VSL
The Netherlands

BIM
Bulgaria

Bilateral Comparison of the realisations of the ITS-90 at the fixed points of Hg, H_20, Ga, Sn and Zn EURAMET.T-K3.1

Final Report

Date: 21.11. 2012

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1. Introduction

A bilateral comparison has been organized between VSL, The Netherlands and BIM, Bulgaria, of the realisations of the international temperature scale ITS-90 at the fixed points of Hg, H_20, Ga, In, Sn and Zn. This comparison is registered as EURAMET project T-K3.1 in the BIPM key comparison database.

The aim of the comparison is to demonstrate the calibration measurement capabilities (CMC's) of BIM in this working field. If this comparison is successful, the CMC's for BIM will be included in Appendix C of the CIPM mutual recognition arrangement (MRA).

The results of this comparison will be linked to CCT-K3 comparison.

This comparison is organized in the framework of Phare project BG 2005/017-353.02.02, Lot 1, and is in this framework financed by the EU. This project runs from March 2008 to the end of February 2009.

This bilateral comparison is intended to compare BIM and VSL realization of the ITS-90. The range of temperature covered in this comparison is from the triple point of Hg (234,3156 K) to the to the freezing point of Zn (692,677 K) using long-stem SPRT.
The laboratories calibrated 1 SPRT. The SPRT used for this comparison was selected for its very good stability.

2. Participants and organisation of the comparison

2.1. List of participants

Participants:

Name:	VSL, Dutch Metrology Institute	
Acronym:	VSL	
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Country:	The Netherlands	
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Name:	Bulgarian Institute of Metrology, National Centre of Metrology	
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Comparison coordinator:
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2.2. Comparison schedule

The travelling standard was circulated in a schedule A-B-A, as shown in the table below. The behaviour of the standard during the comparison was determined from the measurements of participant A.

Each participant was allowed approximately 4 weeks to perform the measurements.

Long-stem standard platinum resistance thermometer		
Participant	Measurements from	to
VSL	08-08-2008	24-09-2008
BIM	22-10-2008	17-11-2008
VSL	20-01-2009	20-02-2009

The travelling standard was hand-carried from one laboratory to the other by experts involved in the Phare project.

3. Travelling standard and measurement instructions

3.1. Description of the standard

The transfer standard is a long-stem standard platinum resistance thermometer provided by VSL:
Manufacturer: Leeds & Northrup
Type: 8167-25 Ω
Serial number: 1773051

3.2. Quantities to be measured and conditions of measurement

The quantity to be measured is resistance of the transfer standard (SPRT) in the fixed points of Mercury, TPW, Gallium, Indium, Tin and Zinc.
For each fixed point the $W=R_{T}/R_{TPW}$ is calculated.
Ambient conditions:

- Temperature – from 21 °C to 24 °C
- Relative humidity – from 30 % rh to 45 % rh

3.3. Measurement instructions

The travelling SPRT is to pass through the following sequence:

1) a measurement at the triple point of water (TPW)
2) a stabilisation procedure
3) a second measurement at the triple point of water
4) measurements at metal fixed points in order of decreasing temperatures alternating with a measurement at the triple point of water.

- The SPRT must be inspected for damage.
- The host must measure the resistance of the travelling SPRT in a TPW cell 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. The 0 mA resistance values of the travelling SPRT at the TPW must be corrected for the hydrostatic head to obtain R_{TPW}. The value of R_{TPW} must be communicated to the pilot laboratory. After receiving approval from the pilot laboratory to proceed with the comparison, the host laboratory can begin the SPRT stabilization procedure:

- Carefully insert the SPRT into a furnace at 480 °C.
- Anneal the SPRT for two hours at 480 °C
- Carefully remove the SPRT from the furnace directly to the room environment.
- Re-determine The value of R_{TPW}

- If the resistance at TPW increases after annealing contact pilot laboratory for further instructions
- If the decrease in the calculated TPW resistance of the SPRT after annealing is equivalent to 0.5 mK or greater proceed to a second SPRT stabilization procedure. Re-determine the value of R_{TPW}. If the decrease in the calculated TPW resistance of the SPRT after second annealing is greater to 0.2 mK communicate with pilot laboratory for further instructions
- If the decrease is less then 0.5 mK the completed calibration can be performed.

Calibrate the SPRT at all of the fixed points in the range of comparison, i.e., measurements at TPW, Zn, TPW, Sn, TPW, In, TPW, Ga, TPW, Hg, and TPW, in that order. Existing techniques as practised by the participating Laboratory must be used. For each metal fixed point the $W=R_T/R_{TPW}$ is calculated. R_{TPW} is the TPW resistance 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 3 different phase transitions (3 freezings for Zn, Sn, In, 3 meltings for Ga and 3 triple points for Hg will be performed. The different values will be delivered together with the calculated mean.

3.4. Deviations from the protocol

There have been some deviations from the original schedule. The actual schedule is given in section 2.2 of this document.

4. Methods of measurement

Resistance of the transfer standard (SPRT) in the fixed points is measured.
For each fixed point the $W=R_T/R_{TPW}$ is calculated. R_{TPW} is the TPW resistance obtained immediately after the measurement of R_T.

4.1. Method of BIM

4.1.1. Standard equipment

Set of fixed points:

- Hg cell: Hg 203/Isotech
- H_2O cell: A11/50 583/Isotech
- Ga cell: Ga 414/Isotech
- In cell: In 138B/Isotech
- Sn cell: Sn 181/Isotech
- Zn cell: Zn 218B/Isotech

4.1.2. Method of measurement

The resistance of the transfer standard (SPRT) in the triple point of water is measured at two measuring currents – 1 mA and $\sqrt{2}$ mA (in order to determine the zero-power value).
A stabilization procedure is made – the SPRT is annealed for two hours at 450 °C. After annealing the value of R_{TPW} is re-determined.

The SPRT is calibrated at all of the fixed points in the range of comparison, i.e., measurements at TPW, Zn, TPW, Sn, TPW, In, TPW, Ga, TPW, Hg, and TPW, in that order. The resistance of the transfer standard R_T is corrected for self-heating and hydrostatic head for each fixed point. The $W=R_T/R_{TPW}$ is calculated.
Three different phase transitions for each fixed point are performed. The report only contains the mean values.

4.2. Method of VSL

4.2.1. Standard equipment

Set of fixed points manufactured in-house.

- Hg cell: VSL89T010
- H2O cells: VSL03T029, VSL06T003 and VSL06T004
- Ga cells: VSL89T020 and VSL89T021
- In cells: In1 (VSL89T056) and In3 (VSL04T233)
- Sn cell: Sn3 (VSL04T104)
- Zn cell: Zn3 (VSL04T073)

4.2.2. Method of measurement

The resistance of the transfer standard (SPRT) in the triple point of water is measured at two measuring currents – 1 mA and $\sqrt{2}$ mA (in order to determine the zero-power value).
A stabilization procedure is made – the SPRT is annealed for two hours at 450 °C. After annealing the value of R_{TPW} is re-determined.
The SPRT is calibrated at all of the fixed points in the range of comparison, i.e., measurements at TPW, Zn, TPW, Sn, TPW, In, TPW, Ga, TPW, Hg, and TPW, in that order. The resistance of the transfer standard R_T is corrected for self-heating and hydrostatic head for each fixed point. The $W=R_T/R_{TPW}$ is calculated.
Three different phase transitions for each fixed point are performed. The report only contains the mean values.

5. Measurement results

5.1. Results of the participants

The results from both laboratories with the expanded uncertainties are given in Table 1.

Table 1.

Point	VSL beginning		BIM		VSL end	
	W	$U_{(T_{VSL})}$, mK	W	$U_{(T_{BIM})}$, mK	W	$U_{(T_{VSL})}$, mK
Hg	0,84416222	0,20	0,8441618	0,44	0,84416239	0,20
Ga	1,11812106	0,33	1,1181211	0,38	1,11812066	0,33
In	1,60970336	0,42	1,6097056	0,78	1,60970386	0,42
Sn	1,89265596	0,56	1,8926577	0,80	1,89265688	0,56
Zn	2,56865977	0,88	2,5686586	1,16	2,56866167	0,88

The results of VSL in the Table 1 are based on the set of cells used in EUROMET.T-K3.1. But the set of VSL fixed point cells used in EUROMET.T-K3 was not the same as the set of fixed point cells used in EURAMET.T-K3.1. The difference (at each fixed point) between the temperature realized in EUROMET.T-K3 and the temperature realized in EURAMET.T-K3.1 is determined knowing the differences between all cells used as follows (from VSL internal inter-comparisons):

Fixed point	**T_{VSL}(EURAMET.T-K3.1) - T_{VSL}(EURAMET.T-K3) /µK**	**$\delta W(FP_i) = W(FP_i)_{EURAMET.T-K3.1} - W(FP_i)_{EURAMET.T-K3}$**
Hg	0	-1,61775·10-7
H_2O	48	0
Ga	0	-2,14277·10-7
In	-130	-8,02496·10-7
Sn	-420	-1,92156·10-6
Zn	-50	-6,66908·10-7

Considering all the corresponding corrections, the results from both laboratories with the expanded uncertainties are given in Table 1A.

Table 1A.

Point	VSL beginning		BIM		VSL end	
	W	$U_{(T_{VSL})}$, mK	W	$U_{(T_{BIM})}$, mK	W	$U_{(T_{VSL})}$, mK
Hg	0,84416238	0,20	0,8441618	0,44	0,84416255	0,20
Ga	1,11812127	0,33	1,1181211	0,38	1,11812087	0,33
In	1,60970416	0,42	1,6097056	0,78	1,60970466	0,42
Sn	1,89265788	0,56	1,8926577	0,80	1,89265880	0,56
Zn	2,56866044	0,88	2,5686586	1,16	2,56866234	0,88

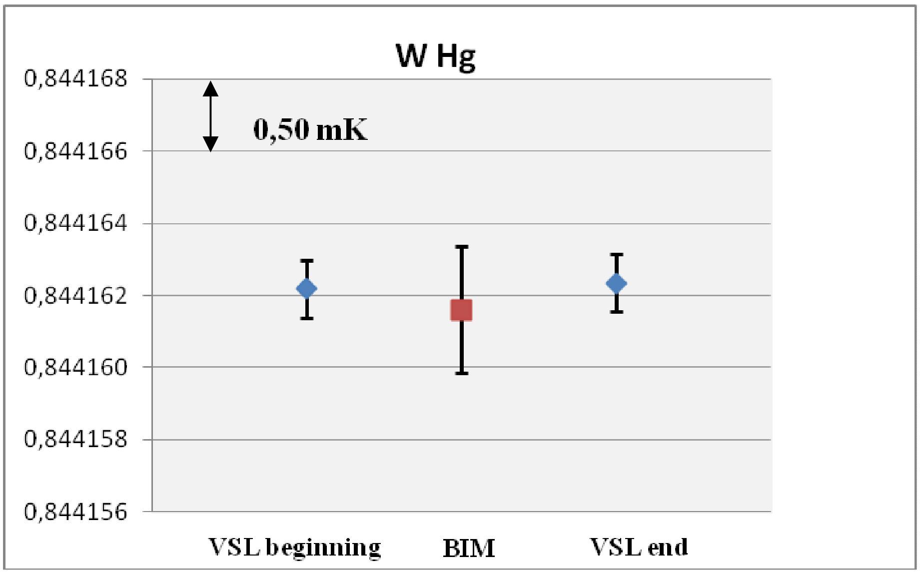

Fig 1. Results of SPRT calibration – Hg

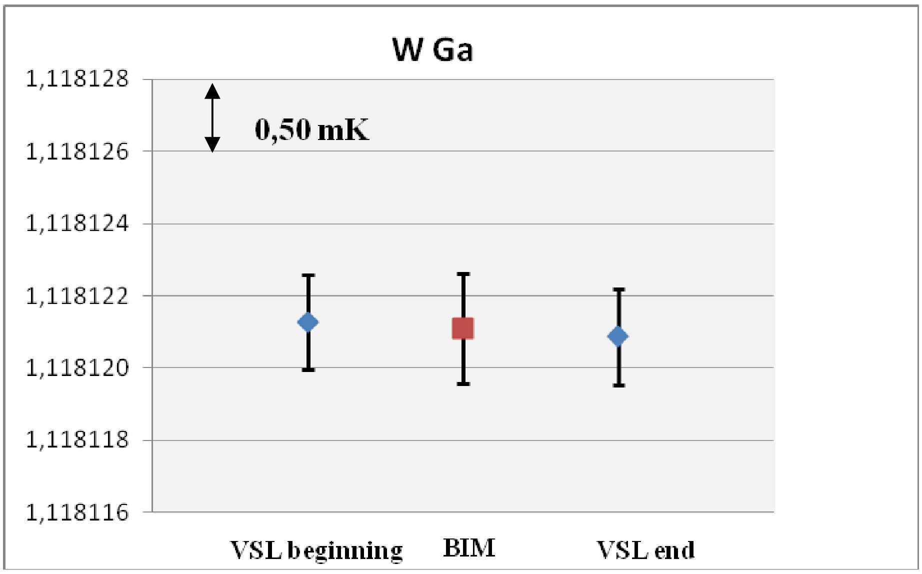

Fig 2. Results of SPRT calibration – Ga

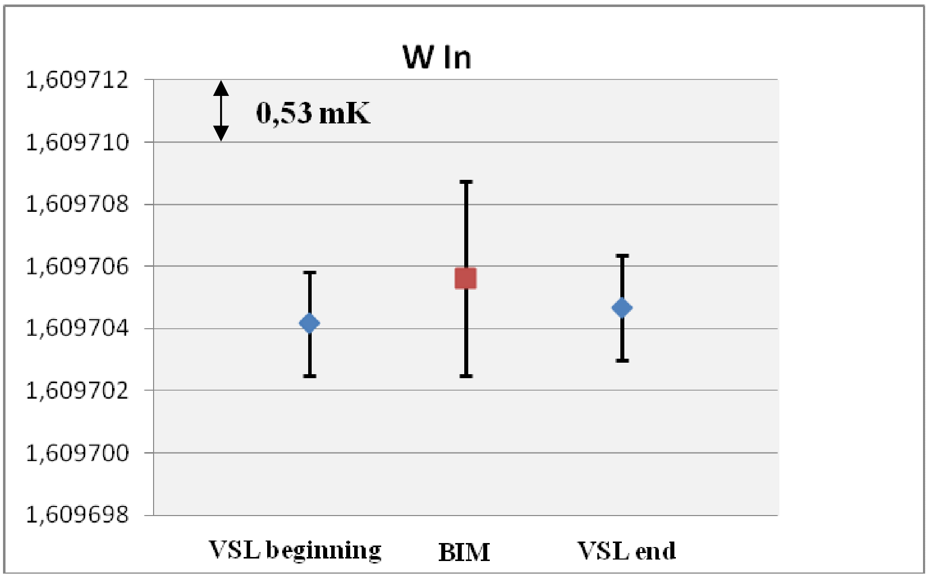

Fig 3. Results of SPRT calibration – In

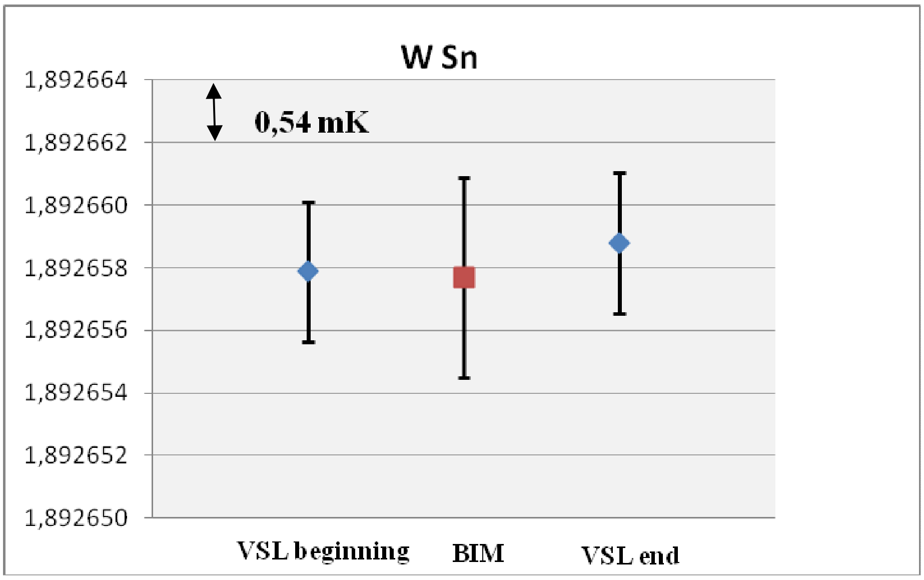

Fig 4. Results of SPRT calibration – Sn

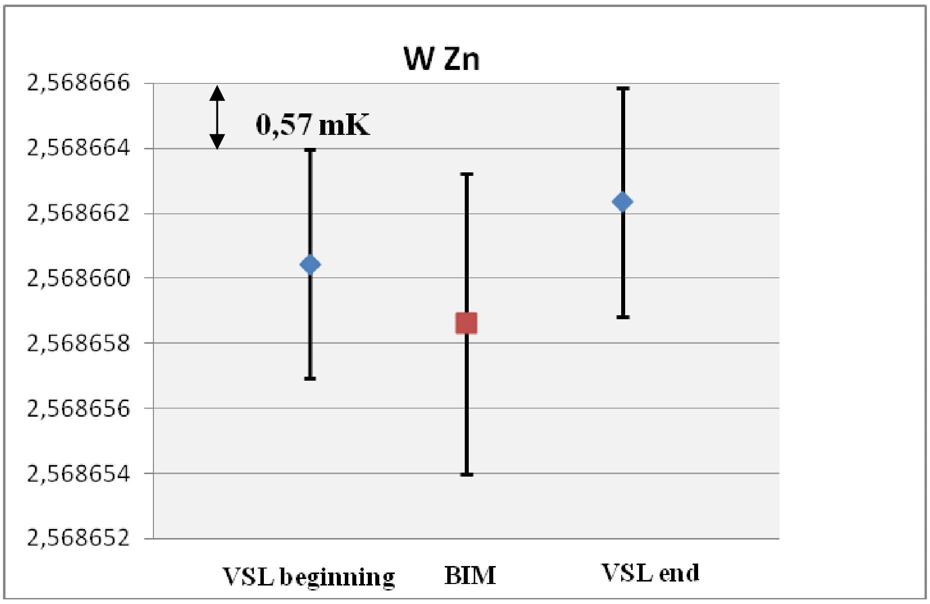

Fig 5. Results of SPRT calibration – Zn

The uncertainty due to the stability of the circulating SPRT during the period of the measurements is:

$$u_{StabSPRT} = \frac{\left|W_{VSLend} - W_{VSLbeginning}\right|}{\sqrt{3}} X \frac{\delta T}{\delta W} \qquad (1)$$

Table 2. Stability of the circulating SPRT

Fixed point	$W_{VSLbeginning}$	W_{VSLend}	$W_{VSLend} - W_{VSLbeginning}$	$W_{VSLend} - W_{VSLbeginning}$ expressed in mK	uncertainty $u_{StabSPRT}$
Hg	0,84416238	0,84416255	0,00000017	0,04	0,03
Ga	1,11812127	1,11812087	-0,00000040	-0,10	0,06
In	1,60970416	1,60970466	0,00000050	0,13	0,08
Sn	1,89265788	1,89265880	0,00000092	0,25	0,14
Zn	2,56866044	2,56866234	0,00000190	0,55	0,32

5.2. Degrees of equivalence

The degree of equivalence *D* is given with respect to the measurement results of VSL, which was taken as comparison reference value:

$$D = T_{BIM} - T_{VSL} \tag{2}$$

with the expanded uncertainty:

$$U(T_{BIM} - T_{VSL}) = 2\sqrt{u^2_{T_{BIM}} + u^2_{T_{VSL}} + u^2_{StabSPRT}} \tag{3}$$

Table 3. Degrees of equivalence

Fixed point	W_{VSL}	W_{BIM}	$W_{BIM} - W_{VSL}$	$T_{BIM} - T_{VSL}$, mK	$U(T_{BIM} - T_{VSL})$, mK
Hg	0,84416247	0,8441618	-0,00000067	-0,17	0,49
Ga	1,11812107	1,1181211	0,00000003	0,01	0,52
In	1,60970441	1,6097056	0,00000119	0,31	0,90
Sn	1,89265834	1,8926577	-0,00000064	-0,17	1,02
Zn	2,56866139	2,5686586	-0,00000279	-0,80	1,59

5.3. Link to the CCT-K3

The chronology of the VSL KCs:

- CCT-K3: 1997 to 2001
- CCT-K7: 2002 to 2004
- EUROMET.T-K3: 2001 to 2004
- EURAMET.T-K7: 2006 to 2007
- EUROMET.T-K3.1: 2008 to 2009

In 2006, after the CIPM 2005 clarification of the isotopic composition defining the TPW and consequent extensive internal investigation of newly manufactured TPW cells, VSL shifted its national reference for the TPW by +73 µK. In the subsequent comparisons (EURAMET.T-K7 and EURAMET.T-K3.1), VSL applied the new definition of the TPW national reference.
As consequence, the DoEs reported in CCT-K3, CCT-K7 and EUROMET.T-K3 reflect the "old" TPW definition while the DoEs reported in EURAMET.T-K7 and EURAMET.T-K3 reflects the "new" TPW definition.
Specifically, in the case of EURAMET.T-K3.1, cells VSL03T029, VSL06T003 and VSL06T004 were used; whose deviation from the new VSL national reference is -44 µK, -18 µK and -13 µK,

respectively. The average of the three cells used is just -25 µK from the new VSL national reference.
For the purpose of the comparison EURAMET.T-K3.1, VSL assumed that the use of the three cells together corresponded to a satisfactory realization of the new VSL national reference. In addition to this, for EURAMET.T-K3 comparison a very conservative uncertainty for the TPW measurements (U = 100 µK) was declared.
VSL should consider the 73 µK shift at the TPW and propagate it to higher temperatures when performing the link.
As clarified above, the TPW cells used in EURAMET.T-K3.1 realized a TPW temperature which is 25 µK lower than the new VSL definition. Consequently, the TPW realized during EURAMET.T-K3.1 was just (+73 – 25) µK = 48 µK higher than the TPW realized by VSL during EUROMET.T-K3, CCT-K7 and CCT-K3. Propagated to Zn, it makes 122 µK.

VSL used the following fixed points in the most recent KCs given in table 4.

Table 4.

Fixed point	**EURAMET.T-K3.1**	**EUROMET.T-K3**	**CCT-K9**
Hg	VSL89T010	VSL89T010	VSL89T009 and VSL89T010
H_2O	VSL03T029, VSL06T003 and VSL06T004	VSL03T028, VSL98T094, VSL95T375, VSL93T350, VSL92T053	VSL03T029, VSL06T003 and VSL06T004
Ga	VSL89T020 and VSL89T021	VSL89T020 and VSL89T021	VSL89T020 and VSL89T021
In	In1 VSL89T056 and In3 VSL04T233	In1 VSL89T056	In1 VSL89T056 and In3 VSL04T233
Sn	Sn3 VSL04T104	Sn1 VSL89T48	Sn1 VSL89T48 and Sn3 VSL04T104
Zn	Zn3 VSL04T073	Zn2 VSL89T099	Zn2 VSL89T099 and Zn3 VSL04T073

For Hg and Ga VSL used the same cells in both EUROMET.T-K3 and EURAMET.T-K3.1.
For In, VSL used In1 in EUROMET.T-K3 and In1 and In3 in EURAMET.T-K3.1. The temperature difference between In1 and In3, as measured recently in CCT-K9 comparison is: T(In1)-T(In3) = 0.26 mK. This means that the In realization of VSL in EURAMET.T-K3.1 is just 0.13 mK lower than VSL In realization in EUROMET.T-K3.
For Sn, VSL used Sn1 in EUROMET.T-K3 and Sn3 in EURAMET.T-K3.1. The temperature difference between Sn1 and Sn3, as measured recently in CCT-K9 comparison is: T(Sn1)-T(Sn3) = 0.42 mK.
For Zn, VSL used Zn2 in EUROMET.T-K3 and Zn3 in EURAMET.T-K3.1. The temperature difference between Zn2 and Zn3, as measured recently in CCT-K9 comparison is: T(Zn2)-T(Zn3) = 0.05 mK.

The link to the CCT-K3 participant LABi calculated through VSL participation in EUROMET.T-K3 is given as follow:

$$D_{BIM,LABi} = (T_{BIM} - T_{VSL})_{EURAMET-K3.1} + (T_{VSL} - T_{wm\,552})_{EURAMET-K3} + (T_{wm\,552} - T_{P\&CP})_{EURAMET-K3} + (T_{P\&CP} - T_{LABi})_{CCT-K3} \qquad (4)$$

$(T_{BIM} - T_{VSL})_{EURAMET-K3.1}$ comes from Table 3 of this Report
$(T_{VSL} - T_{wm552})_{EURAMET-K3}$ comes from Tables 16 to 20 of EURAMET.T-K3

$(T_{wm552} - T_{P\&CP})_{EURAMET-K3}$ comes from Tables 22 to 26 of EURAMET.T-K3

$(T_{P\&CP} - T_{LABi})_{CCT-K3}$ is calculated from the results of Tables 22, 24 to 27 of CCT-K3:

$$(T_{P\&CP} - T_{LABi})_{CCT-K3} = \frac{1}{n}\sum_{j=1}^{n}(T_{P\&CP_j} - T_{LABi}) \quad (5)$$

j- p and cp lab index
n- number of p and cp labs

The expanded uncertainty (U 95 %) of BIM and CCT-K3 participant NIST is:

$$U_{(T_{BIM} - T_{LABi})} = 2\sqrt{u^2_{(T_{BIM}-T_{VSL})} + u^2_{(T_{VSL}-T_{wm552})} + u^2_{reprod(P\&CP)mean} + u^2_{(T_{P\&CP}-T_{LABi})}} \quad (6)$$

$u_{(T_{BIM}-T_{VSL})}$ comes from Table 3 of this Report

$u_{(T_{VSL}-T_{wm552})}$ comes from Tables 16 to 20 of EURAMET.T-K3

$u_{reprod(P\&CP)mean}$ comes from Table 28 of EURAMET.T-K3

$u_{(T_{P\&CP}-T_{LABi})}$ is calculated from the results of Tables 22, 24 to 27 of CCT-K3:

$$u_{(T_{P\&CP}-T_{LABi})} = \frac{1}{n}\sqrt{\sum_{j=1}^{n}\left(\frac{U_{P\&CPj,\,LABi}}{2}\right)^2} \quad (7)$$

For each fixed point the degrees of equivalence and expanded uncertainties between BIM and the participants in CCT – K3 are given in Tables 5, 6 and 7.

Table 5. Link to CCT-K3 participants

	T_{BIM} - T_{NIST}		T_{BIM} - T_{NPL}		T_{BIM} - T_{PTB}		T_{BIM} - T_{BNM}		T_{BIM} - T_{IMGC}	
Point	*D* mK	*U* mK	*D* mK	*U* mK	*D* mK	*U* mK	*D* mK	*U* mK	*D* mK	*U* mK
Hg	-0,15	0,69	-0,35	0,72	-0,12	0,71	0,35	0,74	-0,07	0,71
Ga	-0,10	0,64	0,09	0,67	-0,29	0,65	-0,07	0,66	-0,20	0,65
In	-0,52	1,12	0,27	1,18	-0,19	1,24	-0,08	1,18	-0,35	1,18
Sn	-0,27	1,41	0,22	1,45	-0,58	1,47	0,11	1,48	-0,16	1,44
Zn	-2,02	2,28	-1,89	2,32	-1,08	2,35	-1,93	2,33	-0,57	2,32

Table 6. Link to CCT-K3 participants

	T_{BIM} - T_{KRISS}		T_{BIM} - T_{MSL}		T_{BIM} - T_{NIM}		T_{BIM} - T_{NML}		T_{BIM} - T_{NRC}	
Point	*D* mK	*U* mK	*D* mK	*U* mK	*D* mK	*U* mK	*D* mK	*U* mK	*D* mK	*U* mK
Hg	-0,66	0,75	-0,42	0,74	-0,36	0,76	-0,11	0,72	-0,37	0,71
Ga	-0,09	0,68	-0,27	0,67	0,50	0,70	0,07	0,66	0,04	0,66
In	-1,85	1,20	-0,12	1,22	1,09	1,24	1,13	1,21	0,51	1,15
Sn	0,26	1,51	0,57	1,53	0,91	2,91	0,87	1,45	1,08	1,47
Zn	-0,98	2,38	-1,27	3,35	-2,35	2,43	-1,44	2,34	-0,87	2,35

Table 7. Link to CCT-K3 participants

	T_{BIM} - T_{NRLM}		T_{BIM} - T_{SMU}		T_{BIM} - T_{VNIM}	
Point	D mK	U (95 %) mK	D mK	U (95 %) mK	D mK	U (95 %) mK
Hg	-0,05	0,77				
Ga	0,28	0,66	-0,18	0,66	-0,31	0,65
In	0,48	1,24			-1,03	1,19
Sn	0,93	1,49	-0,15	1,52	-0,38	1,47
Zn	0,41	2,65	-1,47	2,36	-0,26	2,48

5.4. Impact of comparisons on the calibration and measurement capabilities of a participating laboratory (CMCs)

The aim of the comparison is to demonstrate the calibration measurement capabilities (CMC's) of BIM in this working field. The results of this comparison are linked to CCT-K3 comparison. The reported uncertainties of BIM will be basis for CMC entries in appendix C of the CIPM Mutual Recognition Arrangement (MRA).

6. Summary and conclusions

The comparison EURAMET.T-K3.1 was organized with the main objective to show the international equivalence of BIM realisations of the international temperature scale ITS-90 at the fixed points of Hg, H_20, Ga, In, Sn and Zn. For all points of the measurements there is a good agreement between BIM and CCT-K3 comparison.

7. References

- International temperature scale ITS-90.
- Report to the CCT on Key Comparison EUROMET. T-K3, (EUROMET Project 552), Comparison of the realisations of the ITS-90from 83,805 8 K to 692,677 K (Final Report 4 September 2006)
- CCT-K3: Key Comparison of Realizations of the ITS-90 over the Range 83.8058 K to 933.473 K

Annex A. Uncertainty budgets

A1. Detailed uncertainty budget of BIM

Fixed point – Hg

Quantity Q_i	Components	Standard uncertainty $u_{(Qi)\ in\ m\Omega}$	Effective degrees of freedom n_i	Sensitivity coefficient dT/dR K/Ω	Uncertainty contribution u_i in mK
X_t	Repeatability of readings	0,0029	infinite	9,71	0,028
$C_{Xt/1}$	Uncertainty linked with purity	0,0109	infinite	9,71	0,106
$C_{Xt/2}$	Uncertainty linked Hydrostatic pressure correction	0,0081	infinite	9,71	0,079
$C_{Xt/3}$	Uncertainty linked with perturbing heat exchanges	0,0119	infinite	9,71	0,116
$C_{Xt/4}$	Uncertainty linked with self-heating correction	0,0014	infinite	9,71	0,014
$C_{Xt/5}$	Uncertainty linked with bridge linearity	0,0014	infinite	9,71	0,014
$C_{Xt/6}$	Uncertainty linked with AC/DC current	0,0055	infinite	9,71	0,053
$C_{Xt/7}$	Uncertainty linked with gas pressure	0,0053	infinite	9,71	0,051
$X_{0,01\ °C}$	Repeatability of readings				
	Repeatability of temperature realized by cell				
	Short–term repeatability of calibrated SPRT	0,0037	infinite	8,14	0,030
$C_{0,01°C/1}$	Uncertainty linked with purity and isotopic composition	0,0077	infinite	8,14	0,063
$C_{0,01°C/2}$	Uncertainty linked Hydrostatic pressure correction	0,0002	infinite	8,14	0,002
$C_{0,01°C/3}$	Uncertainty linked with perturbing heat exchanges	0,0020	infinite	8,14	0,016
$C_{0,01°C/4}$	Uncertainty linked with self-heating correction	0,0014	infinite	8,14	0,011
$C_{0,01°C/5}$	Uncertainty linked with bridge linearity	0,0015	infinite	8,14	0,012
$C_{0,01°C/6}$	Uncertainty linked with AC/DC current	0,0057	infinite	8,14	0,046
$C_{0,01°C/7}$	Uncertainty linked with internal insulation leakage	0,0031	infinite	8,14	0,025
$D_{RS/1}$	Uncertainty linked with stability of RS	0,0031	infinite	9,71	0,030
$D_{RS/2}$	Uncertainty linked with temperature of RS	0,0009	infinite	9,71	0,009
S_{Wt}	Wt scatter	0,0038	infinite	9,71	0,037
Combined uncertainty					0,22
Effective degrees of freedom					infinite
Expanded uncertainty					0,44

Fixed point – Ga

Quantity Q_i	Components	Standard uncertainty $u_{(Qi)\ in\ m\Omega}$	Effective degrees of freedom n_i	Sensitivity coefficient dT/dR K/Ω	Uncertainty contribution u_i in mK
X_t	Repeatability of readings	0,0003	infinite	9,90	0,003
$C_{Xt/1}$	Uncertainty linked with purity	0,0127	infinite	9,90	0,126
$C_{Xt/2}$	Uncertainty linked Hydrostatic pressure correction	0,0007	infinite	9,90	0,007
$C_{Xt/3}$	Uncertainty linked with perturbing heat exchanges	0,0025	infinite	9,90	0,025
$C_{Xt/4}$	Uncertainty linked with self-heating correction	0,0007	infinite	9,90	0,007
$C_{Xt/5}$	Uncertainty linked with bridge linearity	0,0014	infinite	9,90	0,014
$C_{Xt/6}$	Uncertainty linked with AC/DC current	0,0057	infinite	9,90	0,056
$C_{Xt/7}$	Uncertainty linked with gas pressure	0,0020	infinite	9,90	0,020
$X_{0,01\ °C}$	Repeatability of readings				
	Repeatability of temperature realized by cell				
	Short–term repeatability of calibrated SPRT	0,0007	infinite	11,09	0,008
$C_{0,01°C/1}$	Uncertainty linked with purity and isotopic composition	0,0077	infinite	11,09	0,085
$C_{0,01°C/2}$	Uncertainty linked Hydrostatic pressure correction	0,0002	infinite	11,09	0,002
$C_{0,01°C/3}$	Uncertainty linked with perturbing heat exchanges	0,0020	infinite	11,09	0,022
$C_{0,01°C/4}$	Uncertainty linked with self-heating correction	0,0014	infinite	11,09	0,016
$C_{0,01°C/5}$	Uncertainty linked with bridge linearity	0,0015	infinite	11,09	0,017
$C_{0,01°C/6}$	Uncertainty linked with AC/DC current	0,0057	infinite	11,09	0,063
$C_{0,01°C/7}$	Uncertainty linked with internal insulation leakage	0,0031	infinite	11,09	0,034
$D_{RS/1}$	Uncertainty linked with stability of RS	0,0031	infinite	9,90	0,031
$D_{RS/2}$	Uncertainty linked with temperature of RS	0,0009	infinite	9,90	0,009
S_{Wt}	Wt scatter	0,0038	infinite	9,90	0,038
Combined uncertainty					0,19
Effective degrees of freedom					infinite
Expanded uncertainty					0,38

Fixed point – In

Quantity Q_i	Components	Standard uncertainty $u_{(Qi)\ in\ m\Omega}$	Effective degrees of freedom n_i	Sensitivity coefficient dT/dR K/Ω	Uncertainty contribution u_i in mK
X_t	Repeatability of readings	0,0039	infinite	10,30	0,040
$C_{Xt/1}$	Uncertainty linked with purity	0,0318	infinite	10,30	0,328
$C_{Xt/2}$	Uncertainty linked Hydrostatic pressure correction	0,0019	infinite	10,30	0,020
$C_{Xt/3}$	Uncertainty linked with perturbing heat exchanges	0,0050	infinite	10,30	0,052
$C_{Xt/4}$	Uncertainty linked with self-heating correction	0,0007	infinite	10,30	0,007
$C_{Xt/5}$	Uncertainty linked with bridge linearity	0,0014	infinite	10,30	0,014
$C_{Xt/6}$	Uncertainty linked with AC/DC current	0,0055	infinite	10,30	0,057
$C_{Xt/7}$	Uncertainty linked with gas pressure	0,0048	infinite	10,30	0,049
$X_{0,01\ °C}$	Repeatability of readings				
	Repeatability of temperature realized by cell				
	Short–term repeatability of calibrated SPRT	0,0014	infinite	16,58	0,023
$C_{0,01°C/1}$	Uncertainty linked with purity and isotopic composition	0,0077	infinite	16,58	0,128
$C_{0,01°C/2}$	Uncertainty linked Hydrostatic pressure correction	0,0002	infinite	16,58	0,003
$C_{0,01°C/3}$	Uncertainty linked with perturbing heat exchanges	0,0020	infinite	16,58	0,033
$C_{0,01°C/4}$	Uncertainty linked with self-heating correction	0,0014	infinite	16,58	0,023
$C_{0,01°C/5}$	Uncertainty linked with bridge linearity	0,0014	infinite	16,58	0,023
$C_{0,01°C/6}$	Uncertainty linked with AC/DC current	0,0055	infinite	16,58	0,091
$C_{0,01°C/7}$	Uncertainty linked with internal insulation leakage	0,0029	infinite	16,58	0,048
$D_{RS/1}$	Uncertainty linked with stability of RS	0,0029	infinite	10,30	0,030
$D_{RS/2}$	Uncertainty linked with temperature of RS	0,0009	infinite	10,30	0,009
S_{Wt}	Wt scatter	0,0036	infinite	10,30	0,037
Combined uncertainty					0,39
Effective degrees of freedom					infinite
Expanded uncertainty					0,78

Fixed point – Sn

Quantity Q_i	Components	Standard uncertainty $u_{(Qi)\ in\ m\Omega}$	Effective degrees of freedom n_i	Sensitivity coefficient dT/dR K/Ω	Uncertainty contribution u_i in mK
X_t	Repeatability of readings	0,0108	infinite	10,54	0,114
$C_{Xt/1}$	Uncertainty linked with purity	0,0287	infinite	10,54	0,302
$C_{Xt/2}$	Uncertainty linked Hydrostatic pressure correction	0,0012	infinite	10,54	0,013
$C_{Xt/3}$	Uncertainty linked with perturbing heat exchanges	0,0050	infinite	10,54	0,053
$C_{Xt/4}$	Uncertainty linked with self-heating correction	0,0007	infinite	10,54	0,007
$C_{Xt/5}$	Uncertainty linked with bridge linearity	0,0015	infinite	10,54	0,016
$C_{Xt/6}$	Uncertainty linked with AC/DC current	0,0053	infinite	10,54	0,056
$C_{Xt/7}$	Uncertainty linked with gas pressure	0,0032	infinite	10,54	0,034
$X_{0,01\ °C}$	Repeatability of readings				
	Repeatability of temperature realized by cell				
	Short–term repeatability of calibrated SPRT	0,0014	infinite	19,91	0,028
$C_{0,01°C/1}$	Uncertainty linked with purity and isotopic composition	0,0077	infinite	19,91	0,153
$C_{0,01°C/2}$	Uncertainty linked Hydrostatic pressure correction	0,0002	infinite	19,91	0,004
$C_{0,01°C/3}$	Uncertainty linked with perturbing heat exchanges	0,0020	infinite	19,91	0,040
$C_{0,01°C/4}$	Uncertainty linked with self-heating correction	0,0014	infinite	19,91	0,028
$C_{0,01°C/5}$	Uncertainty linked with bridge linearity	0,0015	infinite	19,91	0,030
$C_{0,01°C/6}$	Uncertainty linked with AC/DC current	0,0057	infinite	19,91	0,114
$C_{0,01°C/7}$	Uncertainty linked with internal insulation leakage	0,0031	infinite	19,91	0,062
$D_{RS/1}$	Uncertainty linked with stability of RS	0,0031	infinite	10,54	0,033
$D_{RS/2}$	Uncertainty linked with temperature of RS	0,0009	infinite	10,54	0,009
S_{Wt}	Wt scatter	0,0038	infinite	10,54	0,040
Combined uncertainty					0,40
Effective degrees of freedom					infinite
Expanded uncertainty					0,80

Fixed point – Zn

Quantity Q_i	Components	Standard uncertainty $u_{(Qi)\ in\ m\Omega}$	Effective degrees of freedom n_i	Sensitivity coefficient dT/dR K/Ω	Uncertainty contribution u_i in mK
X_t	Repeatability of readings	0,0149	infinite	11,21	0,167
$C_{Xt/1}$	Uncertainty linked with purity	0,0404	infinite	11,21	0,453
$C_{Xt/2}$	Uncertainty linked Hydrostatic pressure correction	0,0014	infinite	11,21	0,016
$C_{Xt/3}$	Uncertainty linked with perturbing heat exchanges	0,0050	infinite	11,21	0,056
$C_{Xt/4}$	Uncertainty linked with self-heating correction	0,0007	infinite	11,21	0,008
$C_{Xt/5}$	Uncertainty linked with bridge linearity	0,0014	infinite	11,21	0,016
$C_{Xt/6}$	Uncertainty linked with AC/DC current	0,0050	infinite	11,21	0,056
$C_{Xt/7}$	Uncertainty linked with gas pressure	0,0039	infinite	11,21	0,044
$X_{0,01\ °C}$	Repeatability of readings				
	Repeatability of temperature realized by cell				
	Short–term repeatability of calibrated SPRT	0,0007	infinite	28,75	0,020
$C_{0,01°C/1}$	Uncertainty linked with purity and isotopic composition	0,0077	infinite	28,75	0,221
$C_{0,01°C/2}$	Uncertainty linked Hydrostatic pressure correction	0,0002	infinite	28,75	0,006
$C_{0,01°C/3}$	Uncertainty linked with perturbing heat exchanges	0,0020	infinite	28,75	0,058
$C_{0,01°C/4}$	Uncertainty linked with self-heating correction	0,0014	infinite	28,75	0,040
$C_{0,01°C/5}$	Uncertainty linked with bridge linearity	0,0015	infinite	28,75	0,043
$C_{0,01°C/6}$	Uncertainty linked with AC/DC current	0,0057	infinite	28,75	0,164
$C_{0,01°C/7}$	Uncertainty linked with internal insulation leakage	0,0031	infinite	28,75	0,089
$D_{RS/1}$	Uncertainty linked with stability of RS	0,0031	infinite	11,21	0,035
$D_{RS/2}$	Uncertainty linked with temperature of RS	0,0009	infinite	11,21	0,010
S_{Wt}	Wt scatter	0,0038	infinite	11,21	0,043
Combined uncertainty					0,58
Effective degrees of freedom					infinite
Expanded uncertainty					1,16

A2. Detailed uncertainty budget of VSL

Fixed point – Hg

Quantity Q_i	Components	Standard uncertainty $u_{(Qi)}$ in mK	Effective degrees of freedom ν_i	Sensitivity coefficient	Uncertainty contribution u_i in mK
X_t	Repeatability of readings (in this case the value for the plateau progress has been used)	0,058	inf	1	0,058
$C_{Xt/1}$	Uncertainty linked with purity	0,012	inf	1	0,012
$C_{Xt/2}$	Uncertainty linked Hydrostatic pressure correction	0,020	inf	1	0,020
$C_{Xt/3}$	Uncertainty linked with perturbing heat exchanges	0,035	inf	1	0,035
$C_{Xt/4}$	Uncertainty linked with self-heating correction	0,002	inf	1	0,002
$C_{Xt/5}$	Uncertainty linked with bridge linearity	0,029	inf	1	0,029
$C_{Xt/6}$	Uncertainty linked with AC/DC current (all measurements were performed with a DC bridge)	0,000	inf	1	0,000
$C_{Xt/7}$	Uncertainty linked with gas pressure	0,004	inf	1	0,004
$X_{0,01\ °C}$	Repeatability of readings Repeatability of temperature realized by cell Short–term repeatability of calibrated SPRT	0,030	20	0,834	0,025
$C_{0,01°C/1}$	Uncertainty linked with purity and isotopic composition	0,035	inf	0,834	0,029
$C_{0,01°C/2}$	Uncertainty linked Hydrostatic pressure correction	0,002	inf	0,834	0,002
$C_{0,01°C/3}$	Uncertainty linked with perturbing heat exchanges	0,010	inf	0,834	0,008
$C_{0,01°C/4}$	Uncertainty linked with self-heating correction	0,002	inf	0,834	0,002
$C_{0,01°C/5}$	Uncertainty linked with bridge linearity	0,029	inf	0,834	0,024
$C_{0,01°C/6}$	Uncertainty linked with AC/DC current (included in C Xt/6)				
$C_{0,01°C/7}$	Uncertainty linked with internal insulation leakage				
$D_{RS/1}$	Uncertainty linked with stability of RS				
$D_{RS/2}$	Uncertainty linked with temperature of RS	0,021	inf	1	0,021
S_{Wt}	Wt scatter	0,035	inf	1	0,035
Combined uncertainty					0,10
Effective degrees of freedom					infinite
Expanded uncertainty					0,20

Fixed point – Ga

Quantity Q_i	Components	Standard uncertainty $u_{(Qi)}$ in mK	Effective degrees of freedom ν_i	Sensitivity coefficient	Uncertainty contribution u_i in mK
X_t	Repeatability of readings (in this case the value for the plateau progress has been used)	0,058	inf	1	0,058
$C_{Xt/1}$	Uncertainty linked with purity	0,079	inf	1	0,079
$C_{Xt/2}$	Uncertainty linked Hydrostatic pressure correction	0,003	inf	1	0,003
$C_{Xt/3}$	Uncertainty linked with perturbing heat exchanges	0,017	inf	1	0,017
$C_{Xt/4}$	Uncertainty linked with self-heating correction	0,002	inf	1	0,002
$C_{Xt/5}$	Uncertainty linked with bridge linearity	0,029	inf	1	0,029
$C_{Xt/6}$	Uncertainty linked with AC/DC current (all measurements were performed with a DC bridge)	0,000	inf	1	0,000
$C_{Xt/7}$	Uncertainty linked with gas pressure	0,087	inf	1	0,087
$X_{0,01\ °C}$	Repeatability of readings				
	Repeatability of temperature realized by cell				
	Short–term repeatability of calibrated SPRT	0,030	20	1,128	0,034
$C_{0,01°C/1}$	Uncertainty linked with purity and isotopic composition	0,035	inf	1,128	0,039
$C_{0,01°C/2}$	Uncertainty linked Hydrostatic pressure correction	0,002	inf	1,128	0,002
$C_{0,01°C/3}$	Uncertainty linked with perturbing heat exchanges	0,010	inf	1,128	0,011
$C_{0,01°C/4}$	Uncertainty linked with self-heating correction	0,002	inf	1,128	0,002
$C_{0,01°C/5}$	Uncertainty linked with bridge linearity	0,029	inf	1,128	0,033
$C_{0,01°C/6}$	Uncertainty linked with AC/DC current (included in C Xt/6)				
$C_{0,01°C/7}$	Uncertainty linked with internal insulation leakage				
$D_{RS/1}$	Uncertainty linked with stability of RS				
$D_{RS/2}$	Uncertainty linked with temperature of RS	0,028	inf	1	0,028
S_{Wt}	Wt scatter	0,060	inf	1	0,060
Combined uncertainty					0,16
Effective degrees of freedom					infinite
Expanded uncertainty					0,33

Fixed point – In

Quantity Q_i	Components	Standard uncertainty $u_{(Qi)}$ in mK	Effective degrees of freedom ν_i	Sensitivity coefficient	Uncertainty contribution u_i in mK
X_t	Repeatability of readings (in this case the value for the plateau progress has been used)	0,115	inf	1	0,115
$C_{Xt/1}$	Uncertainty linked with purity	0,027	inf		0,027
$C_{Xt/2}$	Uncertainty linked Hydrostatic pressure correction	0,019	inf	1	0,019
$C_{Xt/3}$	Uncertainty linked with perturbing heat exchanges	0,017	inf	1	0,017
$C_{Xt/4}$	Uncertainty linked with self-heating correction	0,002	inf	1	0,002
$C_{Xt/5}$	Uncertainty linked with bridge linearity	0,115	inf	1	0,115
$C_{Xt/6}$	Uncertainty linked with AC/DC current (all measurements were performed with a DC bridge)	0,000	inf	1	0,000
$C_{Xt/7}$	Uncertainty linked with gas pressure	0,002	inf	1	0,002
$X_{0,01\,°C}$	Repeatability of readings Repeatability of temperature realized by cell Short–term repeatability of calibrated SPRT	0,030	20	1,689	0,051
$C_{0,01°C/1}$	Uncertainty linked with purity and isotopic composition	0,035	inf	1,689	0,059
$C_{0,01°C/2}$	Uncertainty linked Hydrostatic pressure correction	0,002	inf	1,689	0,003
$C_{0,01°C/3}$	Uncertainty linked with perturbing heat exchanges	0,010	inf	1,689	0,017
$C_{0,01°C/4}$	Uncertainty linked with self-heating correction	0,002	inf	1,689	0,003
$C_{0,01°C/5}$	Uncertainty linked with bridge linearity	0,029	inf	1,689	0,049
$C_{0,01°C/6}$	Uncertainty linked with AC/DC current (included in C Xt/6)				
$C_{0,01°C/7}$	Uncertainty linked with internal insulation leakage				
$D_{RS/1}$	Uncertainty linked with stability of RS				
$D_{RS/2}$	Uncertainty linked with temperature of RS	0,040	inf	1	0,040
S_{Wt}	Wt scatter	0,070	inf	1	0,070
Combined uncertainty					0,21
Effective degrees of freedom					infinite
Expanded uncertainty					0,42

Fixed point – Sn

Quantity Q_i	Components	Standard uncertainty $u_{(Qi)}$ in mK	Effective degrees of freedom ν_i	Sensitivity coefficient	Uncertainty contribution u_i in mK
X_t	Repeatability of readings (in this case the value for the plateau progress has been used)	0,115	inf	1	0,115
$C_{Xt/1}$	Uncertainty linked with purity	0,175	inf		0,175
$C_{Xt/2}$	Uncertainty linked Hydrostatic pressure correction	0,013	inf	1	0,013
$C_{Xt/3}$	Uncertainty linked with perturbing heat exchanges	0,017	inf	1	0,017
$C_{Xt/4}$	Uncertainty linked with self-heating correction	0,004	inf	1	0,004
$C_{Xt/5}$	Uncertainty linked with bridge linearity	0,115	inf	1	0,115
$C_{Xt/6}$	Uncertainty linked with AC/DC current (all measurements were performed with a DC bridge)	0,000	inf	1	0,000
$C_{Xt/7}$	Uncertainty linked with gas pressure	0,001	inf	1	0,001
$X_{0,01\,°C}$	Repeatability of readings Repeatability of temperature realized by cell Short–term repeatability of calibrated SPRT	0,030	20	2,033	0,061
$C_{0,01°C/1}$	Uncertainty linked with purity and isotopic composition	0,035	inf	2,033	0,071
$C_{0,01°C/2}$	Uncertainty linked Hydrostatic pressure correction	0,002	inf	2,033	0,004
$C_{0,01°C/3}$	Uncertainty linked with perturbing heat exchanges	0,010	inf	2,033	0,020
$C_{0,01°C/4}$	Uncertainty linked with self-heating correction	0,002	inf	2,033	0,004
$C_{0,01°C/5}$	Uncertainty linked with bridge linearity	0,029	inf	2,033	0,059
$C_{0,01°C/6}$	Uncertainty linked with AC/DC current (included in C Xt/6)				
$C_{0,01°C/7}$	Uncertainty linked with internal insulation leakage				
$D_{RS/1}$	Uncertainty linked with stability of RS				
$D_{RS/2}$	Uncertainty linked with temperature of RS	0,047	inf	1	0,047
S_{Wt}	Wt scatter	0,069	inf	1	0,069
Combined uncertainty					0,28
Effective degrees of freedom					infinite
Expanded uncertainty					0,56

Fixed point – Zn

Quantity Q_i	Components	Standard uncertainty $u_{(Qi)}$ in mK	Effective degrees of freedom ν_i	Sensitivity coefficient	Uncertainty contribution u_i in mK
X_t	Repeatability of readings (in this case the value for the plateau progress has been used)	0,115	inf	1	0,115
$C_{Xt/1}$	Uncertainty linked with purity	0,312	inf		0,312
$C_{Xt/2}$	Uncertainty linked Hydrostatic pressure correction	0,016	inf	1	0,016
$C_{Xt/3}$	Uncertainty linked with perturbing heat exchanges	0,017	inf	1	0,017
$C_{Xt/4}$	Uncertainty linked with self-heating correction	0,004	inf	1	0,004
$C_{Xt/5}$	Uncertainty linked with bridge linearity	0,115	inf	1	0,115
$C_{Xt/6}$	Uncertainty linked with AC/DC current (all measurements were performed with a DC bridge)	0,000	inf	1	0,000
$C_{Xt/7}$	Uncertainty linked with gas pressure	0,001	inf	1	0,001
$X_{0,01\ °C}$	Repeatability of readings Repeatability of temperature realized by cell Short–term repeatability of calibrated SPRT	0,030	20	2,931	0,088
$C_{0,01°C/1}$	Uncertainty linked with purity and isotopic composition	0,035	inf	2,931	0,103
$C_{0,01°C/2}$	Uncertainty linked Hydrostatic pressure correction	0,002	inf	2,931	0,006
$C_{0,01°C/3}$	Uncertainty linked with perturbing heat exchanges	0,010	inf	2,931	0,029
$C_{0,01°C/4}$	Uncertainty linked with self-heating correction	0,002	inf	2,931	0,006
$C_{0,01°C/5}$	Uncertainty linked with bridge linearity	0,029	inf	2,931	0,085
$C_{0,01°C/6}$	Uncertainty linked with AC/DC current (included in $C_{Xt/6}$)				
$C_{0,01°C/7}$	Uncertainty linked with internal insulation leakage				
$D_{RS/1}$	Uncertainty linked with stability of RS				
$D_{RS/2}$	Uncertainty linked with temperature of RS	0,064	inf	1	0,064
S_{Wt}	Wt scatter	0,198	inf	1	0,198
Combined uncertainty					0,44
Effective degrees of freedom					infinite
Expanded uncertainty					0,88

Annex B. Description of instrumentation

B1. Description of instrumentation of BIM

Bridge	
Manufacturer	Automatic Systems Laboratories Ltd. England
Type	F18 Precision Thermometry Bridge
Unity reading	0,0000001
AC or DC	AC
If AC. give Frequency	75 Hz
If DC. give Period of reversal	
Normal measurement current	1 mA
Self-heating current	1,41 mA
Evaluation of linearity of resistance	
bridge (yes or not)	not
If yes. How?	

Reference resistor	
Manufacturer	H.Tinsley&Company Ltd. England
Type	5658A
Reference resistor temperature control (yes or not)	yes
If yes. How?	Standard Resistor Maintenance Bath. Isotech

TPW Cell	
Home made or not	not
Immersion depth of middle of the SPRT sensitive element/cm	23
How are mantles maintained (ice. bath.….)	Isotech WTP Maintenance Bath. Model ITL M 18233

Zn Cell	
Home made or not	not
Closed cell or open	closed
Nominal purity	99,9999%
Immersion depth of middle of the SPRT sensitive element/cm	17

Zn Furnace	
Home or not	not
Type (1 zone. 3 zones. heat pipe. …….)	Isothech Medium temperature 3 zones Furnace. Model ITL M 17703
Typical duration of the freezing plateau	8 h

Sn Cell	
Home made or not	not
Closed cell or open	closed
Nominal purity	99,9999%
Immersion depth of middle of the SPRT sensitive element/cm	17

Sn Furnace	
Home or not	not
Type (1 zone. 3 zones. heat pipe. …….)	Isothech Medium temperature 3 zones Furnace. Model ITL M 17703
Typical duration of the freezing plateau	12 h

In Cell	
Home made or not	not
Closed cell or open	closed
Nominal purity	99,9999%
Immersion depth of middle of the SPRT sensitive element/cm	17

In Furnace	
Home or not	not
Type (1 zone. 3 zones. heat pipe. …….)	Isothech Medium temperature 3 zones Furnace. Model ITL M 17703
Typical duration of the freezing plateau	9 h

Ga Cell	
Home made or not	not
Closed cell or open	closed
Nominal purity	99,9999992%
Immersion depth of middle of the SPRT sensitive element/cm	21

Ga Furnace	
Home or not	not
Type (1 zone. 3 zones. heat pipe. …….)	Isothech Galium Melter. Model 17402B
Typical duration of the melting plateau	13 h

Hg Cell	
Home made or not	not
Closed cell or open	closed
Nominal purity	99,99999%
Immersion depth of middle of the SPRT sensitive element/cm	16,5

Hg cryostat	
Home or not	not
Type (cryostat. bath. …….)	cryostat/ Isotech Apparatus Model 17725
Typical duration of the melting plateau	8 h

B2. Description of instrumentation of VSL

Laboratory name	**NMi-VSL**
Bridge	
Manufacturer	Measurements International Inc
Type	6015T & 6010T
Unity reading	
Manufacturer	Automatic Systems Laboratories
Type	F18
AC or DC	DC and AC
If AC. give Frequency	25 Hz
If DC. give Period of reversal	4 secondens
Normal measurement current	2 mA
Self-heating current	2,82 mA
Evaluation of linearity of resistance	
bridge (yes or not)	yes
If yes. How?	RBC calibrator
Reference resistor	
Manufacturer	Tinsley
Type	5684
Reference resistor temperature control (yes or not)	Yes
If yes. How?	Temperature controlled oilbath
TPW Cell	
Home made or not	Home made
Immersion depth of middle of the SPRT sensitive element/cm	21,6 cm/20,6 cm dependent on cell
How are mantles maintained (ice. bath…..)	stirred bath
Zn Cell	
Home made or not	Home made
Closed cell or open	open
Nominal purity	6N
Immersion depth of middle of the SPRT	14,1

sensitive element/cm	
Zn Furnace	
Home or not	Home made
Type (1 zone. 3 zones. heat pipe. …….)	3 zone
Typical duration of the melting plateau	
Typical duration of the freezing plateau	> 10 hours
Sn Cell	
Home made or not	Home made
Closed cell or open	open
Nominal purity	6N
Immersion depth of middle of the SPRT sensitive element/cm	16,1
Sn Furnace	
Home or not	Home made
Type (1 zone. 3 zones. heat pipe. …….)	3 zones
Typical duration of the melting plateau	
Typical duration of the freezing plateau	> 10 hours
In Cell	
Home made or not	Home made
Closed cell or open	open
Nominal purity	7N
Immersion depth of middle of the SPRT sensitive element/cm	14.6
In Furnace	
Home or not	Home made
Type (1 zone. 3 zones. heat pipe. …….)	3 zones
Typical duration of the melting plateau	
Typical duration of the freezing plateau	> 10 hours
Ga Cell	
Home made or not	Commercial
Closed cell or open	closed
Nominal purity	6N
Immersion depth of middle of the SPRT sensitive element/cm	24.1
Ga Furnace	
Home or not	Commercial
Type (1 zone. 3 zones. heat pipe. …….)	1 zone
Typical duration of the melting plateau	6 hours
Typical duration of the freezing plateau	

Hg Cell	
Home made or not	Home made
Closed cell or open	closed
Nominal purity	7N

Immersion depth of middle of the SPRT sensitive element/cm	13,8
Hg Cryostat	
Home made or not	Isotech
Type (cryostat. bath….)	818
Typical duration of melting plateau	7,5 Hours
Typical duration of freezing plateau	not used

Annex C. Immersion Curves

C1. Immersion curves of BIM

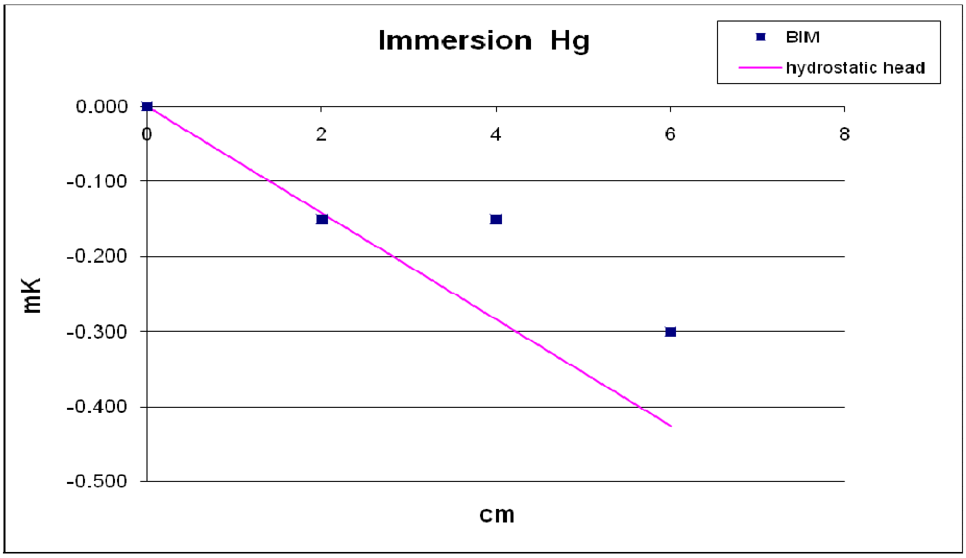

Figure I. Immersion curves for Mercury.

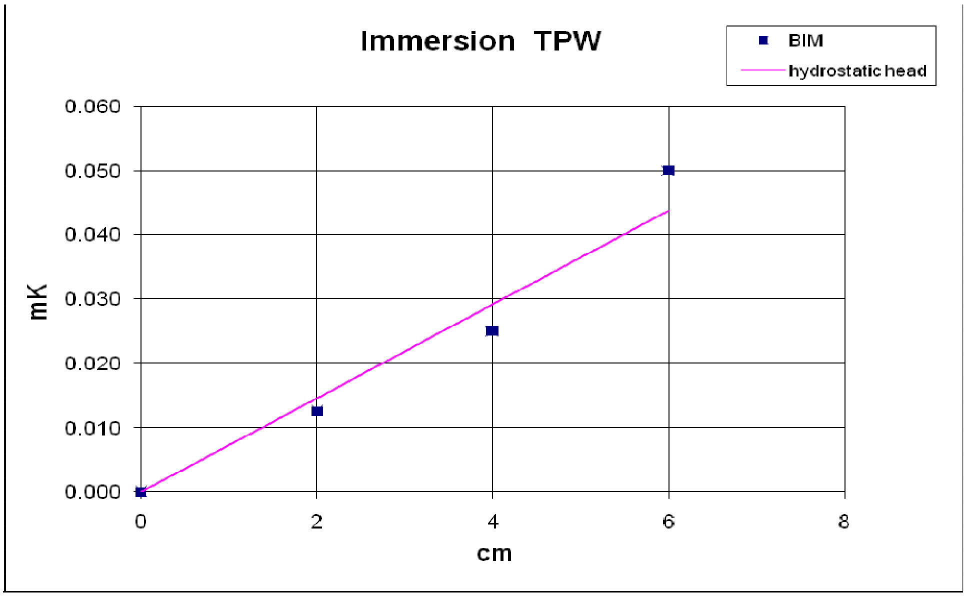

Figure II. Immersion curves for TPW.

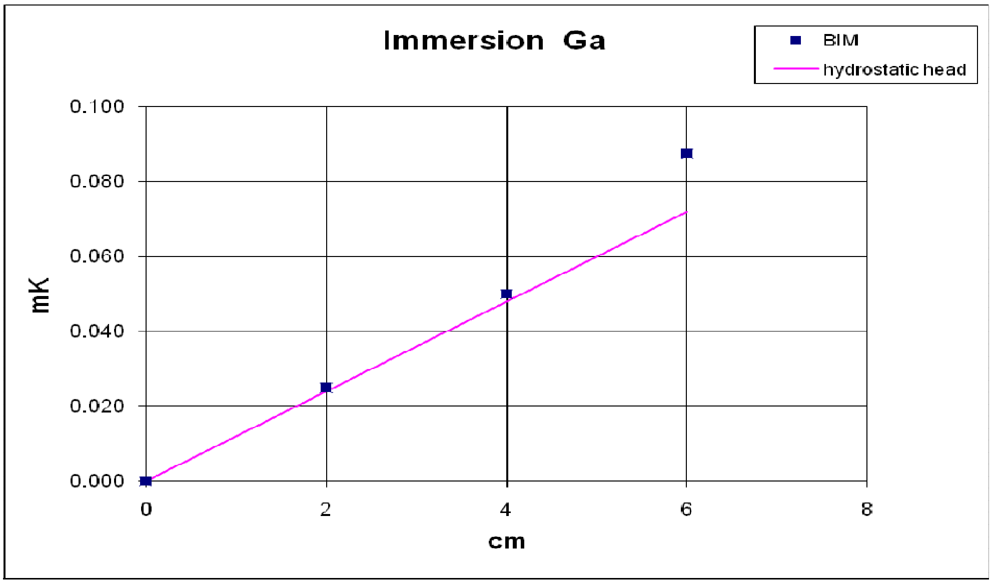

Figure III. Immersion curves for Gallium.

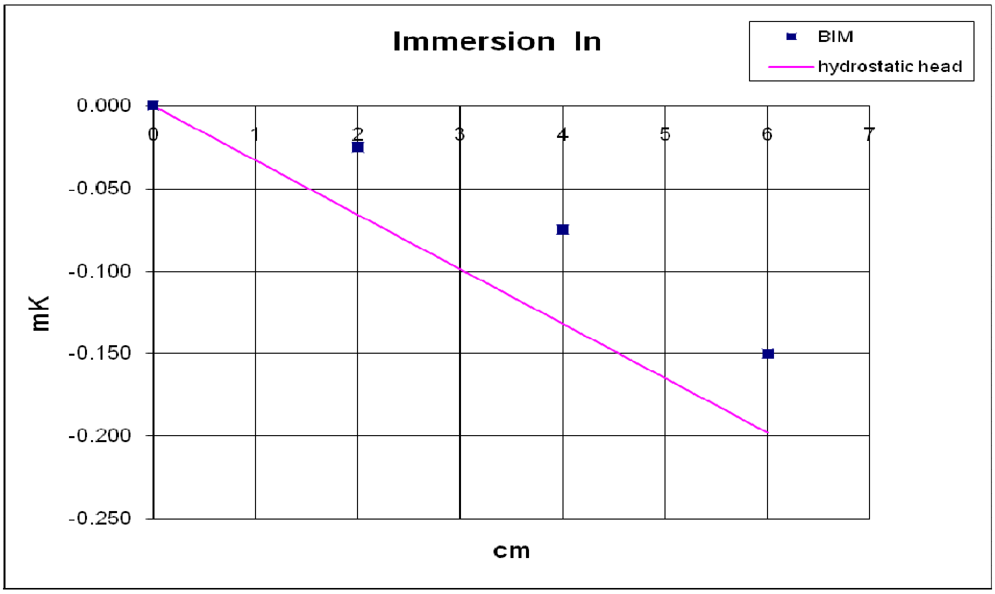

Figure IV. Immersion curves for Indium.

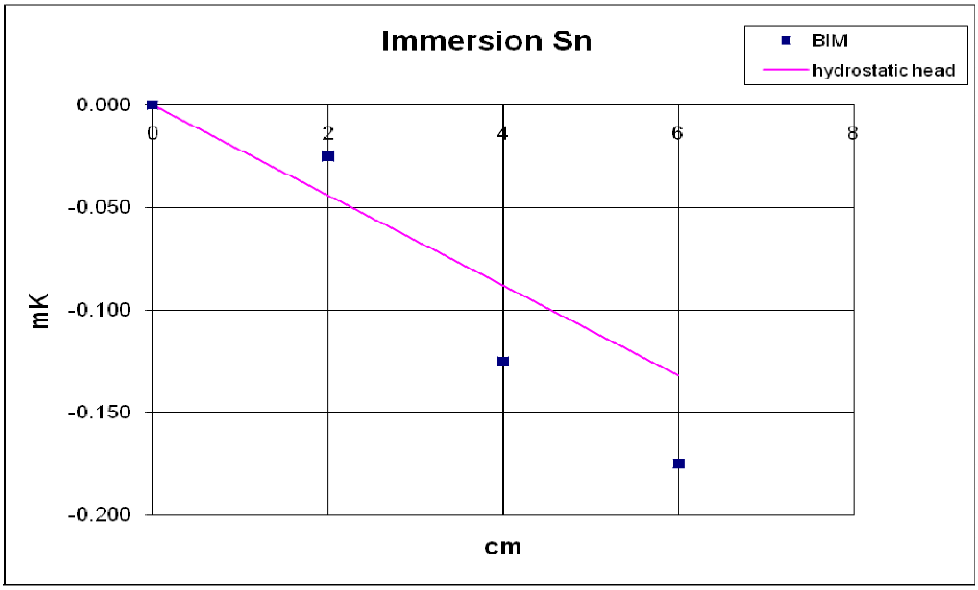

Figure V. Immersion curves for Tin.

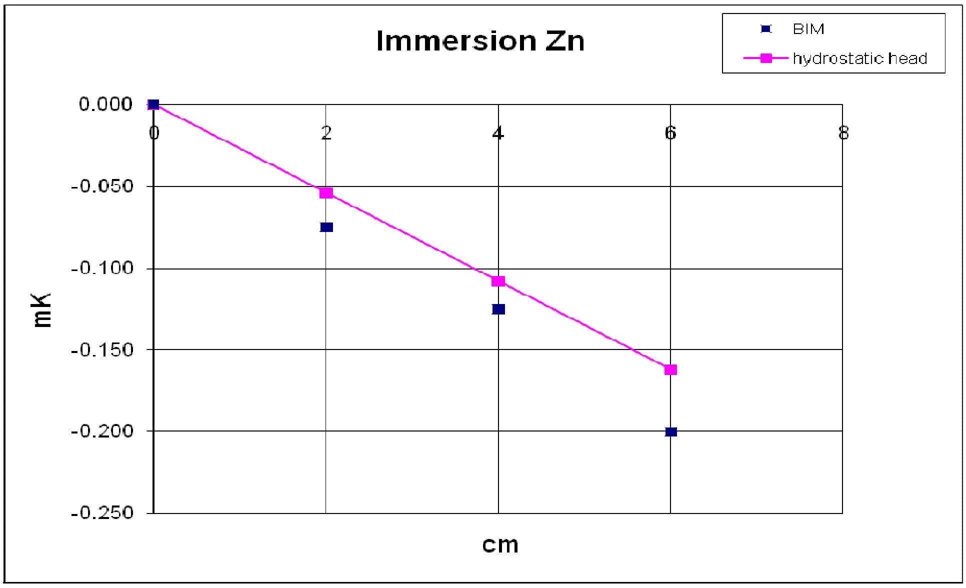

Figure VI. Immersion curves for Zinc.

Annex D. Plateaus curves

D1. Plateaus curves of BIM

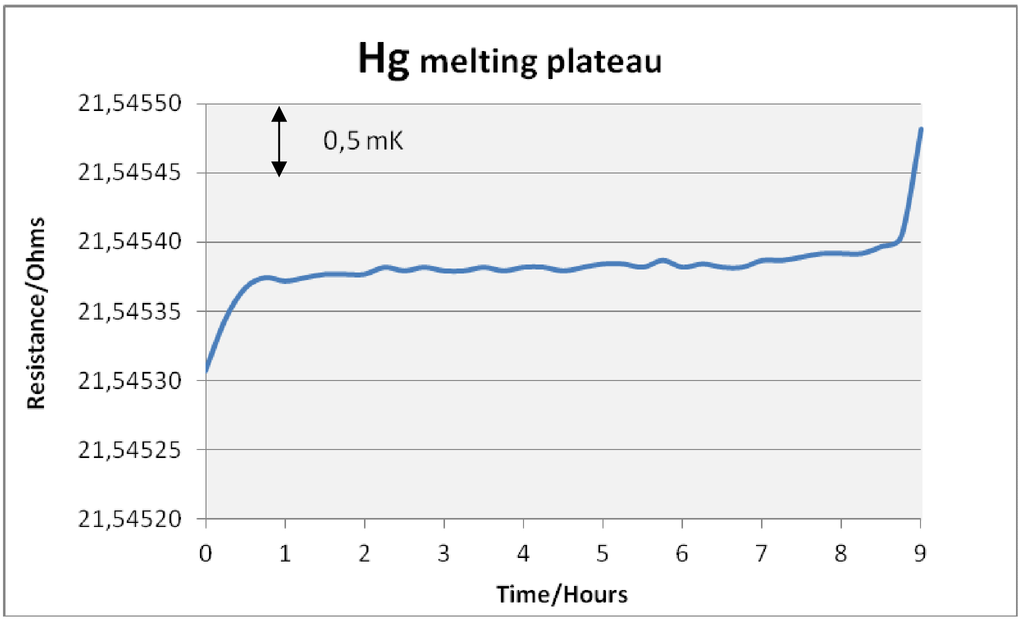

Figure I. Melting curve of Mercury.

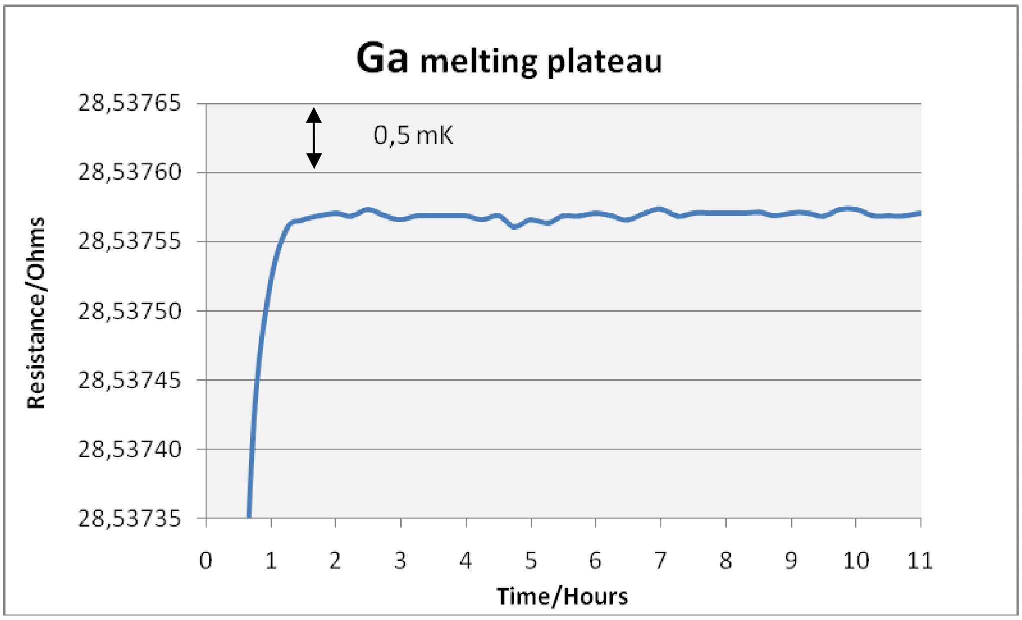

Figure II. Melting curve of Gallium.

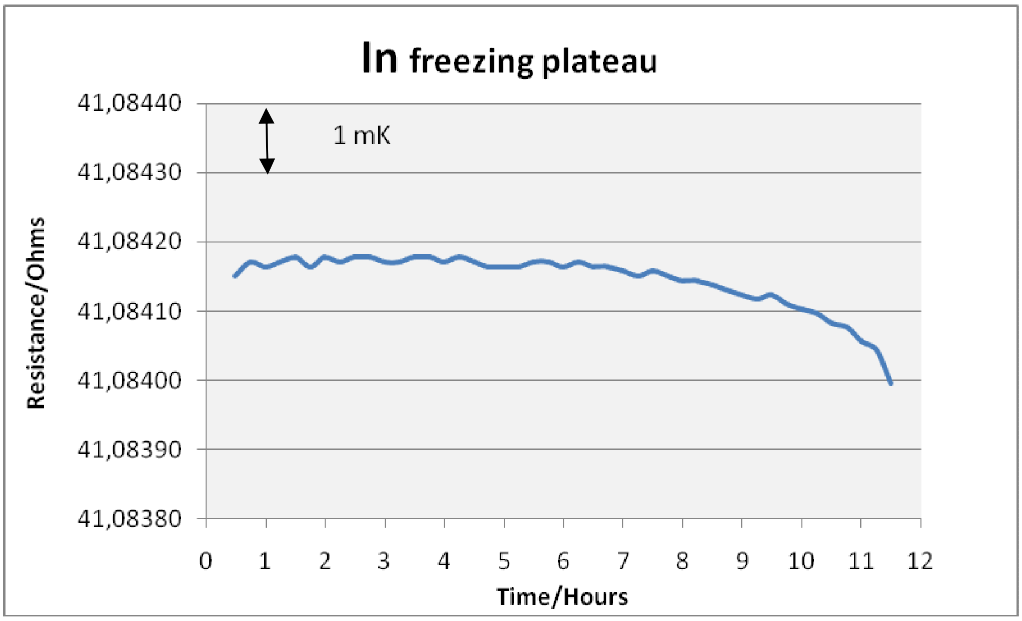

Figure III. Freezing curve of Indium.

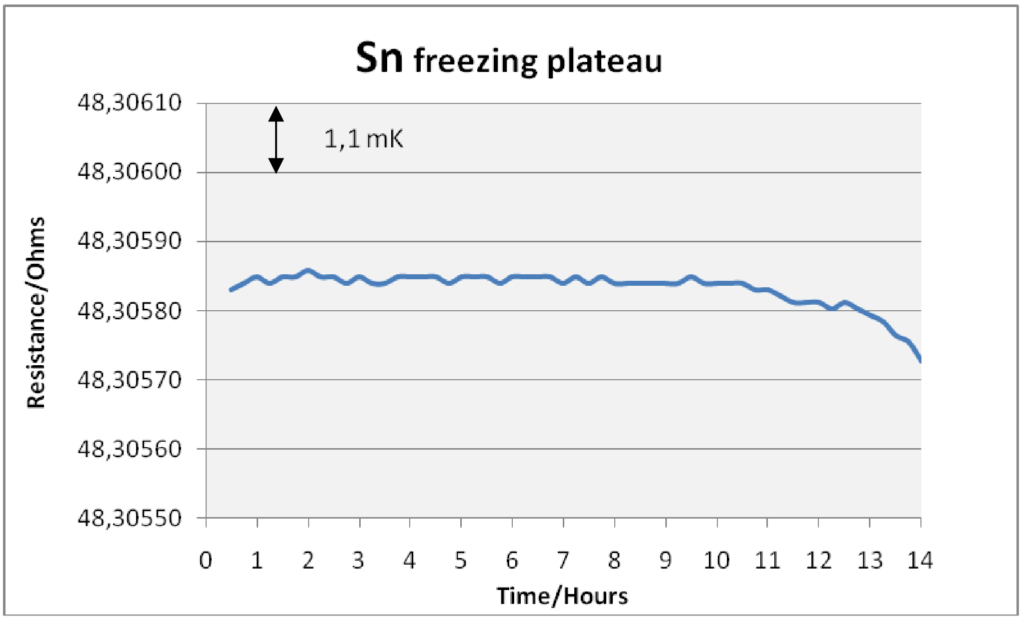

Figure IV. Freezing curve of Tin.

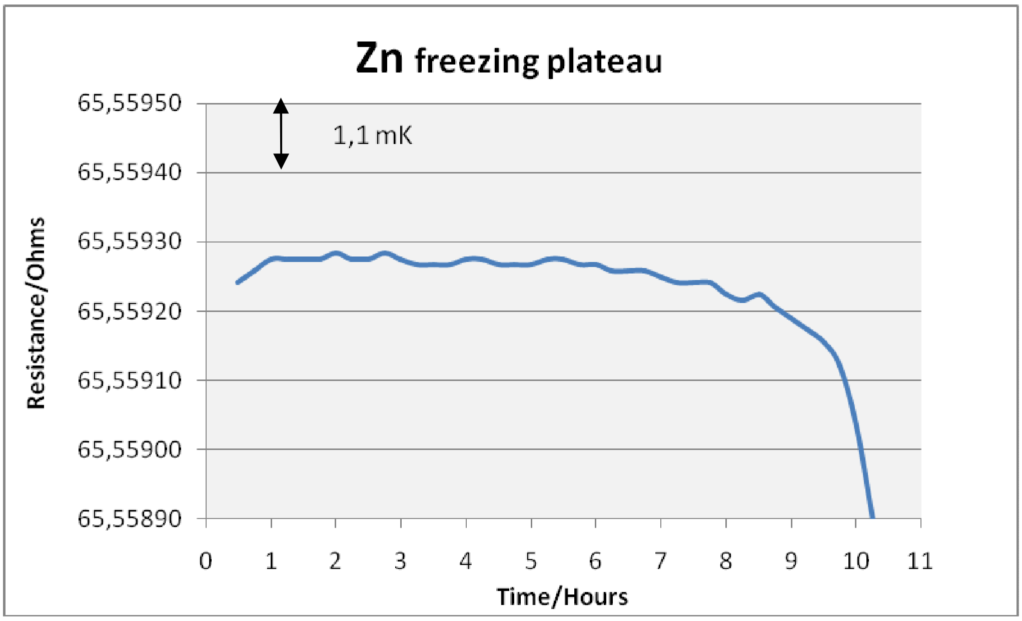

Figure V. Freezing curve of Zinc.

Annex E. Comparison protocol

1. Introduction

In the framework of Phare project BG 2005/017-353.02.02. LOT 1 was decided that a key comparison of temperature measurements shall be carried out between the temperature laboratory of BIM (Bulgaria) acting as organizing laboratory and NMI-VSL acting as reference and linking laboratory to the corresponding CCT-K3 comparison.

This bilateral comparison is intended to compare BIM and NMi VSL realization of the ITS-90. The range of temperature covered in this comparison is from the triple point of Hg (234.3156 K) to the freezing point of Zn (692.677 K) using long-stem SPRT.

The participants of this comparison should follow the instructions which are given below. Moreover each laboratory should follow its normal practice when realizing the ITS-90.

This protocol follows closely the technical protocol of the corresponding CCT-K3 comparison.

The laboratory will calibrate 1 SPRT. The SPRT used for this comparison will be selected for its very good stability.
The transfer standard will be a long-stem standard platinum resistance thermometer provided by NMi VSL:
Manufacturer: Leeds & Northrup
Type: 8167-25°C
Serial number: 1773051

The thermometers are very fragile so it must be handled with extreme care. When not in use it should be stored in a safe place. The SPRT will be hand carried from laboratory to laboratory. Each lab is responsible for carriage of the thermometer to the other lab.

If thermometer has not been received in due time the pilot must be immediately informed in order the timetable be revised accordingly.

The participants will calibrate one thermometer. They will establish a calibration report.

This report will be sent (within 1 month) simultaneously to the pilot. From the delivered data the pilot will establish the difference between participating laboratories. The pilot will establish as well the uncertainty associated with the calculated difference.

3. Participating Laboratories

NMi van Swinden Laboratorium
Contact person: Rien Bosma
Tel: +31 15 269 1663
E-mail: rbosma@nmi.nl
Address: Thijsseweg 11
2629 JA DELT
The Netherlands

BIM
Contact person: Sasho Nedialkov
Tel: +395 2 8762946
Fax: +395 2 9702 735
E-mail: s.nedialkov@bim.government.bg
Address: 52 B blvd. G. M. Dimitrov.
Sofia. Bulgaria

Co-ordinator in the Phare project:
Erik Dierikx
Tel. +31 15 269 1688
E-mail: edierikx@nmi.nl

4. Provisional schedule

Table I

Period	Task	Lab.
October 2008	Selection and calibration of SPRT	NMi VSL
November 2008	Calibration of the SPRT	BIM
December 2008	Calibration of the SPRT	NMi VSL

5. Procedures

The participants of this comparison should follow the instructions which are given below. Moreover each laboratory should follow its normal practice when realizing the ITS-90. The instructions are based on Appendix 1 of Report to the CCT on Key Comparison 3 (B. W. Mangum et al. of NIST. November 1999). The comparison strictly follows the protocols given in the Guidelines for CIPM key comparisons Appendix F to the MRA, 1 March 1999.

The participating NMLs are required to perform calibration of SPRT at ITS-90 fixed points as presented in their own CMCs. it means that restricted ranges are allowed.

The goal of the comparison is to compare the national highest accuracy realization of ITS-90 as the participating laboratories routinely establish them. Each laboratory, therefore, must calibrate the SPRT according its customary process. The uncertainties associated to this calibration will be delivered by filling the document "EUROMET.T-K2.1 uncertaintyanalysis.xls".

The SPRT supporting the comparisons will be carefully selected paying a special attention to the stability of the instrument. The SPRT will be quartz sheathed.

<u>Task of participating labs:</u>

The traveling SPRT is to pass through the following sequence:

1) a measurement at the triple point of water (TPW)
2) a stabilization procedure
3) a second measurement at the triple point of water
4) measurements at metal fixed points in order of decreasing temperatures alternating with a measurement at the triple point of water.

- Upon receipt of the SPRT the host laboratory must inspect the devices for damage.
- If thermometer has not been received in due time pilot must be immediately informed to revise the timetable.
- The host must measure the resistance of the traveling SPRT in a TPW cell 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. The 0 mA resistance values of the traveling SPRT at the TPW must be corrected for the hydrostatic head to obtain R_{TPW}. The value of R_{TPW} must be communicated to the pilot laboratory. After receiving approval from the pilot laboratory to proceed with the comparison. the host laboratory can begin the SPRT stabilization procedure:
    - Carefully insert the SPRT into a furnace at 480 °C.

- Anneal the SPRT for two hours at 480 °C
- Carefully remove the SPRT from the furnace directly to the room

environment.

- Re-determine The value of R_{TPW}

- If the resistance at TPW increases after annealing contact pilot laboratory for further instructions
- If the decrease in the calculated TPW resistance of the SPRT after annealing is equivalent to 0.5 mK or greater proceed to a second SPRT stabilization procedure. Re-determine the value of $R_{TPW.}$ If the decrease in the calculated TPW resistance of the SPRT after second annealing is greater to 0.2 mK communicate with pilot laboratory for further instructions
- If the decrease is less then 0.5 mK the completed calibration can be performed.

  Calibrate the SPRT at all of the fixed points in the range of comparison i.e. measurements at TPW, Zn, TPW, Sn, TPW, In, TPW, Ga, TPW, Hg and TPW in that order. Existing techniques as practiced by the participating Laboratory must be used. For each metal fixed point the $W=R_T/R_{TPW}$ is calculated. R_{TPW} is the TPW resistance 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 3 different phase transitions (3 freezings for Zn. Sn. In. 3 meltings for Ga and 3 triple points for Hg will be performed.
- After completing all of the above measurements, the host laboratory must transmit the calibration report to the linking laboratory within 6 weeks.

Table II. Example:

Run for Zn fixed point	
Measurement in TPW	
Measurement in Zn fixed point	*W Zn 1*
Measurement in TPW	
Measurement in Zn fixed point	*W Zn 2*
Measurement in TPW	
Measurement in Zn fixed point	*W Zn 3*
Measurement in TPW	

6. Reporting of data

After BIM has completed its measurements, BIM will send its measurement report to NMi van Swinden Laboratorium. When NMi laboratory has received this measurement report, BIM is no longer allowed to make any changes in its results or in its uncertainties. When NMi has completed its measurements they will also make a measurement report of its results. When all measurements reports have been completed, they will be sent to BIM who will do the data analysis and write the comparison report.

The measurement report must include the following information:

- For each fixed point cell that was used in the comparison, determine (preferably using the circulating SPRT) and plot the change of phase transition temperature, *dT*, versus immersion, *dh*. On the same graph, plot the theoretical *dT/dh* curve using the hydrostatic pressure coefficients (mK/m of liquid) given in the ITS-90 text.
- Examples of freezing curves in In, Sn and Zn cells, melting curve in Ga cell and triple–point curve in Hg.
- Using the attached spreadsheet named 'EUROMET.T-K2.1 Calibrationdata.xls' to report the resistance ratios

$W=R_T/R_{TPW}$ where R_T is the resistance of the SPRT at each of the fixed points, and R_{TPW} is the resistance in the TPW cell obtained after the measurement of R_T. The values of R_T and R_{TPW} must be corrected for self heating, the hydrostatic head and if applicable, the pressure effect. Measurement should be performed when the plateau has reached a sufficient level of stability with respect to the reported uncertainty.

- Uncertainty analysis using the attached spreadsheet named 'EUROMET.T-K2.1 Uncertaintyanalysis. xls.'
- Details of instrumentation, fixed point cells and techniques used in the realization of the fixed points for this comparison should be given in the attached sheet 'EUROMET.T-K2.1 Instrumentationdetails.xls.'
- The immersion curves. the freezing/melting curves and the completed forms
  'EUROMET.T-K2.1 Calibrationdata.xls'
  'EUROMET.T-K2.1 Uncertaintyanalysis.xls'
  'EUROMET.T-K2.1 Instrumentationdetails.xls'
  should be e-mailed to the linking lab. A paper copy must be send by post to the linking lab.

7. Preparation of final reports

BIM will prepare final reports draft A within 4 weeks after all the results have been received. After this draft A will be sent to the other participant for the comments. The participant will be allowed two weeks for the comments. Then the draft B report will be prepared in two weeks.

Uncertainties

Participants are requested to use the attached spreadsheet 'EUROMET.T-K2.1 Uncertaintyanalysis.xls' to calculate and report their estimated uncertainties for the determination of resistance ratios obtained from the SPRT at the fixed points that were used in this comparison. Calculations of uncertainties should follow the guidelines set out in the ISO Guide (1993) to the Expression of Uncertainty in Measurement.

For each uncertainty component, a standard uncertainty u_i and its associated degrees of freedom must be provided. The value of u_i should be given in terms of temperature. For type A evaluation, the number of degrees of freedom is *n-1* where *n* is the number of measurements. For type B evaluation any input is assumed to have an infinite number of degrees of freedom. The combined uncertainty *U*, the effective degrees of freedom and subsequently the expanded uncertainty at 95% level of confidence are calculated as set out in the Guide.

To assist with the determination of measurement uncertainties, the following section explains the meanings of the uncertainty components given in 'EUROMET.T-K2.1 Uncertaintyanalysis.xls'.

W_t is determined according the following mathematical model obtained from the relationship.

$$W_t = \frac{(R_s + C_{Rs/3} + C_{Rs/4})*(X_t + C_{Xt/1} + C_{Xt/2} + C_{Xt/3} + C_{Xt/4} + C_{Xt/5} + C_{Xt/6} + C_{Xt/7})}{(R_s)*(X_{0.01°C} + C_{X0.01/1} + C_{X0.01/2} + C_{X0.01/3} + C_{X0.01/4} + C_{X0.01/5} + C_{X0.01/6} + C_{X0.01/7})}$$

$$W_t = (1 + D_{Rs/3} + D_{Rs/4}) \cdot \frac{(X_t + C_{Xt/1} + C_{Xt/2} + C_{Xt/3} + C_{Xt/4} + C_{Xt/5} + C_{Xt/6} + C_{Xt/7})}{(X_{0.01°C} + C_{X0.01/1} + C_{X0.01/2} + C_{X0.01/3} + C_{X0.01/4} + C_{X0.01/5} + C_{X0.01/6} + C_{X0.01/7})}$$

Where

Rs	reference resistor value at the time of TPW measurement
$D_{Rs/3}$	relative drift of the resistance of the reference between TPW and FP measurements $= C_{Rs/3} / R_s$
$D_{Rs/4}$	relative temperature variation of resistance of the reference between TPW and FP Measurements $= C_{Rs/4} / R_s$

Effects linked with triple point of water calibration:

$X_{0.01°C}$	reading on the bridge at the triple point of water
$C_{x0.01/1}$	water triple point reference including isotope variation
$C_{x0.01/2}$	Hydrostatic pressure correction
$C_{x0.01/3}$	Perturbing heat exchanges
$C_{x0.01/4}$	Self-heating correction
$C_{x0.01/5}$	Bridge linearity
$C_{x0.01/6}$	Ac/dc measurement correction
$C_{x0.01/7}$	SPRT internal insulation leakage correction

Effects linked with the considered fixed point calibration:

X_t	Reading on the bridge
$C_{Xt/1}$	Chemical impurities
$C_{Xt/2}$	Hydrostatic pressure correction
$C_{Xt/3}$	Perturbing heat exchanges
$C_{Xt/4}$	Self-heating correction
$C_{Xt/5}$	Bridge measurement correction, lack of linearity
$C_{Xt/6}$	Ac/dc measurement correction
$C_{Xt/7}$	Gas pressure correction

S_{Wt} W_t scatter

Any participant can complete this table with additional component for taking in account specific experimental conditions. In particular, it could be necessary to include a component linked with SPRT internal insulation leakage correction at the Ga fixed point. On the other hand, if component is considered as negligible they have to be quoted as "negligible" and it value must be justified.

Combined standard uncertainty on Wt

$$\sigma^2_{Wt} = \left(\frac{\delta Wt}{\delta D_{RS/3}}\right)^2 * \sigma^2_{D_{RS/3}} + \left(\frac{\delta Wt}{\delta D_{RS/4}}\right)^2 * \sigma^2_{D_{RS/4}}$$

$$+ \left(\frac{\delta Wt}{\delta X_{0.01°C}}\right)^2 * \sigma^2_{X_{0.01}} + \left(\frac{\delta Wt}{\delta C_{X0.01/1}}\right)^2 * \sigma^2_{C_{X0.01/1}} + ... + \left(\frac{\delta Wt}{\delta C_{X0.01/7}}\right)^2 * \sigma^2_{C_{X0.01/7}}$$

$$+ \left(\frac{\delta Wt}{\delta X_t}\right)^2 * \sigma^2_{X_t} + \left(\frac{\delta Wt}{\delta C_{Xt/1}}\right)^2 * \sigma^2_{C_{Xt/1}} + ... + \left(\frac{\delta Wt}{\delta C_{Xt/7}}\right)^2 * \sigma^2_{C_{Xt/7}}$$

$$+ 2 \cdot \rho_1 \cdot \left(\frac{\delta Wt}{\delta C_{X0.01/1}}\right) \cdot \left(\frac{\delta Wt}{\delta C_{Xt/1}}\right) \cdot \sigma_{C_{X0.01/1}} \cdot \sigma_{C_{Xt/1}}$$

$$+ .....$$

$$+ 2 \cdot \rho_6 \cdot \left(\frac{\delta Wt}{\delta C_{X0.01/6}}\right) \cdot \left(\frac{\delta Wt}{\delta C_{Xt/6}}\right) \cdot \sigma_{C_{X0.01/6}} \cdot \sigma_{C_{Xt/6}} + S_{W_t}$$

The values of $\rho_1, \rho_2, \rho_3, \rho_4, \rho_5, \rho_6$ will be taken as equal to Zero if the laboratory have not better information on these values. Taking these values as zero is justified because:
1) $\delta W_t / \delta C_{X0.001/i}$ is negative
2) The values of $\rho_1. \rho_2. \rho_3. \rho_4. \rho_5. \rho_6$ are positive.

Consequently to give a null value to these correlation coefficients leads to maximize the value of σ_{Wt}^2.

$$\sigma^2_t = \left(\frac{\delta t}{\delta W_t}\right)^2 * \sigma^2_{W_t}$$

In sheet "EUROMET.T-K2.1 Uncertainty analysis.xls" the sensitivity coefficient correspond to

$$\left(\frac{\delta t}{\delta W_t}\right) * \left(\frac{\delta W_t}{\delta i}\right)$$

for example the sensitivity coefficient linked with the quantity $C_{Xt/1}$ is

$$\left(\frac{\delta t}{\delta W_t}\right) * \left(\frac{\delta W_t}{\delta C_{Xt/1}}\right)$$

Components explanation and proposal of evaluation

Quantity	Standard Uncertainty	Method	Evaluation
X_t	Repeatability of readings. No change during a short time	- Same SPRT - Same cell - Same freezing - Same day	Type A Type B: PD rectangular and symmetrical: (Max value-Min value)/ 2√3
$C_{Xt/1}$	Purity	-During the recent EUROMET Workshop Dr B.Fellmuth from PTB explained clearly that it was physically impossible to quote the uncertainty linked to the impurities in simply using the Raoult's Law. Therefore it is proposed to quote this uncertainty from the dispersion of a batch of cells. This batch can be the property of the laboratory or it is the set of cells which have been participated to previous comparison (see proposal of PTB in attach document). -Obtained in scientific literature and/or technical specifications.	
$C_{Xt/2}$	hydrostatic pressure correction	Estimated from the uncertainty of the sensitive element position and the uncertainty of the free liquid level	Established by the Laboratory
$C_{Xt/3}$	Perturbing heat exchanges (between the sensor and the surrounding parts different in temperature from the liquid-solid phase change)	-Deviation from expected hydrostatic pressure correction obtained by changing immersion depth over 5 cm (length of the sensor) -Modification of the thermal exchange between thermometer and its environment -Use of different container design	Established by the Laboratory Type B (Max value-Min value)/ 2√3
$C_{Xt/4}$	self-heating correction	Resolution of the bridge readings. uncertainty on the ratio between the two measuring currents Variation in self heating correction observed in an apparent similar environment	Established by the Laboratory or obtained in scientific literature Type B (Max value-Min value)/ 2√3
$C_{Xt/5}$	bridge linearity	Use of calibrated resistor for checking the bridge. Comparison between readings on different bridges. Checking the symmetry of the bridge (R1/R2 = 1/ (R2/R1)?)	Established by the Laboratory or obtained in scientific literature
$C_{Xt/6}$	Difference between AC and DC measurements	Estimated by using DC and AC bridge	Established by the Laboratory or obtained in scientific literature Type B (Max value-Min value)/ 2√3
$C_{Xt/7}$	Gas pressure in the cell	Uncertainty on neutral gas pressure value during fixed point. 1 – open cells: uncertainty on line pressure measurement	Established by the Laboratory or obtained in scientific literature Type B

		2 - sealed cells: uncertainty on pressure measurement during sealing combined with temperature profile	(Max value-Min value)/ 2√3

Quantity	Standard Uncertainty	Method	Evaluation
$X_{0.01°C}$	a) Repeatability of readings. No change during a short time	- Same SPRT - Same cell and same mantle realization - Same day	Type A
	b) Repeatability of temperature realized by cell	- Same SPRT (assumed stable) - Same cell - Different realizations of the mantle (1) - Different dates of measurement for take into account mantle ageing (2)	Reasonably large set of data: type A Small number of data: type B (1)PD rectangular and symmetrical: (Max value-Min value)/ 2√3 (2)PD rectangular and not symmetrical: (Max value-Min value)/ √3
	c) Short-term Repeatability of SPRT to be calibrated	- Same cell - Variation between TPW measurement before and after the considered fixed point	
$C_{X0.01/1}$	Purity and isotopic composition	Comparison between several cells from different sources in the same conditions. Use of the interlaboratory comparison data.	Established by the Laboratory or obtained in scientific literature Type B (Max value-Min value)/ 2√3
$C_{X0.01/2}$	Hydrostatic pressure correction	Estimated from the uncertainty on the distance between the platinum sensor and the free liquid level	Established by the Laboratory Type B (Max value-Min value)/ 2√3
$C_{X0.01/3}$	Perturbing heat exchanges (between the sensor and the surrounding parts different in temperature from the liquid-solid phase change)	-Deviation from expected hydrostatic pressure correction obtained by changing immersion depth over 5 cm (length of the sensor) -Modification of the thermal exchange between thermometer and its environment -Use of different container design	Established by the Laboratory Type B (Max value-Min value)/ 2√3
$C_{X0.01/4}$	self-heating correction	Resolution of the bridge readings. uncertainty on the ratio between the two measuring currents Variation in self heating correction observed in an apparent similar environment	Established by the Laboratory or obtained in scientific literature Type B (Max value-Min value)/ 2√3
$C_{X0.01/5}$	bridge linearity	Use of calibrated resistor for checking the bridge. Comparison between readings on	Established by the Laboratory or obtained in

		different bridges. Checking the symmetry of the bridge (R1/R2 = 1/ (R2/R1)?)	scientific literature Type B (Max value-Min value)/ 2√3
$\boldsymbol{C_{X0.01/6}}$	Difference between AC and DC measurements	Estimated by using DC and AC bridge	Established by the Laboratory or obtained in scientific literature Type B (Max value-Min value)/ 2√3
$\boldsymbol{C_{X0.01/7}}$	SPRT internal Insulation leakage (if any)	Decrease in resistance over some hours in the triple point	Established by the Laboratory or obtained in scientific literature Type B (Max value-Min value)/ 2√3

Quantity	Standard Uncertainty	Method	Evaluation
$\boldsymbol{D_{RS/3}}$	Lack of stability of the reference resistance value	Negligible if measurement performed in a short time (within two successive days)	Established by the aboratory Type B (Max value-Min value)/ √3 (PD: rectangular no ymmetrical) (Max value-Min value)/ 2√3 (PD: rectangular symmetrical)
$\boldsymbol{D_{RS/4}}$	Change in value of the standard resistor with thermostat temperature	- uncertainty on calibrating temperature - uncertainty on temperature at time of use - uncertainty on temperature coefficient	Established by the Laboratory Type B (Max value-Min value)/ √3
$\boldsymbol{S_{Wt}}$	W_t scatter	- Same SPRT - Same cell - Different W values	Reasonably large set of data: type A Small number of data: type B PD rectangular and symmetrical: (Max value-Min value)/ 2√3 Different days