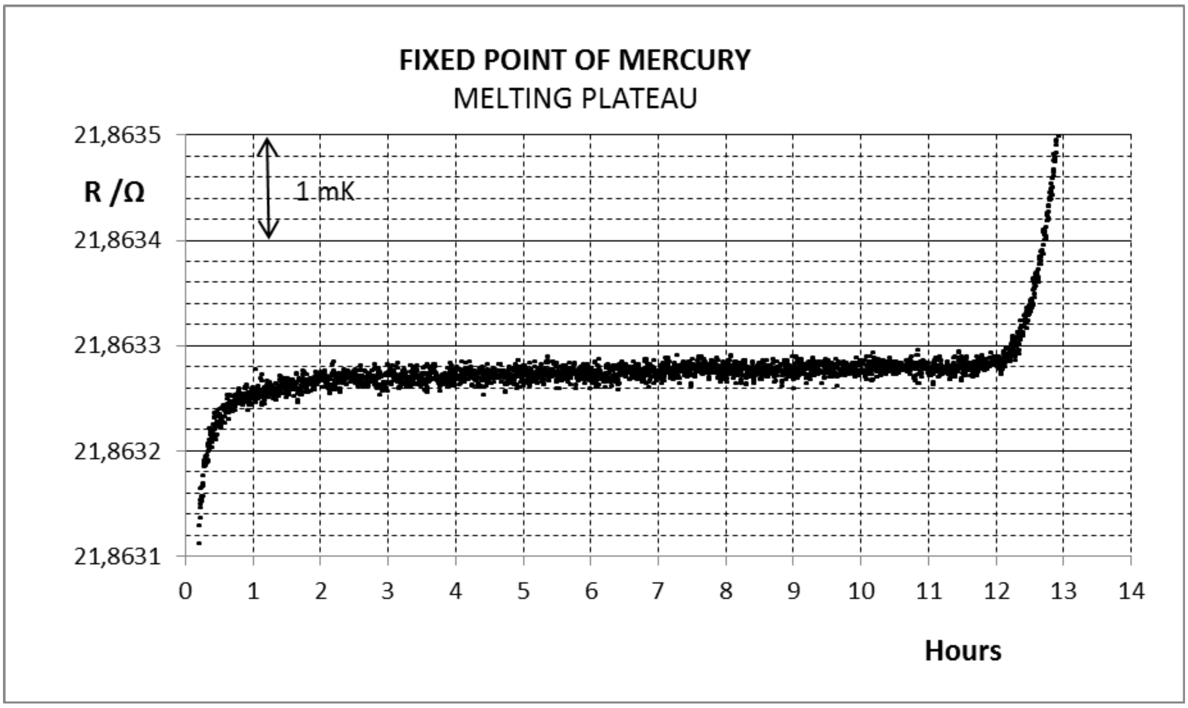

Figure 28. Mercury melting plateau

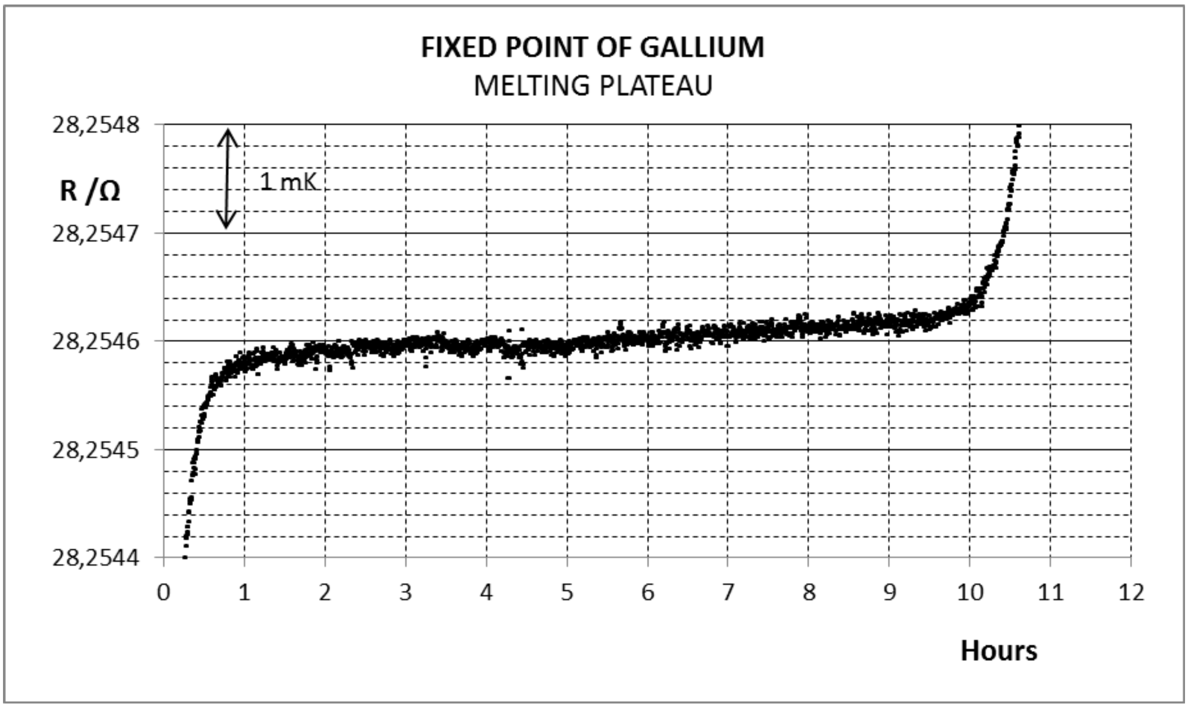

Figure 29. Gallium melting plateau

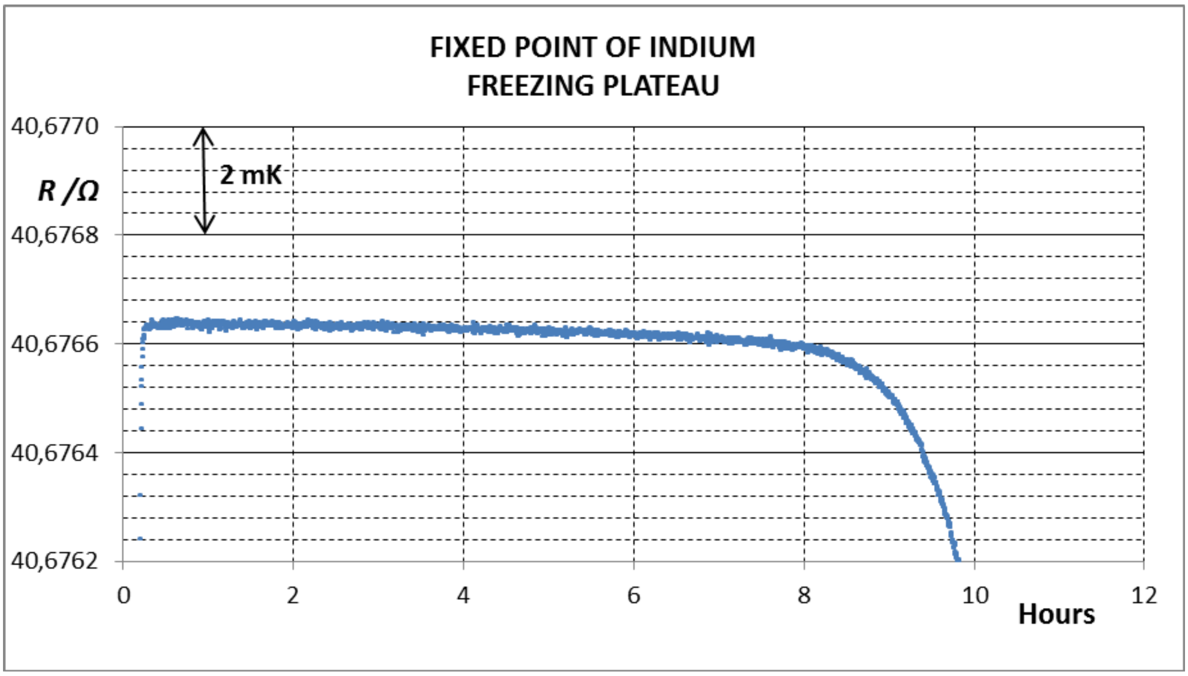

Figure 30. Indium freezing plateau

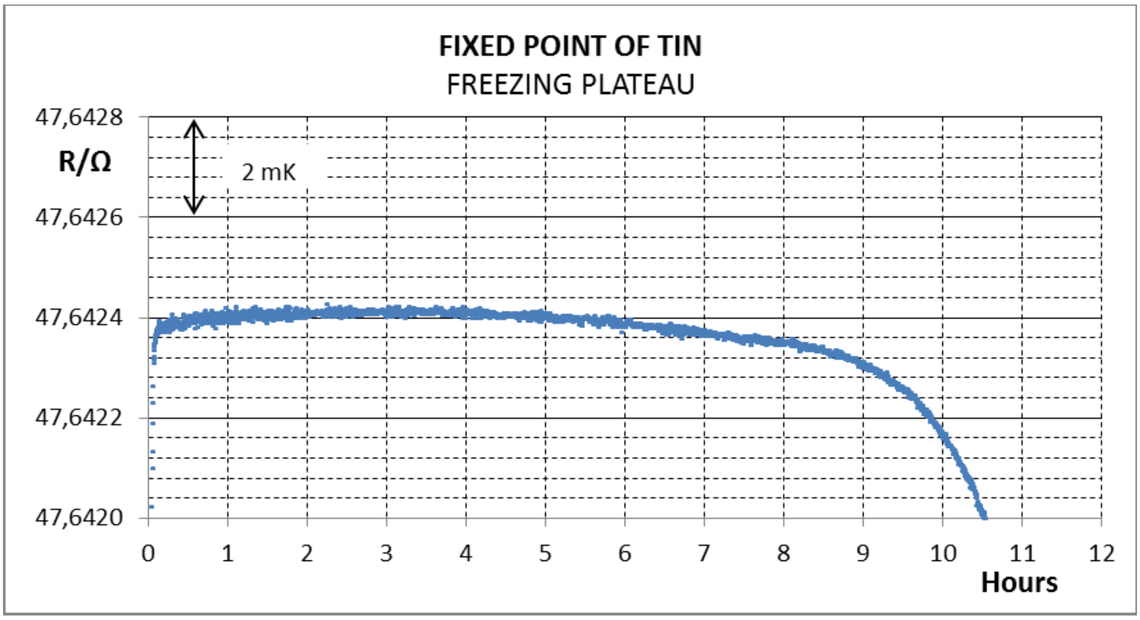

Figure 31. Tin freezing plateau

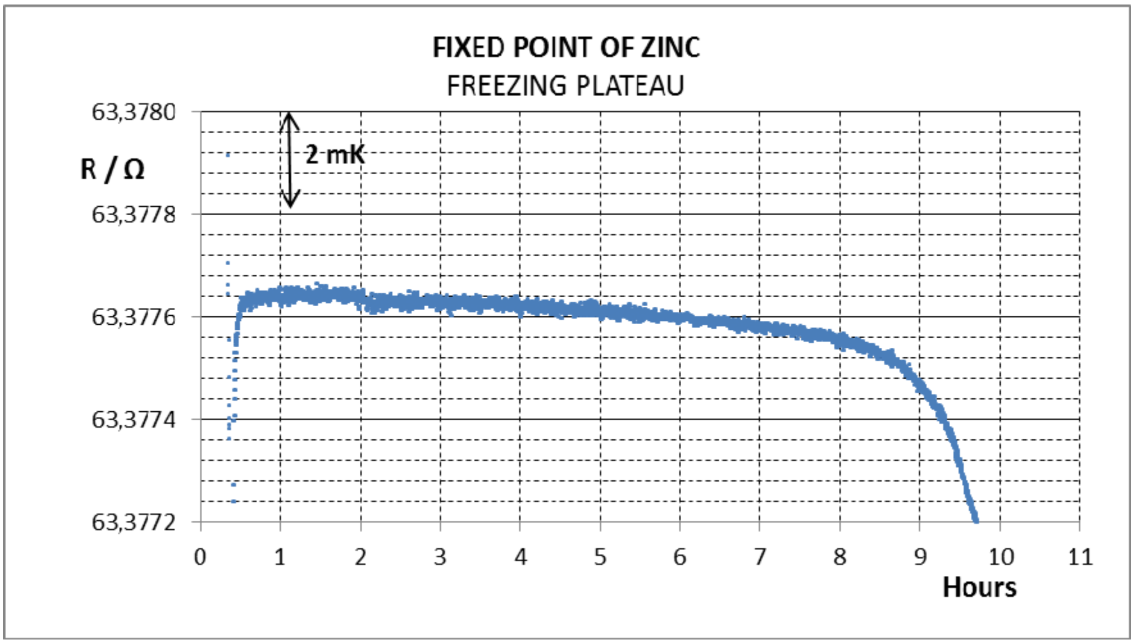

Figure 32. Zinc freezing plateau

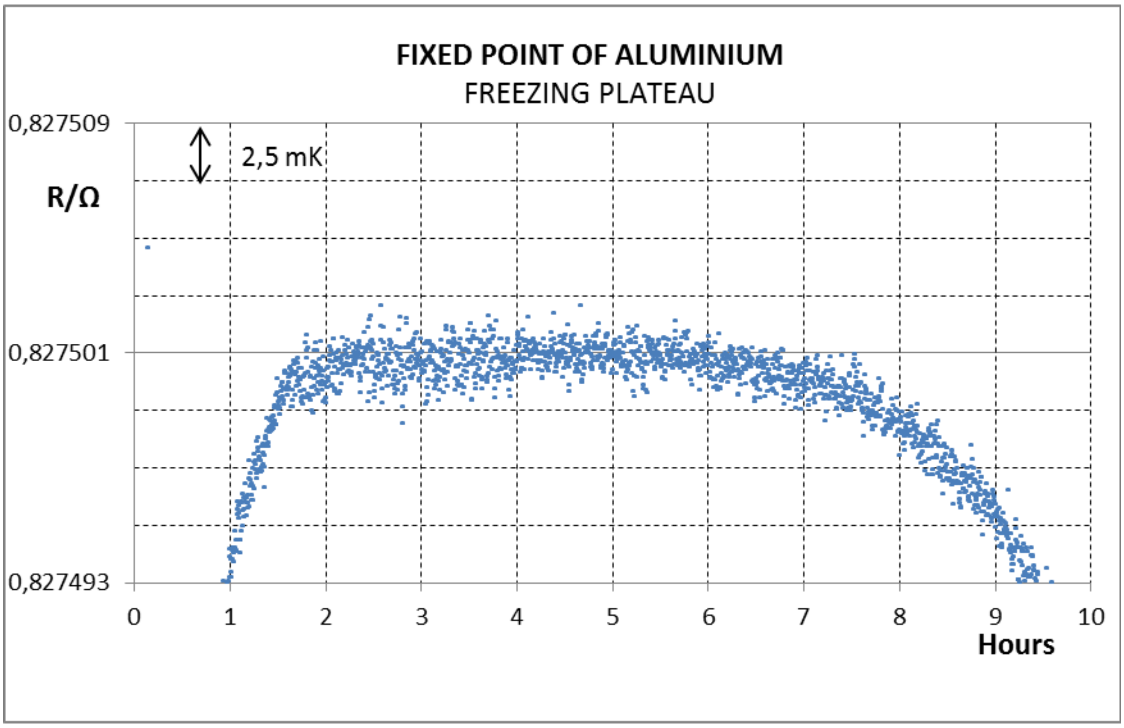

Figure 33. Aluminium freezing plateau

ANNEX 5. PROTOCOL

TECHNICAL PROTOCOL

Comparison of the realisations of the ITS-90 over the range 83,805 8 K to 933,473 K

Version 1
March 2009

Prepared by:
Dolores del Campo
Centro Español de Metrología

1. Introduction

The objective of this project is to compare the realization of the International Temperature Scale of 1990 (ITS- 90) over the range 234,315 6 K (triple point of mercury) to 993,473 K (freezing point of aluminium) in the National Metrology Institutes of Spain (Centro Español de Metrología - CEM) and Costa Rica (Laboratorio Costarricense de Metrología - LACOMET) with the aim to provide support to the Calibration Measurement Capabilities claimed by LACOMET in this range. As CEM participated in the EUROMET.T-K3 and EUROMET.T-K4 comparisons it is possible the linkage with the key comparisons and the calculation of the degrees of equivalence with other laboratories.

There will be an additional comparison point close to the triple point of argon (83,805 8 K) that CEM will realize using an Argon Triple Point Apparatus and LACOMET using a Nitrogen Boiling Point Apparatus to allow LACOMET to support their calibration by comparison capabilities at this temperature.

Both participants of this comparison should follow the instructions, which are given below. Moreover, each laboratory should follow its normal practice when realising the ITS-90. The instructions are based on the protocols of the EUROMET.T-K3 comparison (Appendix A of the Final Report in http://kcdb.bipm.org/AppendixB/appbresults/euromet.t-k3/euromet.t-k3_final_report.pdf), EUROMET.T-K4 (Appendix A of the Final Report in http://kcdb.bipm.org/AppendixB/appbresults/cct-k4/euromet.t-k4_final_report.pdf) and the Guidelines for CIPM key comparisons, Appendix F to the MRA, 1 March 1999. The Pilot Laboratory of this comparison is CEM which will be responsible for the analysis of the results and the preparation of the comparison report. The range of temperature covered in this comparison is from the triple point of Ar (83,805 8 K) to the freezing point of Al (993,473 K) using long-stem SPRTs.

2. The transfer standards

The transfer standards will be two 25 Ω Standard Platinum Resistance Thermometers (SPRT) one for the fixed points of Hg, Ga, In, Sn and Zn and the additional Ar point and another for the freezing point of aluminium. The thermometers will have proven stability and will be provided by LACOMET.

3. Scheme of the organization

LACOMET will measure the two SPRTs following the instructions described in paragraph 4, before CEM measurements. After CEM measurements LACOMET will check the stability of the SPRTs as is described in paragraph 6.

LACOMET will be responsible for the transport of the thermometers according to the procedures required by the Department of Customs of Costa Rica and Spain. Due to the extreme fragility of the SPRTs they will be hand carried with extreme care.

The time allowed for the calibration of the SPRTs will be approximately 9 weeks (see schedule in table 1). In order that calibration results performed by LACOMET do not influence the CEM operator the report of the LACOMET results will be sent to CEM just after the measurements at CEM will have finished.

LACOMET will send their results to CEM within 1 month since the finish of the final checking of the SPRTs. CEM will prepare the Draft A of the final Report within 1 month of the receipt of the LACOMET report of results.

Date	Laboratory	Action
27th July 2009	LACOMET	Start of measurements
18th September 2009	LACOMET	End of measurements
21th - 24th September 2009	LACOMET	SPRTs transported to CEM
28th September 2009	CEM	Start of measurements
27th November 2009	CEM	End of measurements
30th November – 4th December 2009	CEM/LACOMET	SPRTs transported to LACOMET
7th – 18th December 2009	LACOMET	Checking of the SPRT's stability

Table 1. Schedule of the comparison

4. Measurement procedure

The resistance of the travelling SPRTs should always be measured at two measuring currents, in order to determine the zero-power value. 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.

4.1. 25 Ω SPRT (fixed points Hg, Ga, In, Sn, Zn and additional Ar point)

4.1.1. Stabilization procedure

Before starting measurements at the fixed points, the SPRT 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 resistance at TPW increases after annealing repeat steps 2 to 5.

    - If the decrease of the resistance value at TPW is equivalent to 0,3 mK or greater repeat steps 2 to 5.

    - If the decrease of the resistance value at TPW is less than 0,3 mK the calibration can be performed

4.1.2. 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, Zn, TPW, Sn, TPW, In, TPW, Ga, TPW, Hg, TPW, Ar[1] and TPW.

Both laboratories have to follow their normal practice when realizing the ITS-90. For each fixed point the value of the reduced resistance $W=R_T / R_{TPW}$ is calculated, being R_{TPW} the TPW resistance value obtained immediately after the measurement of R_T. R_T 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 freezings for Zn, Sn, In, 2 meltings for Ga, 2 triple points for Hg and Ar) 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

[1] CEM will perform the measurements at the triple point of argon, LACOMET will perform a calibration by comparison measurement point close to the Ar triple point using its nitrogen boiling point apparatus as described in 4.1.3.

measurements will be reported in a graph where the theoretical d*T*/d*h* curve, using the hydrostatic pressure coefficients (mK/m of liquid) given in the ITS-90 text, and the measured d*T*/d*h* curve will be plotted.

4.1.3. Measurements to be performed by LACOMET close to the triple point of Argon

LACOMET will use its nitrogen boiling point apparatus to perform a calibration by comparison measurement point close to the triple point of Argon, using as reference standard another SPRT calibrated by comparison at the boiling point of nitrogen (-195.798 ºC). Two different results measured in two different days will be carried out.

In this case an immersion depth graph will also be provided to assess the heat conduction of the apparatus.

4.2. 25 Ω SPRT (fixed point of Al)

In order to avoid any damage of the SPRT 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.

4.2.1. Stabilization procedure

Before starting measurements at the fixed point of aluminium, 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.

4.2.2. Measurement procedure

Measurements at the fixed point of aluminium should be performed in at least 2 different phase transitions (2 freezing points of aluminium). Both laboratories have to follow their normal practice when realizing the ITS-90. The value of the reduced resistance $W = R_T / R_{TPW}$ is calculated, being R_{TPW} the TPW resistance value obtained immediately after the measurement of R_T. R_T and R_{TPW} should have been corrected for self-heating, hydrostatic head and if any the pressure effect. The different values will be delivered together with the calculated mean. The sequence of the operations for each plateau 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 aluminium freezing point cell and calibrated in the stable plateau of the freezing curve of aluminium.

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 (R_{TPW}).

For the aluminium cell used in the comparison, It has to be determined (using the circulating SPRT) and plotted the change of phase transition temperature, d*T*, versus immersion depth, d*h*. On the same graph, it should be plotted the theoretical d*T*/d*h* curve using the hydrostatic pressure coefficients (mK/m of liquid) given in the ITS-90 text.

5. Final check of the thermometers

Once the SPRTs are back to LACOMET, it is necessary to check their stability. To do so, LACOMET will carry out the ensuing actions:

- Stabilization of the 25 Ω SPRT used for the fixed points of Hg, Ga, In, Sn , Zn and the additional Ar point as in 4.1.1

- Measurement of the *W* at the freezing point of zinc following 4.1.2.

- Stabilization of the 25 Ω SPRT used for the Al fixed point as in 4.2.1

- Measurement of the *W* at the freezing point of aluminium following 4.2.2.

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", ISO 1993, ISBN 92-67-10188-9 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
- Uncertainty propagated from the TPW
- SPRT self heating errors
- 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)

In case of the calibration by comparison measurement point close to the triple point of Argon, LACOMET will take into account, at least, the following measurement uncertainties:

- Uncertainties coming from the SPRT used as reference
  - Calibration
  - Drift
- Bridge measurement errors:
  - effects of changes in reference resistors
  - non-linearity of bridge
  - quadrature effects in ac measurements

- Uncertainty propagated from the TPW
- SPRT self heating errors
- Uncertainties due to the interpolation at the Ar fixed point.
- Uncertainties coming from the isothermal enclosure:
    - Stability
    - Uniformity (vertical and axial)

7. Report of Results

Participants are requested to use the formats included in the appendix to present their individual measurements that will be provide by the pilot as EXCEL sheets.

For each fixed point cell used in the comparison, a graph where is plotted the measured and theoretical d*T*/d*h* 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.

APPENDIX

FORMATS

Instrumentation Details

Bi-Lateral comparison CEM-LACOMET

Laboratory name	
Bridge	
Manufacturer	
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	
Manufacturer / type	
Reference resistor temperature control (yes or not)	
If yes, How?	
TPW Cell	
Manufacturer / 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	
Manufacturer / 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	
Manufacturer / 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 sensible element/cm	
Zn Furnace	
Type (1 zone, 3 zones, heat pipe, …….)	
Typical duration of the melting / freezing plateaux	
Sn Cell	
Manufacturer / 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	

Instrumentation Details

Bi-Lateral comparison CEM-LACOMET

Laboratory name	
In Cell	
Manufacturer / 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	
Manufacturer / 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	
Manufacturer / 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 (cryostat, bath, …….)	
Typical duration of the melting / freezing plateaux	
Ar Cell	
Manufacturer / 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	
Ar cryostat	
Type (cryocooler, bath,..)	
Typical duration of the melting plateau	
N Boliling Point Apparatus	
Manufacturer / model	
Typical duration of the melting plateau	
SPRT used as reference manufacturer/model	
Calibrated at (Laboratory name)	
Calibration date	

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Calibration data

Bi-Lateral comparison CEM-LACOMET

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						
	Ar						
	TPW						
	Ar						
	TPW						
	Average of W for Ar						

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						

Uncertainty analysis

Bi-Lateral comparison CEM-LACOMET

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

Quantity Q_i	Components	Standard uncertainty $u_{(Qi)}$	Degrees of freedom components evaluated by a type A method * ν_i	Sensitivity coefficient	Uncertainty contribution u_i in mK
Combined uncertainty					
Effective degrees of freedom					
Expanded uncertainty					

* for type B method the number of degres of freedom will be considered as being infinite