Final Report

23 April 2008

Report to the CCT on Key Comparison EUROMET.T-K4 (EUROMET Project 820)

Comparison of the realisations of the ITS-90 at the freezing points of Al (660,323 °C) and Ag (961,78 °C)

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CONTENT

1. Introduction

The EUROMET regional key comparison EUROMET.T-K4 was initiated during the EUROMET TC THERM meeting on 30./31. March 2004 in Ljubljana, Slovenia. PTB was chosen to be the pilot laboratory. All participants have globally agreed the protocol in its version 2 from 2005-01-12 (see Appendix A) during the EUROMET meeting in March 2005 in Vienna, Austria. The protocol was accepted by EUROMET as EUROMET Project 820. A previous version of the protocol was agreed by the chairman of CCT WG7 on 22.11.2004.

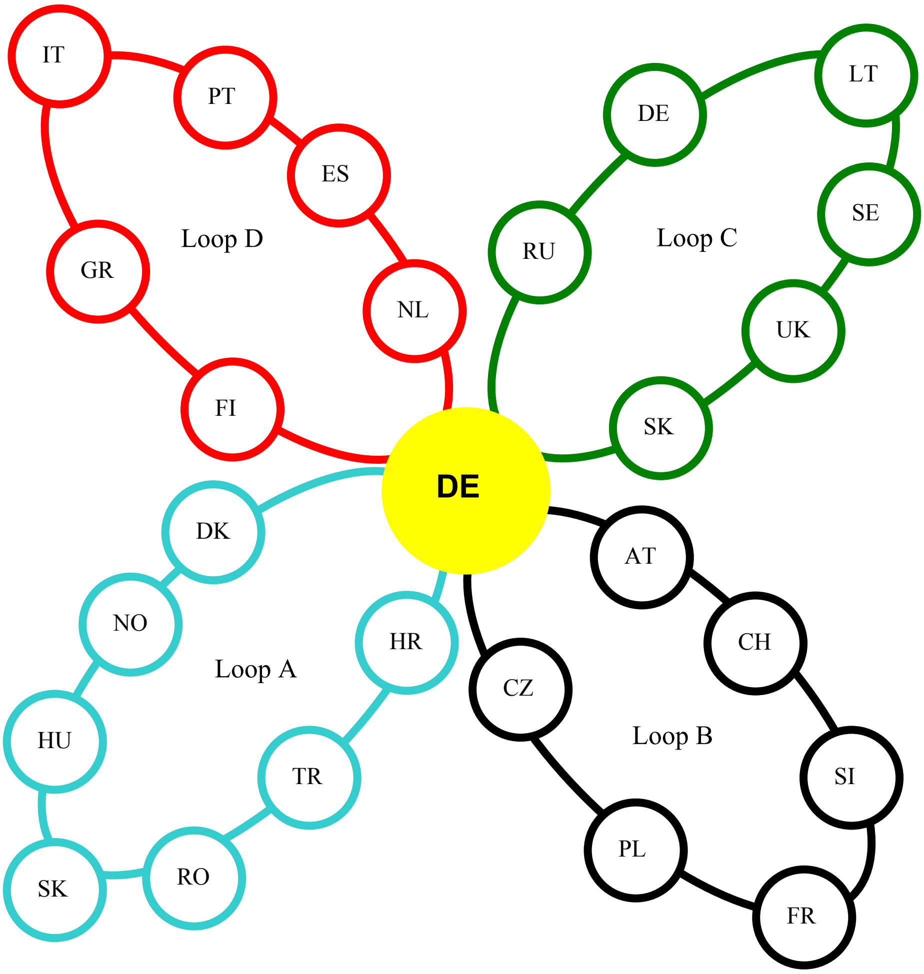
The procedure of EUROMET.T-K4 is not identical with the procedure of CCT-K4 [1]: While CCT-K4 was a direct comparison of Al and Ag fixed point cells, EUROMET.T-K4 requires the calibration of an SPRT (25 Ω) at the freezing point of Al and 2 HTSPRTs at the freezing point of Ag by each participant. Participants only interested in a comparison at the Al freezing point will only calibrate the SPRT (25 Ω). The procedure follows partly the procedures used in APMP.T-K4 [2].

During the preliminary characterization of the HTSPRTs by the pilot laboratory it was found that the thermometers are not as stable as desirable. Several improvements of the protocol have been considered and mostly rejected, because the necessary effort would have been too large. As a compromise, one additional Ag fixed point cell (provided by NMi/VSL) was compared only among the pilot and sub-pilot laboratories (see Appendix B). This allows for a better control of the stability of the thermometers. Moreover, the linkage of the loops can be based on more reliable measurements.

The comparison involves the 5 EUROMET NMIs that participated previously in CCT-T-K4 [BNM-INM (FR), INRiM (IT), NMi/VSL (NL), NPL (GB), PTB (DE)] as pilot or sub-pilots and additional nearly all European national laboratories. The comparison is divided in 4 loops. Besides PTB there will be another participant of CCT-K4 and/or CCT-K3 in each of the loops of EUROMET.T-K4.

The organisation of the comparison is presented in Fig. 1.1 and Tab. 2.1.

Thermometers



Ag fixed point cell

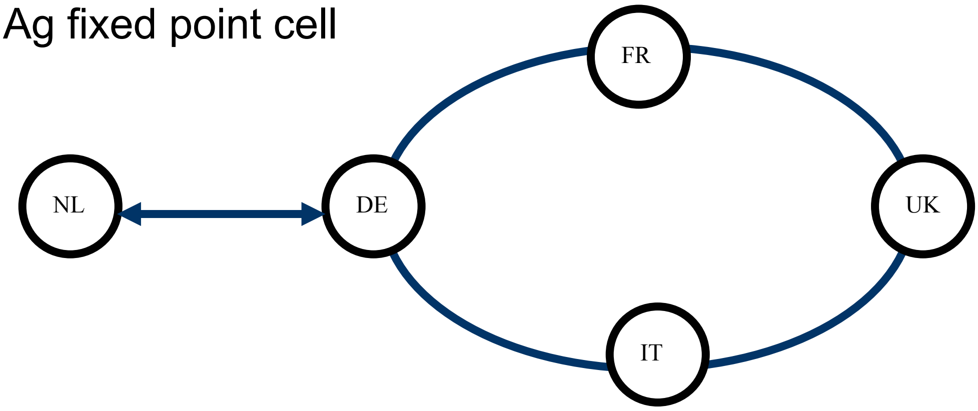


Fig. 1.1: Organisation of the comparison. Sub-pilots are SMU (SK) for loop A, LNE-INM (FR) for loop B, NPL (UK) for loop C and INRiM (IT) for loop D.

2. Schedule of the project

For a given laboratory, the time allowed for the measurements (calibration of 1 SPRT at the Al freezing point and 2 HTSPRTs at the freezing point of Ag) was estimated to be less than 4 weeks. The travelling time between two NMIs could be rated at 2 weeks. In agreement with these estimations the schedule followed Tab 2.1. Due to delays caused by several reasons the provisional schedule had to be changed several times. In brackets the actual dates when PTB received the first results from the NMIs (typically the first TPW measurements) are given.

Loop A (only Pt-25)	Loop B	Loop C	Loop D
DE	DE		
DK (02.12.04)	CZ (10.01.05)	DE	
NO (28.01.05)	PL (18.02.05)	SK (11.04.05)	DE
HU (18.03.05)	FR + Ag cell (27.04.05)	UK + Ag cell (16.05.06)	FI (29.04.05)
SK (SA)	SI (22.08.05)	SE (14.07.05)	GR (10.06.05)
RO (02.07.05)	CH (23.09.06)	LT (12.09.05)	IT + Ag cell (19.07.05)
TR (06.09.05)	AT	DE (19.10.05)	PT (03.01.06)
HR (02.11.06)	DE	RU (02.12.05)	ES (13.03.06)
DE(01.03.06)		DE (03.12.05)	NL + Ag cell (08.05.06)
			DE

Table 2.1: Schedule for Thermometers in EUROMET.T-K4. Sub-pilots are indicated in blue.

The Ag fixed point cell will be used by the pilot and sub-pilot laboratories to compare the realisations of the freezing point of Ag and to control the stability of the HTSPRTs. The schedule was organised in such a way that thermometers and the Ag fixed point cell should be at the same time in the laboratories of the sub-pilots, but arriving from and going to different laboratories.

3. Participating Laboratories

In alphabetic order of the following NMIs participated in the project
BEV (AT), CEM (ES), CMI (CZ), DTI (DK), EIM (GR), FSB (HR), GUM (PL), INM (RO), INRiM (IT), IPQ (PT), JV (NO), LNE-INM (FR), METAS (CH), MIKES (FI), MIRS (SI), NPL (UK), OMH (HU), PTB (DE), SMU (SK), SP (SE), UJ-PFI (LT), UME (TR), VNIIM (RU)

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4. Protocol and organisation of the project

The final version of the protocol (see Appendix A) was sent to the participants on January 20, 2005. Details for the intercomparison with an Ag freezing point cell can be found in Appendix B.

The comparison was divided into 4 loops. The participants of loop A performed measurements at the Al freezing point only, the participants of loops B, C and D performed measurements at the freezing points of Ag and Al.

The devices used for the intercomparison are listed in Tab. 4.1. It is worthwhile to note that a total of 11 HTSPRTs have been checked for stability. Only the most stable thermometers were used in the comparison but even for these thermometers the stability was in most cases not as good as desired. Four thermometers were destroyed during the project and have partly been repaired.

Tab. 4.1: SPRTs used in the project EUROMET 820

Loop	PRT	Manufacturer	Type	Ser. Number	Provider
A	SPRT	Hart	5681	1444	Hart
B	HTSPRT	Chin. Man.		93103	metas
	HTSPRT	Hart	5684	1068	Hart
	SPRT	Hart	5681	1445	Hart
C	HTSPRT	Chino	R800-3L	944RS13	MIKES
	HTSPRT	Hart	5684	1065	Hart
	SPRT	Hart	5681	1446	Hart
D	HTSPRT	Hart	5684	1041	BIPM
	HTSPRT	Hart	5684	1043	BIPM
	SPRT	Hart	5681	1450	Hart
Spare	HTSPRT	Hart	5684		BIPM
	HTSPRT	Hart	5684		Hart

PTB performed the initial and final measurements for all loops, while the measurements of the sub-pilots were done in the middle of the loops, including the measurements with the additional Ag freezing point cell.

All laboratories sent their results to the pilot, their measurement uncertainty budget and a list of their equipment used for the measurements. Additionally the measured immersion profiles for the Al freezing point were reported. The equipment list is given in Appendix C, a short version of the uncertainty budget can be found in Appendix D.

5. Results for measurements at the Al freezing point

The comparison at the freezing point of Al was carried out in 4 different loops. The first and last measurements were performed by the pilot laboratory PTB. In each loop a subpilot was included; the sub-pilot was also a participant of CCT K3 and except for loop A also of CCT K4.

5.1 Loop results

Tables 5.1a to 5.1d and Figures 5.1a to 5.1d present the *W*-values given by the participants. The uncertainties are given for *k* = 2. Only the average values of at least 3 realisations of the Al freezing points are given.

The uncertainties in Tab 5.1 and Fig. 5.1 are taken from the reports of the laboratories. The calculation of the combined uncertainty from the data provided by the participants has been critically reviewed, but no calculation errors have been found. This does not mean that the uncertainties given by the laboratories do not need further scrutiny.

Tab. 5.1a

Loop A, SPRT Ser. No. 1444		
Participant	*W* Al	*U*/mK
PTB (DE)	3,3758467	2,44
DTI (DK)	3,3758478	4,75
JV (NO)	3,3758479	3,55
OMH (HU)	3,3758345	2,64
SMU (SK)	3,3758515	1,97
INM (RO)	3,3758312	2,20
UME (TR)	3,3758458	3,08
FSB (HR)	3,3758356	12,24
PTB (DE)	3,3758582	2,44

Tab. 5.1b

Loop B, SPRT Ser. No. 1445		
Participant	*W* Al	*U*/mK
PTB (DE)	3,3756893	1,62
CMI (CZ)	3,3756921	3,63
GUM (PL)	3,3757013	2,84
LNE-INM (FR)	3,3757024	2,36
MIRS (SI)	3,3756860	2,5
METAS (CH)	3,3757072	1,51
BEV (AT)	3,3756977	3,41
PTB (DE)	3,3757032	1,31

Table 5.1c

Loop C, SPRT Ser. No. 1446		
Participant	*W* Al	*U*/mK
PTB (DE)	3,3756619	2,01
SMU (SK)	3,3756730	2,00
NPL (UK)	3,3756659	1,00
SP (SE)	3,3756709	2,25
UJ-PFI (LT)	3,3756709	3,33
VNIIM (RU)	3,3756715	1,11
PTB (DE)	3,3756756	2,34

Tab. 5.1d

Loop D, SPRT Ser. No. 1450		
Participant	*W* Al	*U*/mK
PTB (DE)	3,3757974	2,02
MIKES (FI)	3,3757968	3,23
EIM (GR)	3,3757568	5,06
INRiM (IT)	3,3757940	3,33
IPQ (PT)	3,3757984	3,04
CEM (ES)	3,3757994	6,77
VSL (NL)	3,3758028	3,34
PTB (DE)	3,3758112	2,81

Tab. 5.1: Results for the measurements at the Al freezing point. Uncertainties are given for *k* = 2.

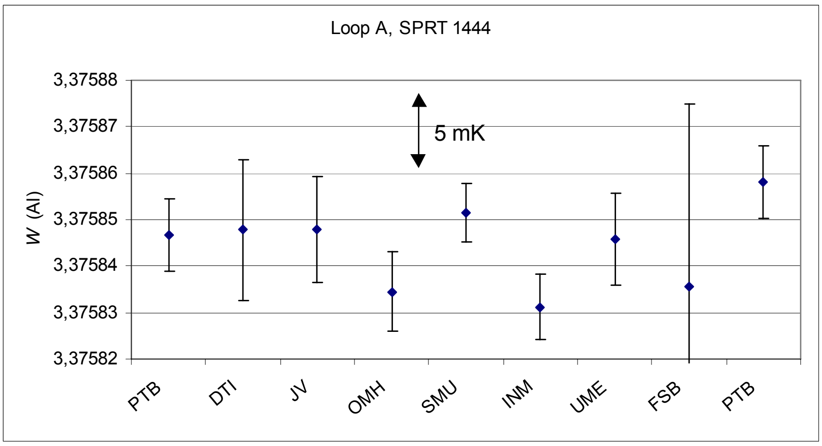


Fig. 5.1a

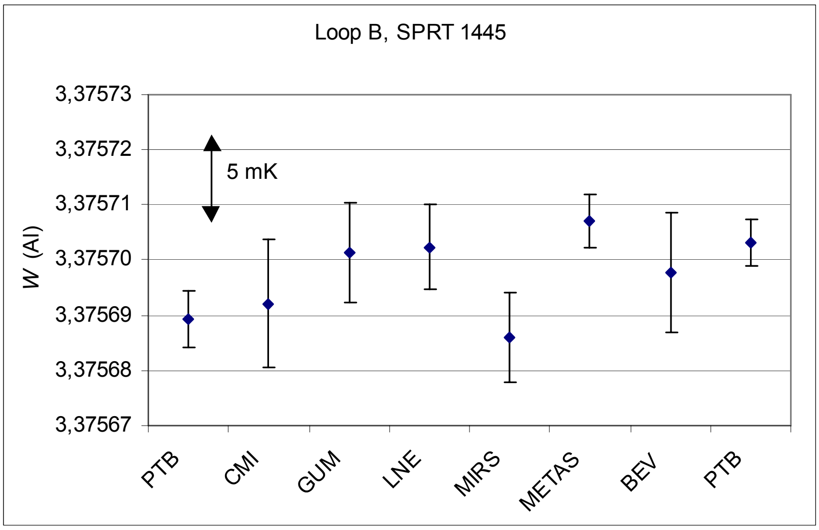


Fig. 5.1b

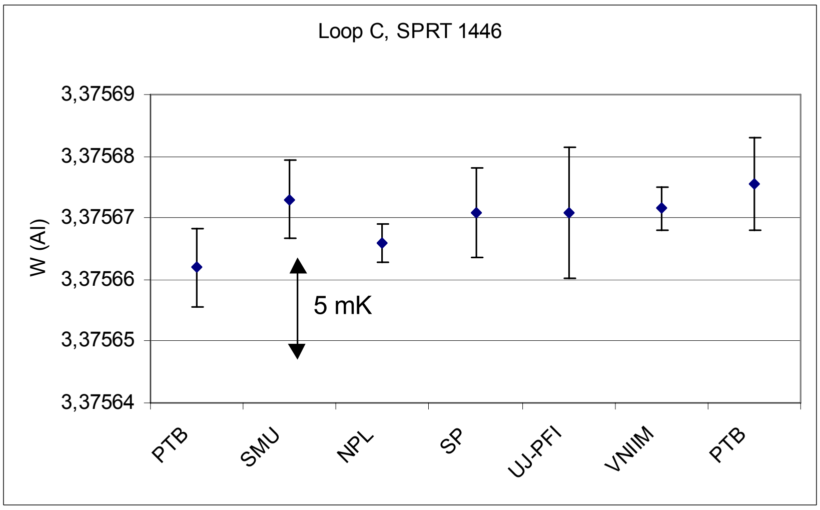


Figure 5.1c

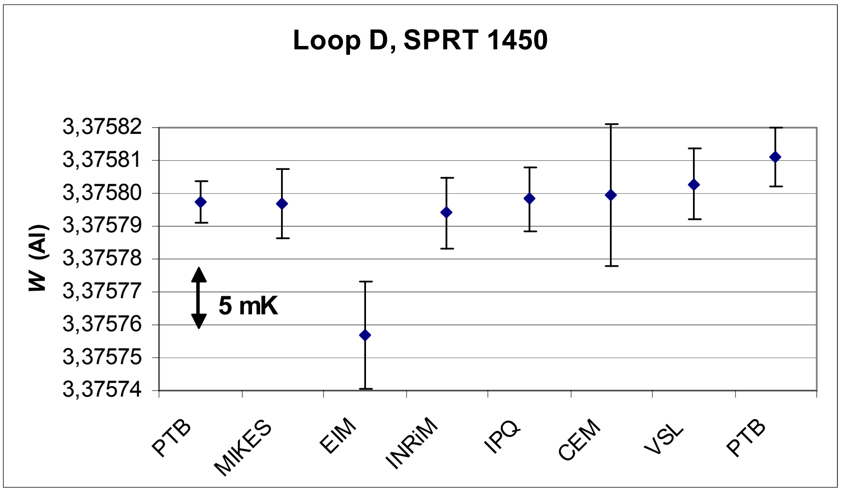


Figure 5.1d

Fig.5.1: Results for the measurements at the Al freezing point

For all loops the final measurements at PTB result in a value which is higher by an equivalent of 3 mK to 4 mK than the first measurement. It has carefully been checked if the reason might be a shift in the standards of PTB (see Appendix F). The check SPRT used parallel to all measurement did not show such an effect. It is therefore assumed that the reason is a drift of the thermometers. This is strange, but not completely unlikely, because all thermometers were new and from the same batch of fabrication. But it should be mentioned that after a minimum of 5 realisations of the Al freezing points all thermometers were stable at the TPW better than 0,3 mK.

5.2 Linking the loops for the measurements at the Al freezing point

The procedure for linking the loops in EUROMET 820 follows basically the procedure used for EUROMET.T-K3 (EUROMET 552). In the report of EUROMET 552 [3] all formulas are given in detail, and they will not be repeated in full length here.

The pilot PTB is the only laboratory with measurements in all loops. Therefore results will be given relative to the PTB results. The reference value for each loop is the simple mean of the first and last measurements at PTB. All values are converted to a temperature scale using the following formula:

$$T_{Lab} - T_{PTB} = (\mathrm{W}_{\mathrm{Lab}} - \mathrm{W}_{\mathrm{PTB}}) \times \left(\frac{\delta T}{\delta W} \right)_{Al}$$

The conversion factor has the value $\left(\frac{\delta T}{\delta W} \right)_{Al}$ = 312,02 K

The uncertainty of the temperature difference to PTB is given by

$$U_{(T_{Lab} - T_{PTB})} = 2 \sqrt{\left(u^2_{T_{Lab}} + u^2_{rep(PTB)} + u^2_{inst(PTB)} + u^2_{Stab} \right)}$$

The contributions to the uncertainty have been determined as follows:

- The uncertainty u_{Tlab} is taken from Tab. 5a to 5d.
- The uncertainty contribution $u_{\mathrm{rep(PTB)}}$ resulting from the reproducibility of the PTB measurement is taken from the scatter of the *W*-values during the measurements (see Appendix D). The maximum of the values from the initial and final measurements was used.
- The uncertainty $u_{\mathrm{inst(PTB)}}$ is caused by changes in the instrumentation of PTB between initial and final measurements. The main source is the replacement of the Al fixed point cell.
- The contribution u_{Stab} from the instability of the thermometers has been calculated from the initial and final calibration at PTB according to

$$u_{Stab} = \frac{\left| (W_{final}) - (W_{initial}) \right|}{2\sqrt{3}} \times \left(\frac{\delta T}{\delta W} \right)_{Al}$$

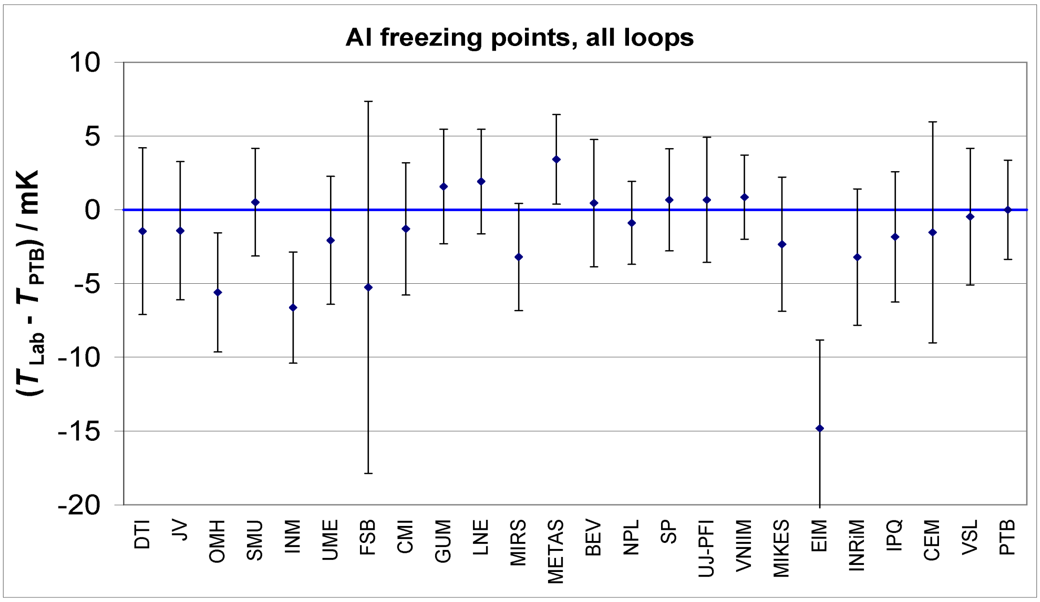
The contribution for the four thermometers are listed in Tab. 5.2

Tab. 5.2 Uncertainty contribution ($k = 1$) for the linkage of the loops

Thermometer	$u_{\mathrm{rep(PTB)}}$ / mK	$u_{\mathrm{inst(PTB)}}$ / mK	u_{Stab} / mK
SN 1444	1,08	0,30	1,04
SN 1445	0,291	0,30	1,25
SN 1446	0,359	0,30	1,23
SN 1450	0,97	0,30	1,24

The summary of differences between PTB and the other participants is presented in Fig. 5.2.

Fig. 5.2 All loops relative to PTB. Uncertainties are given for $k = 2$.



5.3 EUROMET Reference Value (*ERV*) for the Al freezing point

The designation of the *ERV* follows again basically the procedure proposed in EUROMET 552. There are 3 possibilities for the determination of the *ERV*: the simple mean, the weighted mean and the median applied to the data of all participants. These methods have been used to determine the temperature difference between a possible *ERV* and the temperature measured by PTB, i.e. the methods were applied to the data given in Figure 5.2. For the calculation the result of EIM was eliminated as an obvious outlier. The possible *ERV*s are presented in Tab. 5.3. For the mean two different uncertainties are given, calculated as the standard deviation of the mean

$$u^2 = \frac{1}{\mathrm{n}(\mathrm{n}-1)} \sum_1^n (x_i - \bar{x})^2 \text{ with } x_\mathrm{i} = T_\mathrm{Lab} - T_\mathrm{PTB}$$

or the uncertainty (given in brackets) calculated from the uncertainty of the individual laboratories:

$$u^2 = \frac{1}{\mathrm{n}^2} \sum_1^n u^2(x_i)$$

Tab 5.3: Possible *ERV*s and their uncertainty U (k = 2)

$ERV - T_\mathrm{mean}$ (PTB) / mK		
	Value / mK	U / mK (k = 2)
Mean	-1,180	1,03 (1,06)
Weighted mean	-0,691	0,84
Median	-1,295	1,272

The three possible *ERV*s agree within their uncertainty. It was decided to use the simple mean as the *ERV* with the uncertainty calculated from the uncertainties of the laboratories:

ERV (820) = T_PTB - (1,18 ± 1,06) mK (Uncertainty for k = 2)

When calculating the uncertainty of the deviation of the laboratory results from the *ERV*, it has to be taken into account that there is a correlation between the uncertainty of the laboratories and the uncertainty of the *ERV*. With the uncertainty of the *ERV* being calculated from the uncertainty of the participants, with an argument similar to that given by Cox [4] the uncertainty of the deviation from the *ERV* can be approximated by:

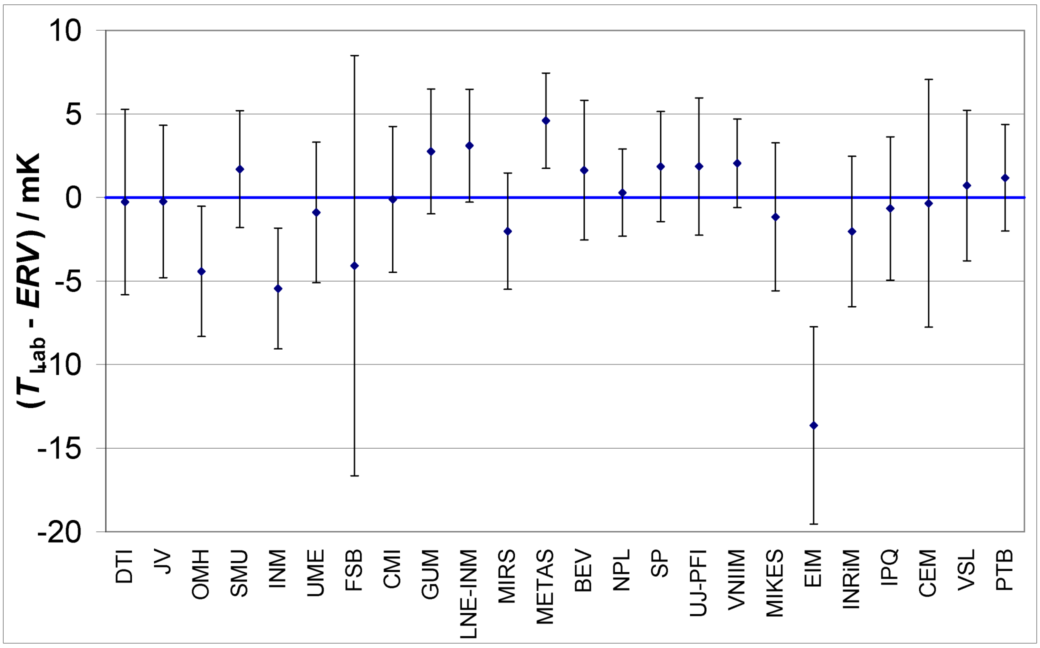
$$u^2(T_\mathrm{Lab} - ERV) = u^2(T_\mathrm{Lab}) - u^2(ERV)$$

The values for (T_Lab – *ERV* (820)) are given in Tab. 5.4 and in Fig. 5.3.

Tab. 5.4: Summary of the results for the Al freezing point. Uncertainties are given for k = 2.

Participant	T_{Lab} - T_{PTB}	U/mK, k = 2	T_{Lab}-ERV / mK	U / mK, k = 2	E_{n}
DTI	-1,45	5,65	-0,27	5,55	-0,05
JV	-1,42	4,69	-0,24	4,56	-0,05
OMH	-5,60	4,04	-4,42	3,90	-1,13
SMU	0,52	3,65	1,70	3,50	0,48
INM	-6,63	3,77	-5,45	3,61	-1,51
UME	-2,08	4,34	-0,90	4,21	-0,21
FSB	-5,26	12,62	-4,08	12,57	-0,32
CMI	-1,30	4,49	-0,12	4,36	-0,03
GUM	1,58	3,88	2,76	3,73	0,74
LNE	1,92	3,54	3,10	3,38	0,92
MIRS	-3,20	3,64	-2,02	3,48	-0,58
METAS	3,42	3,04	4,60	2,85	1,61
BEV	0,45	4,31	1,63	4,18	0,39
NPL	-0,89	2,81	0,29	2,61	0,11
SP	0,67	3,46	1,85	3,29	0,56
UJ-PFI	0,67	4,24	1,85	4,11	0,45
VNIIM	0,86	2,86	2,04	2,65	0,77
MIKES	-2,34	4,55	-1,16	4,43	-0,26
EIM	-14,82	5,99	-13,64	5,90	-2,31
INRiM	-3,21	4,62	-2,03	4,50	-0,45
IPQ	-1,84	4,42	-0,66	4,29	-0,15
CEM	-1,53	7,49	-0,35	7,41	-0,05
VSL	-0,47	4,63	0,71	4,51	0,16
PTB	0,00	3,36	1,18	3,19	0,37

Fig. 5.3: All participants relative to the *ERV* for Al



5.3.1 Birge ratio test

The statistical consistency of a comparison can be investigated by the so-called Birge ratio R_B [5]. This test compares the observed spread of the results with the spread expected from the individual uncertainties. The relevance of this test applied to the data given in Tab. 5.4 is somewhat doubtful, because the method used for the linkage of the loops leads to the result that uncertainties from the PTB measurements contribute to all data. Therefore they cannot be considered to be independent.

The Birge ratio is defined as

$$R_B = \frac{\mathrm{u_{ext}}(ERV)}{\mathrm{u_{int}}(ERV)}, \qquad \text{with} \qquad u_{ext} = \sqrt{\frac{\sum_{i=1}^{n}\left[(T_i - ERV)/u_i\right]^2}{(n-1)\sum_{i=1}^{n} u_i^{-2}}} \quad .$$

u_{int} is the uncertainty of the *ERV* as given in Tab. 5.3. A value of R_B close to 1 or less suggests that results are consistent, whereas values much greater than is suggests that results are inconsistent.

The data presented in Tab. 5.4 lead to a Birge ratio of R_B = 1,28; if the result from EIM is neglected the Birge ratio is reduced to R_B = 1,04. Considering the problems with linking the loops this seems to be reasonable.

5.4 Linkage between EUROMET 820 and CCT K3 and CCT K4

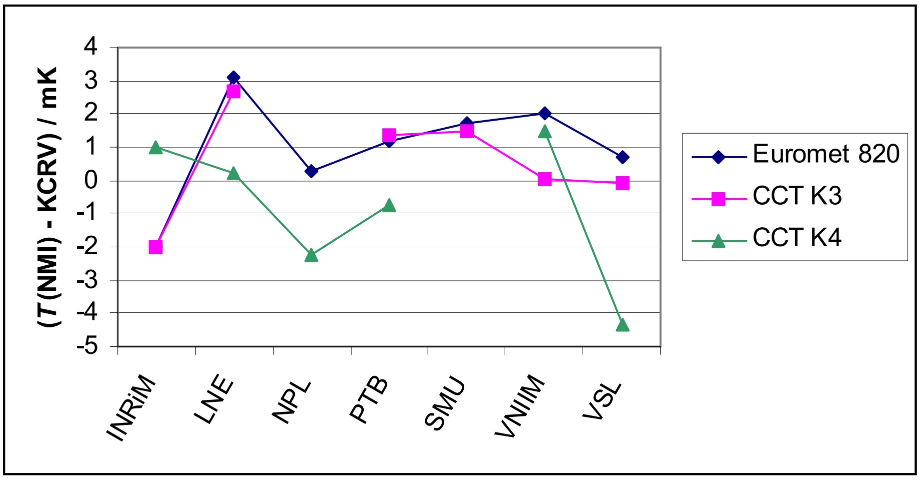
The linkage to CCT K3 and CCT K4 is made via those NMIs which participated in both key comparisons. It is assumed that for this group of NMIs the average value remained the same between the comparisons. Therefore a deviation in the average difference to the *KCRV* is caused by a difference in the *KCRV*s. Tab. 5.5 gives an overview for the relevant key comparisons. The average was taken as the simple mean, and the uncertainty is the standard deviation of the mean, multiplied with *k* = 2.

Tab. 5.5: Al freezing point: T_{Lab} – *KCRV* in mK for different interlaboratory comparisons. Uncertainties are given for *k* = 2. For the CCT K3 the "unofficial" *ARV* was used.

	(*T*(NMI) – *KCRV*) / mK for the Al freezing point		
Laboratory	EUROMET 820	CCT K3	CCT K4
INRiM	-2,03 ± 4,16	-2,02 ± 1,29	1,00 ± 1,42
LNE-INM	3,10 ± 3,40	2,67 ± 2,27	0,25 ± 1,44
NPL	0,29 ± 2,63		-2,25 ± 2,20
PTB	1,18 ± 3,22	1,38 ± 1,79	-0,75 ± 1,50
SMU (mean loops A and B)	1,70 ± 3,15	1,48 ± 1,31	
VNIIM	2,04 ± 2,68	0,05 ± 1,85	-1,50 ± 1,74
VSL	0,71 ± 4,17	-0,07 ± 1,58	-4,35 ± 3,98
Average (INRiM, LNE, PTB, SMU, VNIIM, VSL)	1,12 ± 1,42	0,58 ± 1,33	
Average (INRiM, LNE, NPL, PTB, VNIIM, VSL)	0,88 ± 1,42		-0,77 ± 1,80

The average was taken as the simple mean, and the uncertainty is the standard deviation of the mean.

Fig. 5.4: Results for the freezing points of Al for participants in EUROMET 820, CCT K3 and CCT K4. Uncertainties are given in Tab. 5.5.



The results are alos shown in Fig. 5.4. In most cases the agreement between the different comparisons is within uncertainties. The agreement between EUROMET 820 and CCT K3 seems to be better than between EUROMET 820 and CCT K4. From the last two lines in Tab. 5.5 it follows:

ERV **(Al, 820) = ARV (Al, CCT K3) + (0,54 ± 1,96) mK (*k* = 2)**

ERV **(Al, 820) =** ***KCRV*** **(Al, CCT K4) + (1,65 ± 2,28) mK (*k* = 2)**

The uncertainty of the correction is the sum in quadrature of the average values.

6. Results for Measurements at the Ag freezing point

The results of the measurements at the freezing point of Ag suffer from the instability of the used High Temperature Standard Platinum Resistance Thermometers (HTSPRTs). This was known after the initial measurements at PTB, and for this reason 2 HTSPRTs were used in each loop and additionally the sub-pilot carried out an additional intercomparison with an Ag fixed point cell provided by NMi-VSL.

6.1. Intercomparison with a Ag Freezing Point Cell

An additional interlaboratory comparison among the pilot and the sub-pilots was organised for the freezing point of silver. This allowed to correct some doubtful data caused by instable HTSPRTs.

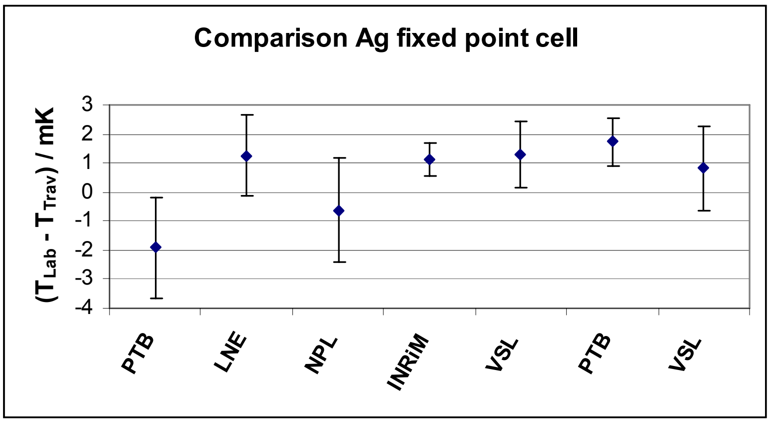
This additional comparison followed basically the protocol of CCT-K4. The travelling instrument was a re-sealable Ag freezing point cell which was provided by NMi-VSL. During the transportation the crucible with the silver ingot was taken out of the cell in order to minimise the risk of damage.

The participating NMIs measured the temperature difference between their cell used in EUROMET 820 and the travelling cell of NMi-VSL. The result of the measurements is given in Tab. 6.1 and Fig. 6.1 . The uncertainty of the temperature difference was estimated to be the standard deviation of the individual measurements. It should be mentioned that the uncertainties given in Tab. 6.1 and Fig. 6.1 are the uncertainty for the temperature difference between two fixed point cells and not the uncertainty of the ITS-90 realisation of the Ag freezing point.

Tab. 6.1: Ag freezing point: $T_{\text{Lab}} - T_{\text{Trav}}$ for the sub-pilots of EUROMET 820. Uncertainties for the difference between the national standard and the travelling cell are given for $k = 1$.

Laboratory	$(T_{\text{Lab}} - T_{\text{Trav}})$ / mK	u / mK
PTB	-1,92	0,86
LNE-INM	1,26	0,69
NPL	-0,62	0,90
INRiM	1,13	0,28
VSL	1,30	0,56
PTB	1,72	0,42
VSL	0,81	0,72

Fig. 6.1: Ag freezing point: $T_{\text{Lab}} - T_{\text{Trav}}$ for the sub-pilots of EUROMET 820. Uncertainties are given for $k = 2$.



PTB made an initial and a final measurement. The deviation of 3,64 mK between these two measurement is quite large. There are indications that a contamination of the silver in the travelling cell (resulting in a decreased freezing point temperature) between the first PTB and the LNE measurement can not be excluded. This suspicion is based on the fact that in the beginning of the measurements at LNE-INM the head of the travelling cell was overheated, resulting in a melting of the sealing. This may have lead to additional impurities in the cell, leading probably to a to a decrease in the freezing temperature of the silver cell. Unfortunately no measurement of NMI-VSL before the measurement of PTB is available, but NMI-VSL repeated the measurement after the final measurement of PTB and received good agreement with the previous measurement. Due to the unclear situation the initial PTB measurement is neglected in the further evaluation.

For the evaluation of the HTSPRT measurement the difference between the sub-pilots and the PTB must be calculated. For the estimation of the uncertainty a contribution for stability of the travelling cell was estimated as

$$u_{Stab} = \frac{|T_{VSL1} - T_{VSL2}|}{\sqrt{3}} = 0{,}28\,\mathrm{mK}$$

The uncertainty for the difference between sub-pilot and PTB is then calculated according to

$$U_{(T_{Lab} - T_{PTB})} = 2\sqrt{u_{T(Lab)}^2 + u_{T(PTB)}^2 + u_{Stab}^2}$$

The uncertainty of T(PTB) was estimated to be the smaller value (0,42 mK) of both uncertainties given in Tab 6.1, because partly the PTB uncertainties are included in u_{Stab}. The results are given in Tab. 6.2.

Table 6.2: Ag freezing point: $T_{\mathrm{Lab}} - T_{\mathrm{PTB}}$

Laboratory	($T_{\mathrm{Lab}} - T_{\mathrm{PTB}}$) / mK	U / mK (k = 2)
LNE-INM	-0,46	1,71
NPL	-2,34	2,06
INRiM	-0,59	1,15
VSL	-0,67	1,51

The values given in Tab. 6.2 will be used to link the measurements with the HTSPRTs of the subpilots to the corresponding measurements at PTB.

6.2 Intercomparison with HTSPRTs: Loop results

The comparison at the freezing point of Ag was carried out in 3 different loops. The first and last measurements were performed by the pilot laboratory PTB. In each loop a sub pilot was included; the sub-pilot was also a participant of CCT K4. Tables 6.3a to 6.3f and Fig. 6.3a to 6.3f present the W-values given by the participants. The uncertainties are given for k = 2.

Only the average values of at least 3 realisations of the Ag freezing points are given. The calculation of the combined uncertainty from the data provided by the participants has been critically reviewed, but no calculation errors were found. This does not mean that the uncertainties given by the laboratories do not need further scrutiny. Please notice that the increased uncertainties for the final PTB measurements are caused by the instability of the thermometers.

Tab. 6.3: Results for the HTSPRTs. Uncertainties are given for $k = 2$.

Tab. 6.3a

Loop B, HTSPRT Ser. No. 1068		
Participant	*W*(Ag)	*U*/mK
PTB (DE)	4,2864783	5,36
CMI (CZ)	4,2864403	4,66
GUM (PL)	4,2864393	4,31
LNE-INM (FR)	4,2864485	3,12
MIRS (SI)	4,2864542	11,00
METAS (CH)	4,2864254	6,34
BEV (AT)	4,2864227	3,31
PTB (DE)	4,2863253	10,20

Tab. 6.3b

Loop B, HTSPRT Ser. No. 93103		
Participant	*W*(AG)	*U*/mK
PTB (DE)	4,2866423	3,81
CMI (CZ)	4,2866176	5,14
GUM (PL)	4,2866365	3,51
LNE-INM (FR)	4,2866518	8,61
MIRS (SI)	4,2866398	6,94
METAS (CH)	4,2866377	6,99
BEV (AT)	4,2866386	6,40

Tab. 6.3c

Loop C, HTSPRT Ser. No. 1065		
Participant	*W*(AG)	*U*/mK
PTB (DE)	4,2864822	2,88
SMU (SK)	4,2864397	2,70
NPL (UK)	4,2864433	3,67
SP (SE)	4,2864565	7,94
UJ-PFI (LT)	4,2864497	8,30
PTB (DE)	4,2863535	16,10
VNIIM (RU)	4,2862177	2,89
PTB (DE)	4,2861253	22,13

Tab. 6.3d

Loop C, HTSPRT Ser. No. 944RS13		
Participant	W(AG)	*U*/mK
PTB (DE)	4,2861421	4,45
SMU (SK)	4,2854753	3,29
NPL (UK)	4,2855277	6,63
SP (SE)	4,2855697	8,12
UJ-PFI (LT)	4,2856229	8,52
PTB (DE)	4,2855281	8,00
VNIIM (RU)	4,2855851	8,77
PTB (DE)	4,2854623	36,34

Tab. 6.3e

Loop D, HTSPRT Ser. No. 1041		
Participant	*W*(AG)	*U*/mK
PTB (DE)	4,2864090	7,33
MIKES (FI)	4,2863581	10,12
EIM (GR)	4,2863639	9,61
INRiM (IT)	4,2863307	4,95
IPQ (PT)	4,2863423	8,01
CEM (ES)	4,2862974	15,41
VSL (NL)	4,2863028	5,42
PTB (DE)	4,2862115	26,63

Tab. 6.3f

Loop C, HTSPRT Ser. No. 1043		
Participant	*W*(AG)	*U*/mK
PTB (DE)	4,2864869	5,76
MIKES (FI)	4,2864518	9,77
EIM (GR)	4,2864742	8,02
INRiM (IT)	4,2864607	3,18
IPQ (PT)	4,2864728	10,14
CEM (ES)	4,2864648	15,70
VSL (NL)	4,2864430	3,41
PTB (DE)	4,2863748	10,97

Fig. 6.3: Results for the HTSPRTs

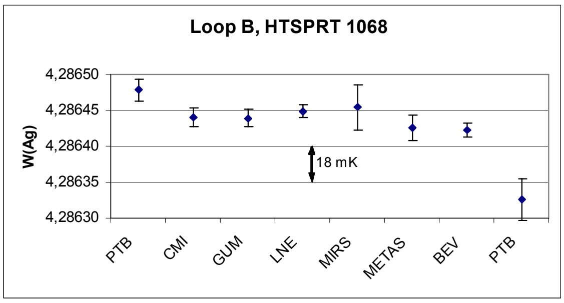


Fig. 6.3a

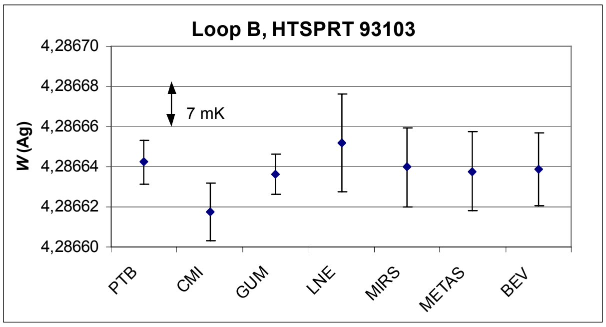


Fig. 6.3b

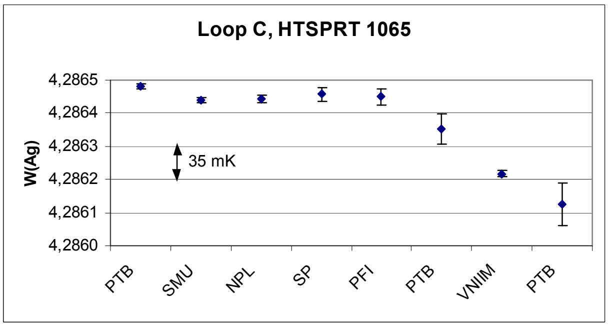


Fig. 6.3c

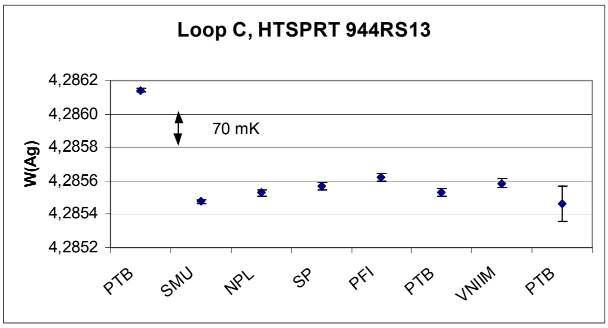


Fig. 6.3d

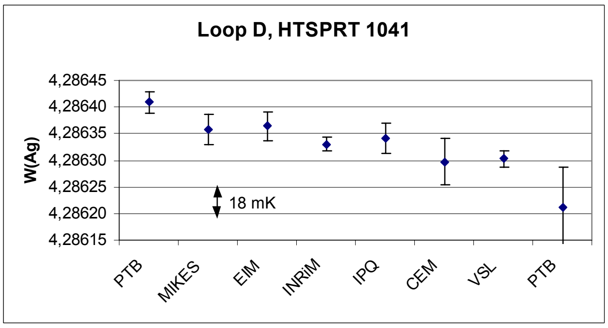


Fig. 6.3e

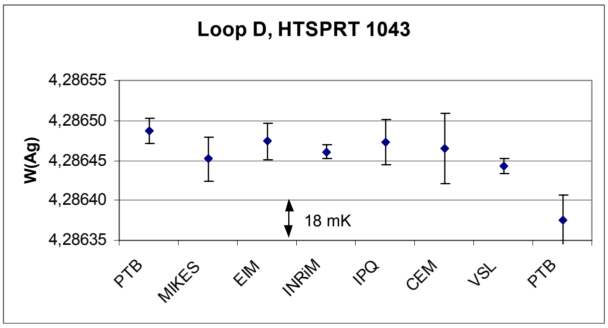


Fig. 6.3f

6.3 Review and correction / compensation of the data

It is known that the instability of HTSPRTs is caused mainly by two reasons: mechanical stress of the sensor and poisoning of the sensor by impurities. Metallic and other impurities can diffuse at high temperatures (for instance the freezing temperature of Ag) through the thermometer sheath and into the sensor material. For this reason all HTSPRTs should be cleaned with acid (for instance soaked in 20% nitric acid) before inserting them into an annealing furnace or a Ag fixed point cell. From the final measurements at PTB it seems that not all laboratories followed this procedure: for some thermometers the drift during the measurements was extremely large.

For a test of the hypothesis of contamination for some thermometers an extra cleaning procedure with acid was applied and the acid then was analysed by mass spectrometry. Several metals were found, in particular Ag, Pb, Cu, Mo and Pt.

In few cases a thermometer has also been measured at the Al freezing point before starting the loops. It was found that also the *W*-value for Al has changed by a large amount. It therefore was concluded that the drift of the thermometers was not caused by other instrumental instabilities at PTB, but by poisoning of the sensors in the thermometers.

The drift of a HTSPRT caused by contamination can partly be compensated [6]: Basically the change of the resistance is not or only slightly temperature dependent. By comparing the resistance *R*(TPW) at the triple of water measured by the laboratory with the resistance measured by the pilot or sub-pilot a compensation can be applied:

$$W = \frac{R(t)}{R(TPW)} \rightarrow W^* = \frac{R(t)+\Delta R}{R(TPW)+\Delta R}$$

The shift ΔR in the resistance can easily be determined from the measured resistance *R*(TPW) at the triple point of water. It has been found for the measurements at the Al freezing point that all measurements at the TPW agreed within 1 mK or better (see Appendix F). The reference value for *R*(TPW) is more or less arbitrary and will only lead to the same shift of all compensated W^*. For convenience as reference the measurements of the subpilots were chosen, because these measurements are in the middle of the loops. Details and an estimation of the uncertainty of the compensation are given in Appendix E.

This compensation has been applied to both thermometers in all 3 loops. For each loop it will be discussed if the compensation should be applied or not. If the decision is not clear, no compensation for the drift of the thermometer will be applied. It will also be discussed if measurements should be eliminated (outliers).

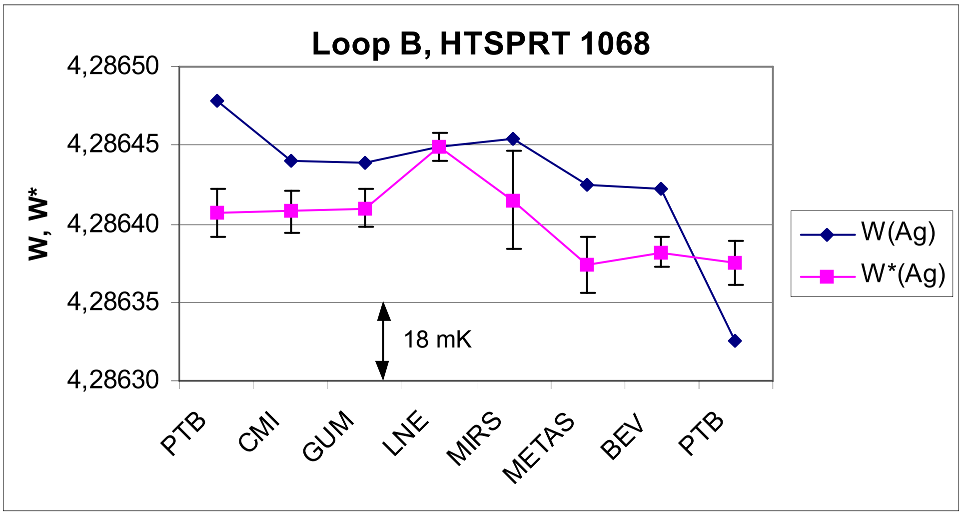
For the freezing point of Ag there is an additional comparison between pilot and sub-pilots using an Ag fixed point cell. The temperature difference between pilot and sub pilots is therefore known with relatively small uncertainty, and the results of the sub-pilots can also be used for the linkage of the loops. The details will be discussed for all thermometers of the comparison.

The procedure for linking the loops together will be similar to the procedure used for the measurements at the Al freezing point: all measurement will be given as difference to the PTB measurement, because the pilot PTB is the only participant in all loops. For this purpose a value called “Thermometer PTB Value, *TPV*” will be calculated for each thermometer. As already explained the measurement of the sub-pilots can also be used for the calculation of the *TPV*, because the differences between pilot and sub-pilots have been determined by an additional intercomparison with an Ag fixed point cell. Because in each loop 2 HTSPRTs were used, for the calculation of the deviation to the PTB measurements the average of both thermometers will be used.

6.3.1 Loop B

For comparison the original *W* and compensated *W** for both HTSPRTs in the loop are shown in Figs. 6.4a and 6.4b.

Fig. 6.4a: Original *W* and compensated *W**. The uncertainty is only given for W* (see Appendix E)



HTSPRT Ser. No. 1068: The compensation leads to a better agreement between the first and the final measurement of PTB, while the deviations between the other measurements seem even to be improved. In particular there seems to be a shift between the first three and the last three measurements. Because the improvement for the PTB measurements is quite substantial, the compensated values will be used, although there are also good arguments to delete this loop completely. The details for the compensation that was applied are given in Tab. 6.4a

Tab 6.4a: Compensation for thermometer HTSPRT 1068

Euromet 820(C) th. 1065						
Participant	W_{AG}	R_{TP} / Ω	$U(W)$ / mK	ΔR_{TP} / μΩ	W_{AG}*	$U(W^*)$ / mK
PTB	4,286478	0,2494043	5,36	-5,4	4,2864075	5,72
CMI	4,286440	0,2494072	4,66	-2,5	4,2864078	5,07
GUM	4,286439	0,2494075	4,31	-2,2	4,2864098	4,75
LNE	4,286449	0,2494097	3,12	0	4,2864485	3,71
MIRS	4,286454	0,2494067	11,00	-3,0	4,2864150	11,18
METAS	4,286425	0,2494058	6,34	-3,9	4,2863735	6,65
BEV	4,286423	0,2494066	3,40	-3,1	4,2863820	3,94
PTB	4,286325	0,2494135	10,2	3,75	4,2863775	10,39

The thermometer PTB value (*TPV*) will be calculated as the simple mean of both PTB measurements and the LNE measurement. The uncertainty component (k = 2) for the instability of the thermometer is set to be the difference between the initial PTB measurement and the corrected LNE measurement. The uncertainty of the linkage of sub-pilot via the measurement of the Ag freezing point cell is neglected in all cases. The details for the calculation of the *TPV* are given in Tab. 6.4b.

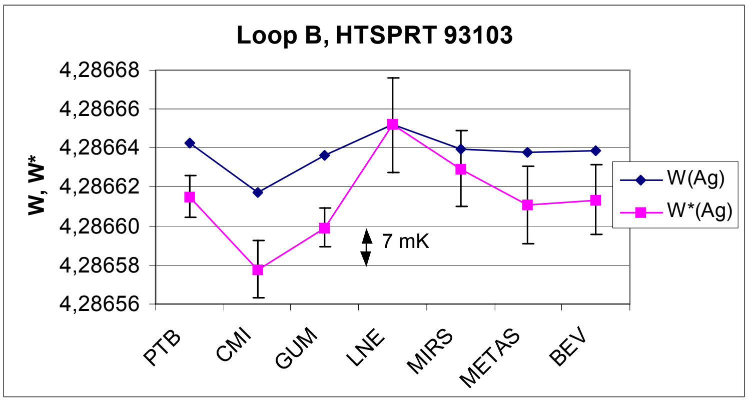
Tab 6.4b: Thermometer PTB value for HTSPRT Ser. No. 1068

Pilot / Sub-pilot	*W**	Deviation from PTB (from FP comparison)	Corrected *W* (reference: PTB)	*TPV*	*u*(*TPV*) / mK (*k* = 1)
PTB	4,2864075				
LNE	4,2864485	+0,46 mK ΔW = +1,31E-6	4,2864498		
PTB	4,2863748				
				4,2864107	7,44

HTSPRT Ser. No. 93103: This thermometer was destroyed close to the end of the measurements at BEV; therefore no final measurement at PTB is available. There is no reason to apply a compensation. It is remarkable that for both HTSPRTs the compensation results in a considerable increase of the LNE measurement, but of no other measurements. The reason for this effect is not clear.

The *TPV* will be calculated from the measurements at PTB and LNE as the simple mean. The uncertainty component (*k* = 1) for the instability of the thermometer is set to the difference between the first PTB measurements and the LNE measurement. Because of the strange behaviour of the LNE measurement if the compensation is applied, the uncertainty of the TPV will be increased by 50%.

Fig. 6.4b: Original *W* and corrected *W**. The uncertainty is only given for *W**.



The details for the calculation of the *TPV* are given in Tab. 6.4b.

Tab 6.4b: Thermometer PTB value for HTSPRT Ser. No. 93103

Pilot / Sub-pilot	*W*	Deviation from PTB (from FP comparison)	Corrected *W* (reference: PTB)	*TPV*	*u*(*TPV*) / mK (*k* = 1)
PTB	4,2866432		4,2866432		
LNE	4,2866518	+0,46 mK ΔW = +1,31E-6	4,2866531		
				4,2866482	5,23

Average Values for the Loop

The temperature difference T_{Lab} - $T(TPV)$ is calculated as already explained for the measurements at the Ag freezing point according to the equation

$$T_{Lab} - T(TPV) = (\mathrm{W_{Lab}} \text{ - } \mathrm{TPV}) \times \left(\frac{\delta T}{\delta W}\right)_{Ag} \quad \text{with} \quad \left(\frac{\delta T}{\delta W}\right) = 352K$$

The results for both HTSPRTs of loop B are shown in Fig. 6.4c.

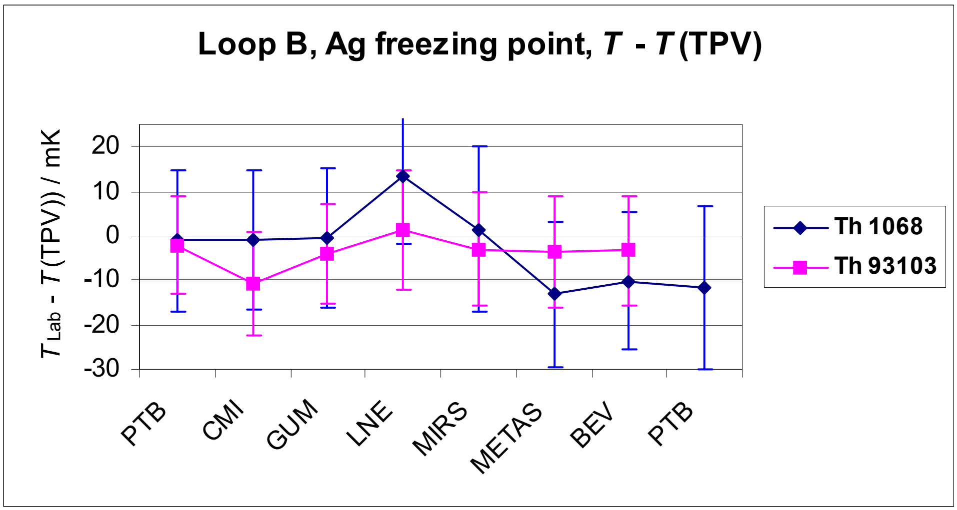


Fig. 6.4c

The results for both HTSPRTs are then summarized by calculating the weighted mean for (T - $T(TPV)$) of both thermometers, and the final result is presented in Fig. 6.4d. The details for the calculation are given in Tab. 6.4c.

Tab. 6.4c: **Details for the calculation of (T_{Lab} – T(TPV)) for loop B and its uncertainty.**

Loop B

Participant	Thermometer	W	$U(W)$ / mK	W^*	$U(W^*)$ / mK	TPV	U(TPV) / mK	T_{Lab}- T(TPV)	$U(T_{\mathrm{Lab}}$- T(TPV))	**T_{Lab}- T(TPV)**	**U (average)**
			k = 2		k = 2		k = 2	/ mK	/ mK, k = 2	**/mK, average**	**/ mK, k = 2**
PTB	Th 1068			4,2864075	5,72	4,2864107	14,89	-1,13	15,95	**-1,77**	**9,12**
	Th 93103	4,2866423	3,81			4,2866482	10,45	-2,08	11,12		
CMI	Th 1068			4,2864078	5,07	4,2864107	14,89	-1,01	15,73	**-7,32**	**9,36**
	Th 93103	4,2866176	5,14			4,2866482	10,45	-10,77	11,65		
GUM	Th 1068			4,2864098	4,75	4,2864107	14,89	-0,31	15,63	**-2,85**	**9,01**
	Th 93103	4,2866365	3,51			4,2866482	10,45	-4,12	11,02		
LNE-INM	Th 1068			4,2864485	3,71	4,2864107	14,89	13,31	15,34	**6,54**	**10,15**
	Th 93103	4,2866518	8,61			4,2866482	10,45	1,27	13,54		
MIRS	Th 1068			4,286415	11,18	4,2864107	14,89	1,50	18,62	**-1,57**	**10,40**
	Th 93103	4,2866398	6,94			4,2866482	10,45	-2,96	12,54		
METAS	Th 1068			4,2863735	6,65	4,2864107	14,89	-13,11	16,31	**-7,21**	**9,96**
	Th 93103	4,2866377	6,99			4,2866482	10,45	-3,70	12,57		
BEV	Th 1068			4,286382	3,94	4,2864107	14,89	-10,11	15,40	**-5,99**	**9,59**
	Th 93103	4,2866386	6,40			4,2866482	10,45	-3,38	12,25		
PTB	Th 1068			4,2863775	10,39	4,2864107	14,89	-11,70	18,16		
	Th 93103					4,2866482	10,45				

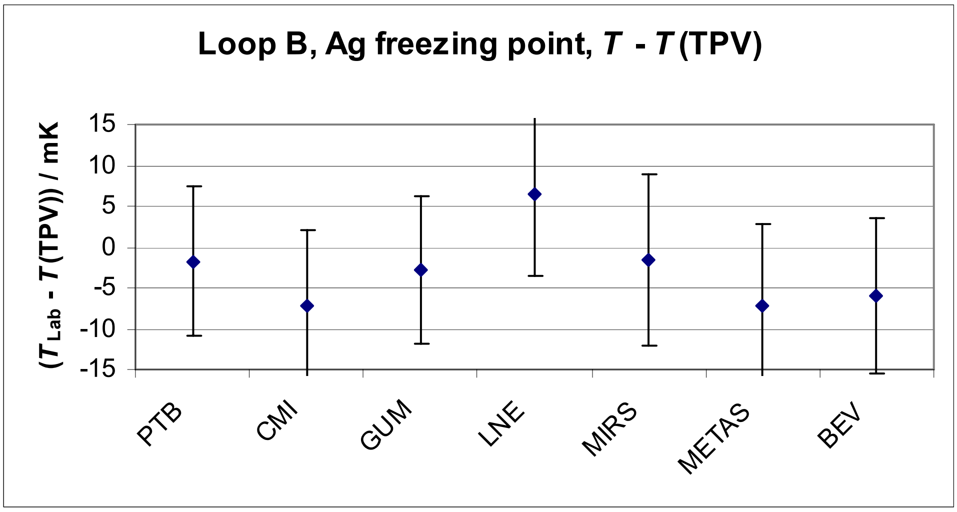
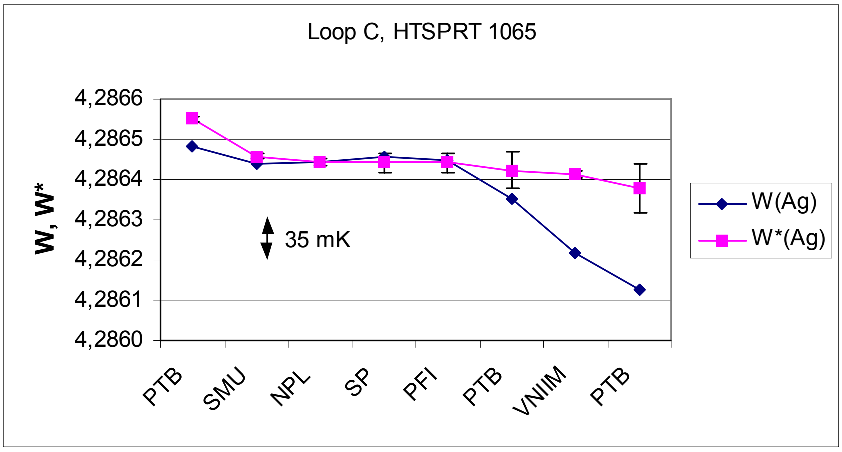


Fig. 6.4d

6.3.2 Loop C

In loop C there are 3 measurements of PTB. For comparison the original *W* and corrected *W* for both HTSPRTs in the loop are shown in Figs. 6.5a and 6.5b.

Fig. 6.5a: Original *W* and corrected *W**. The uncertainty is only given for *W**



For HTSPRT Ser. No. 1065 the compensated values will be used. It is remarkable how well the jump between PFI and PTB is cancelled out by the compensation. The details for the compensation that was applied are given in Tab. 6.5a.

Tab 6.5a

Euromet 820(C) th. 1065						
Participant	W_{AG}	R_{TP} / Ω	U(W) / mK	ΔR_{TP} / μΩ	W_{AG}*	U(W*) / mK
PTB	4,2864822	0,2518220	2,88	5,4	4,2865500	3,51
SMU	4,2864397	0,2518180	2,70	1,4	4,2864585	3,36
NPL	4,2864433	0,2518166	3,67	0	4,2864433	4,18
SP	4,2864565	0,2518155	7,94	-1,1	4,2864421	8,19
PFI	4,2864497	0,2518161	8,30	-0,5	4,2864428	8,54
PTB	4,2863535	0,2518238	16,09	7,3	4,2864233	16,21
VNIIM	4,2862177	0,2518315	2,89	14,9	4,2864122	3,51
PTB	4,2861253	0,2518354	22,13	18,8	4,2863784	22,22

The *TPV* will be the simple mean of the second PTB measurements and the NPL measurement.

The details for the calculation of the *TPV* are given in Tab. 6.5b. The uncertainty of the *TPV* is set to be the difference between the NPL and the second PTB (compensated) values.

Tab 6.5b: Thermometer PTB value for HTSPRT Ser. No. 1065

Pilot / Sub-pilot	*W**	Deviation from PTB (from FP comparison)	Corrected *W** (reference: PTB)	*TPV*	*u*(*TPV*) / mK (*k* = 1)
PTB	4,2864233		4,2864233		
NPL	4,2864433	+2,34 mK ΔW = +6,65E-6	4,2864500		
				4,2864367	9,40

HTSPRT Ser. No. 944RS13

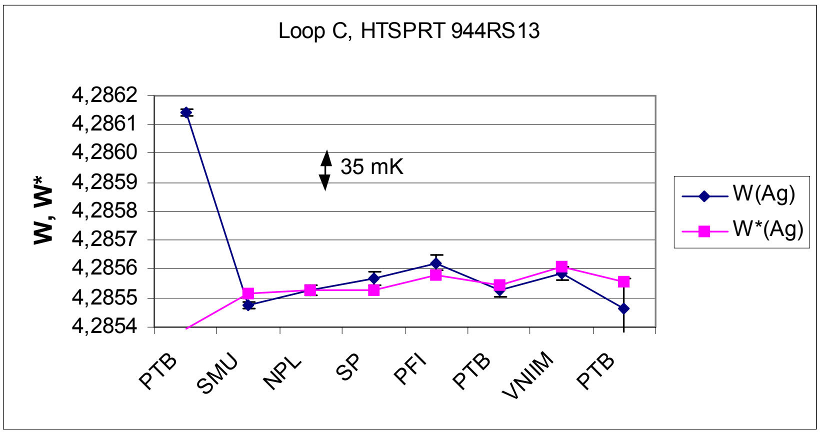


Fig. 6.5b

For HTSPRT Ser. No. 944RS13 the compensated values *W** will be used. The compensation does not correct the first PTB measurement completely. It should be noted that the thermometer was at BEV between the measurements at PTB and SMU, but the measurements were cancelled due to technical problems at BEV. It is not clear what happened to the thermometer in the meantime. The details for the compensation that was applied are given in Tab. 6.5c.

Tab. 6.5c

Euromet 820(C) th. 944RS13						
Participant	W_{AG}	R_{TP} / Ω	U(W) / mK	ΔR_{TP} / μΩ	W_{AG}*	*U*(*W**) / mK
PTB	4,2861421	0,2614649	4,55	-59,4	4,2853949	4,97
SMU	4,2854753	0,2615274	3,29	3,1	4,2855146	3,85
NPL	4,2855277	0,2615243	6,63	0	4,2855277	6,93
SP	4,2855697	0,2615209	8,12	-3,4	4,2855271	8,36
PFI	4,2856229	0,2615209	8,52	-3,4	4,2855796	8,75
PTB	4,2855281	0,2615256	8,00	1,3	4,2855445	8,25
VNIIM	4,2855851	0,2615263	8,77	1,9	4,2856094	9,00
PTB	4,2854623	0,2615318	36,34	7,5	4,2855589	36,39

The *TPV* will be the simple mean of the last PTB measurements and the NPL measurement. The uncertainty of the *TPV* is set to be the difference between these two measurements. The details for the calculation of the *TPV* are given in Tab. 6.5d.

Tab 6.5d: Thermometer PTB value for HTSPRT Ser. No. 944RS13

Pilot / Sub-pilot	*W**	Deviation from PTB (from FP comparison)	Corrected *W** (reference: PTB)	*TPV*	*u*(TPV) / mK (*k* = 1)
NPL	4,2855277	+2,34 mK ΔW = +6,65E-6	4,2855344		
PTB	4,2855589		4,2855589		
				4,2855467	8,62

The results for both HTSPRTs of loop C are shown in Fig. 6.5d.

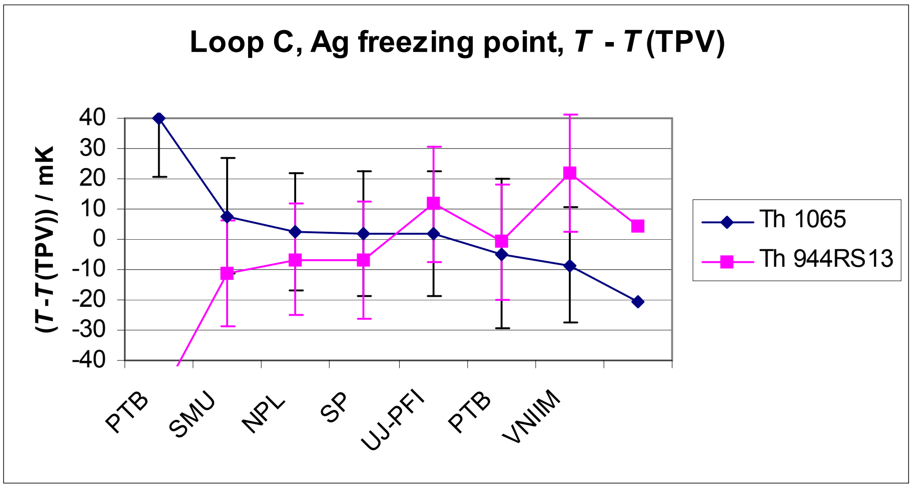


Fig. 6.5d

The results for both HTSPRTs are then summarized by calculating the weighted mean for Δ*T* of both thermometers, and the final result is presented in Fig. 6.5e. The details for the calculation are given in Tab. 6.5e.

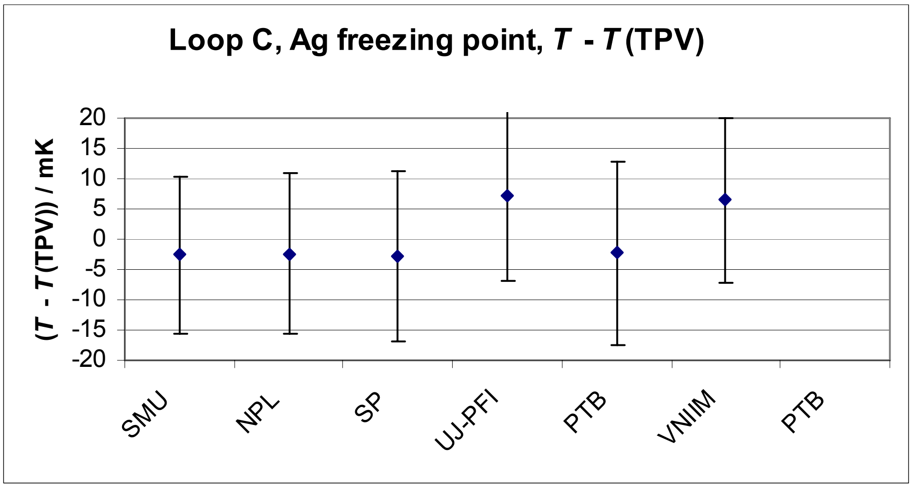


Fig. 6.5e

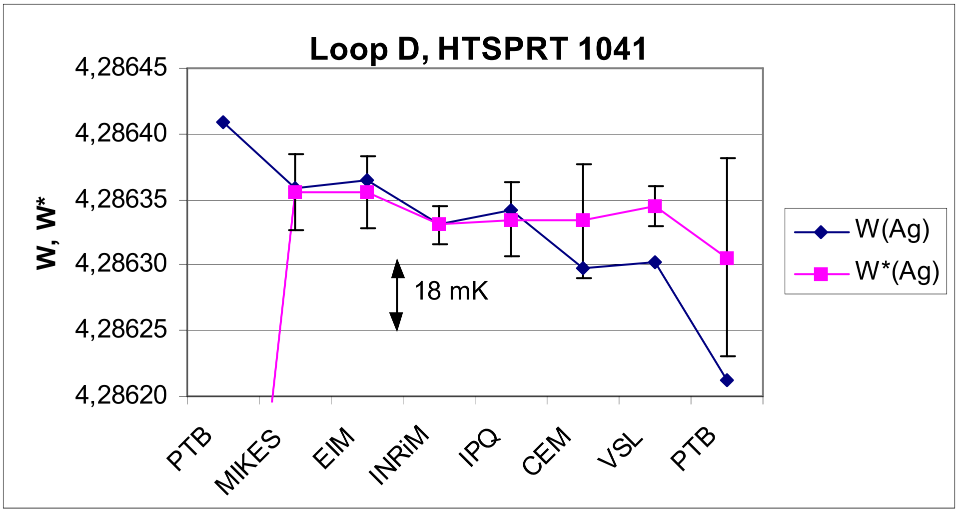
Tab. 6.5e: Details for the calculation of (T_{Lab} – T(TPV)) for loop C and its uncertainty.

Loop C									
Participant	Thermomter	W^*	$U(W^*)$ / mK	TPV	U(TPV) / mK	T_{Lab}- T(TPV)	U(T_{Lab}-T(TPV))	**T_{Lab}- T(TPV)**	**U (average)**
			k = 2		**k** = 2	/ mK	/ mK, **k** = 2	**/ mK, average**	**/ mK, k = 2**
PTB	Th 1065	4,2865500	3,51	4,2866437	18,80	39,88	19,12		
	Th 944RS13	4,2853949	4,97	4,2855467	17,24	-53,43			
SMU	Th 1065	4,2864585	3,36	4,2866437	18,80	7,67	19,10	**-2,55**	**12,97**
	Th 944RS13	4,2855146	3,85	4,2855467	17,24	-11,30	17,66		
NPL	Th 1065	4,2864433	4,18	4,2866437	18,80	2,32	19,26	**-2,34**	**13,37**
	Th 944RS13	4,2855277	6,93	4,2855467	17,24	-6,69	18,58		
SP	Th 1065	4,2864421	8,19	4,2866437	18,80	1,90	20,51	**-2,80**	**14,00**
	Th 944RS13	4,2855271	8,36	4,2855467	17,24	-6,90	19,16		
UJ-PFI	Th 1065	4,2864428	8,54	4,2866437	18,80	2,15	20,65	**7,17**	**14,11**
	Th 944RS13	4,2855796	8,75	4,2855467	17,24	11,58	19,33		
PTB	Th 1065	4,2864233	16,21	4,2866437	18,80	-4,72	24,82	**-2,24**	**15,14**
	Th 944RS13	4,2855445	8,25	4,2855467	17,24	-0,77	19,11		
VNIIM	Th 1065	4,2864122	3,51	4,2866437	18,80	-8,62	19,12	**6,47**	**13,64**
	Th 944RS13	4,2856094	9,00	4,2855467	17,24	22,07	19,45		
PTB	Th 1065	4,2863784	22,22	4,2866437	18,80	-20,52			
	Th 944RS13	4,2855589	36,39	4,2855467	17,24	4,29			

6.3.3 Loop D

For comparison the original *W* and corrected *W** for both HTSPRTs in the loop are shown in Figs. 6.6a and 6.6b.

Fig. 6.6a: Original *W* and corrected *W**. The uncertainty is only given for *W**



For HTSPRT Ser. No. 1041 the corrected *W**-values will be used. The change between the initial PTB measurement and the MIKES measurement is largely overcorrected by the compensation; it is not understood what happened. Therefore the initial PTB measurement will be ignored. During the measurements of the other participants there seems to be a continuous poisoning of the thermometer, which can be corrected quite well. The compensation does not work sufficiently for the last PTB measurement, which therefore also will be ignored. *TPV* will be chosen as the simple mean of the INRiM and the NMi-VSL values. The uncertainty of the *TPV* was set to be the difference between the compensated INRiM and VSL measurement.

The details for the compensation that was applied is given in Tab. 6.6a.

Tab. 6.6a

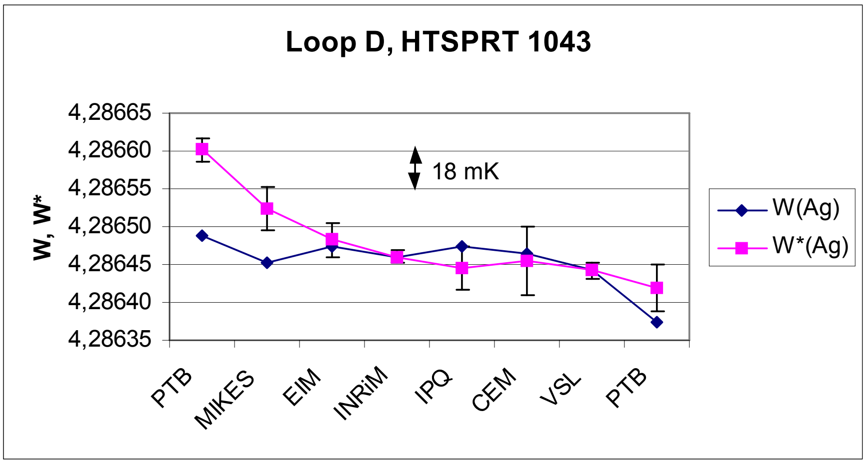
Euromet 820(D) th. 1041						
Participant	W_{AG}	R_{TP} / Ω	*U*(*W*)	ΔR_{TP} / μΩ	W_{AG}*	*U*(*W**) / mK
PTB	4,2864090	0,2537027	7,33	-42,5	4,2858584	7,60
MIKES	4,2863581	0,2537450	10,12	-0,2	4,2863549	10,32
EIM	4,2863639	0,2537445	9,61	-0,7	4,2863552	9,82
INRiM	4,2863307	0,2537452	4,95	0	4,2863307	5,34
IPQ	4,2863423	0,2537446	10,00	-0,6	4,2863346	10,20
CEM	4,2862974	0,2537480	15,40	2,8	4,2863336	15,53
VSL	4,2863028	0,2537484	5,42	3,3	4,2863451	5,78
PTB	4,2862115	0,2537525	26,63	7,3	4,2863057	26,70

The details for the calculation of the *TPV* are given in Tab. 6.6b.

Tab. 6.6b: Thermometer PTB value for HTSPRT Ser. No. 1041

Pilot / Sub-pilot	*W**	Deviation from PTB (from FP comparison)	Corrected *W** (reference: PTB)	*TPV*	*u*(*TPV*) / mK (*k* = 1)
INRiM	4,2863307	+0,59 mK ΔW = +1,68E-6	4,2863324		
NMi-VSL	4,2863451	+0,67 mK ΔW = +1,90E-6	4,2863470		
				4,2863397	5,14

Fig. 6.6c: Original *W* and corrected *W**. The uncertainty is only given for *W**



For HTSPRT Ser. No. 1043 the compensation does not seem to make any sense. There is not enough information about he details of the drift of the thermometer: it would be helpful to have information on the drift of *W*(Al) and *W*(Ga), but that is beyond the scope of this intercomparison. The uncompensated values (from Tab. 6.3f) will be used, but without the PTB measurements. *TPV* will be chosen as the simple mean of the INRiM and the NMi-VSL values. The uncertainty of *TPV* is set to be the difference between INRiM and the VSL value.

The details for the calculation of the *TPV* are given in Tab. 6.6c.

Tab. 6.6c: Thermometer PTB value for HTSPRT Ser. No. 1043

Pilot / Sub-pilot	*W*	Deviation from PTB (from FP comparison)	Corrected *W** (reference: PTB)	*TPV*	*u*(*TPV*) / mK (*k* = 1)
INRiM	4,2864607	+0,59 mK Δ*W* = +1,68E-6	4,2864624		
NMi-VSL	4,2864430	+0,67 mK Δ*W* = +1,90E-6	4,2864449		
				4,2864537	6,16

The results for both HTSPRTs of loop D are shown in Fig. 6.6d

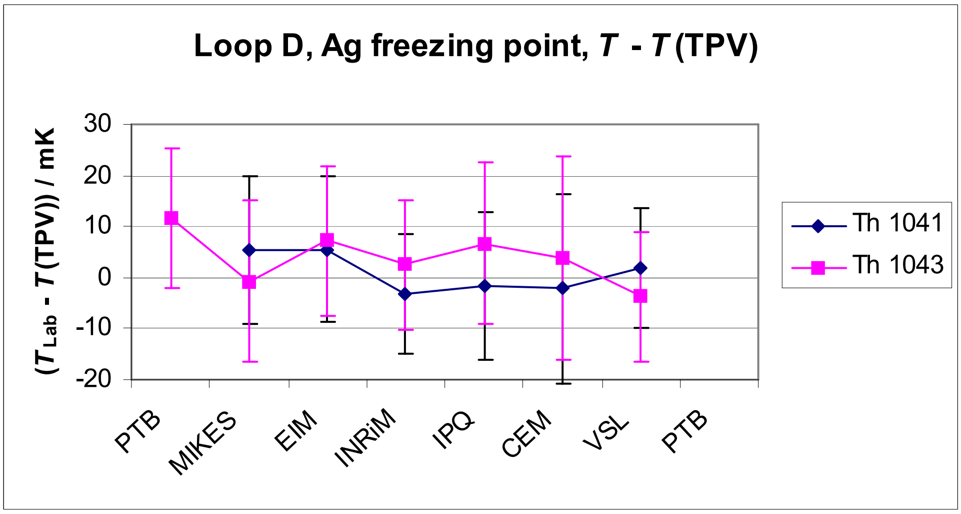


Fig. 6.6d

The results for both HTSPRTs are then summarized by calculating the weighted mean for $T_{Lab} - T(TPV)$ of both thermometers, and the final result is presented in Fig. 6.6e. The details for the calculation are given in Tab. 6.6d.

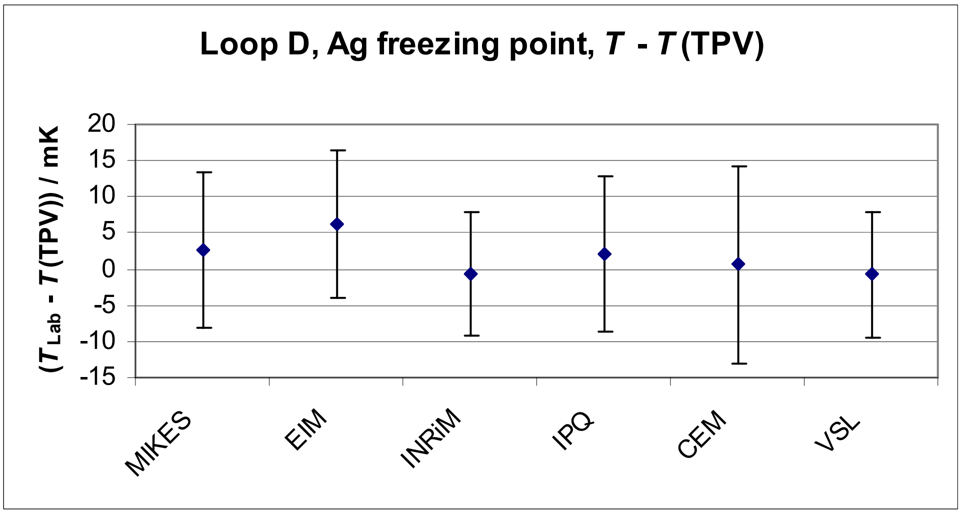


Fig. 6.6e

Tab. 6.6d: Details for the calculation of (T_{Lab} – T(TPV)) for loop D and its uncertainty.

Loop D

Participant	Thermometer	W	$U(W)$	W^*	$U(W^*)$	TPV	U(TPV) / mK	T_{Lab}- T(TPV)	$U(T_{\mathrm{Lab}}$-T(TPV))	T_{Lab}- T(TPV)	U (average)
			k = 2		k = 2		k = 2	/ mK	k = 2	/mK, average	/ mK, k = 2
PTB	Th 1041			4,2858584	7,60	4,2863397	10,28				
	Th 1043	4,2864869	5,76			4,2864537	12,36	11,69	13,60		
MIKES	Th 1041			4,2863549	10,32	4,2863397	10,28	5,35	14,57	2,57	10,69
	Th 1043	4,2864518	9,77			4,2864537	12,36	-0,67	15,72		
EIM	Th 1041			4,2863552	9,82	4,2863397	10,28	5,46	14,22	6,31	10,22
	Th 1043	4,2864742	8,02			4,2864537	12,36	7,22	14,70		
INRiM	Th 1041			4,2863307	5,34	4,2863397	10,28	-3,17	11,58	-0,62	8,57
	Th 1043	4,2864607	3,18			4,2864537	12,36	2,46	12,72		
IPQ	Th 1041			4,2863346	10,20	4,2863397	10,28	-1,80	14,48	2,08	10,70
	Th 1043	4,2864728	10,14			4,2864537	12,36	6,72	15,87		
CEM	Th 1041			4,2863336	15,53	4,2863397	10,28	-2,15	18,62	0,67	13,62
	Th 1043	4,2864648	15,70			4,2864537	12,36	3,91	19,96		
VSL	Th 1041			4,2863451	5,78	4,2863397	10,28	1,90	11,79	-0,70	8,67
	Th 1043	4,2864430	3,41			4,2864537	12,36	-3,77	12,78		
PTB	Th 1041			4,2863057	26,70	4,2863397	10,28				
	Th 1043	4,2863748	10,97			4,2864537	12,36				

6.3.4 Treatment of PTB measurements

The measurements of PTB need a special treatment, because PTB is the only NMI with measurements in all loops and the PTB measurements are used to calculate the Temperature PTB value (TPV). Because also the measurements of the sub-pilots are considered for the calculation of the TPV, the TPV is not identical with the PTB reference for all loops. Tab. 6.6e the data for all thermometers for which the data from PTB and at one other NMI were used to calculate TPV. The value for (*T*(PTB) – TPV) is calculated as the average of all thermometers.

Tab. 6.6e: Calculation of T_{PTB} – *T*(TPV)

Thermometer	(*T*(PTB) – TPV) / mK	*U* /mK (*k* = 2)
TH 1068 (average)	-6,88	
TH 93103	-1,76	
TH1065	-4,72	
TH 944RS13	4,29	
Average	-2,27	9,77

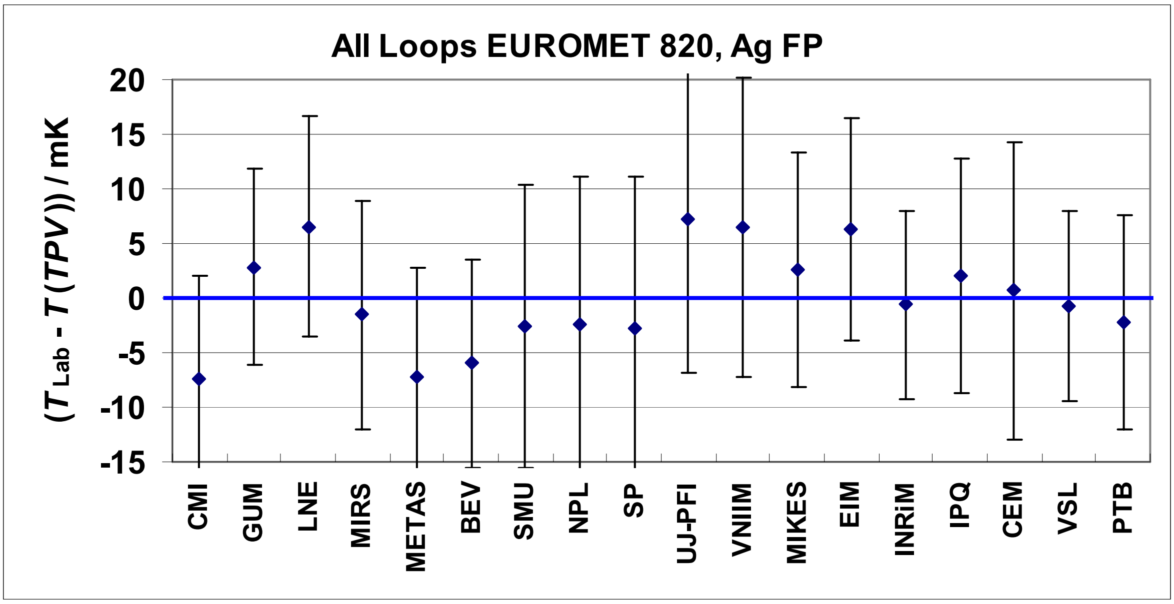
The uncertainty of (*T*(PTB) – TPV) is estimated from the uncertainty of the PTB measurements for all thermometers. The uncertainty is estimated as the average of all uncertainties (11 values) given for the PTB measurements in Tab. 6.3a to 6.3f, except the last value of HTSPRT 944RS13.

6.4 Linking of the loops for the Ag freezing point

The procedure for linking the loops together is similar to the procedure used for the measurements at the Al freezing point: all measurement will be given as difference to the Temperature PTB Value (*TPV*).

The summary of differences between TPV and the participants is presented in Fig. 6.7a and Tab. 6.7c.

Fig. 6.7a: All loops relative to PTB



6.5 EUROMET Reference Value (*ERV*) for the Ag freezing point

The procedure for the determination of the *ERV*(Ag) is the same as for the determination of the *ERV*(Al). Three possible values for the *ERV* are shown in Tab. 6.7b. The method for is the same as in the case of the Al freezing point.

Tab 6.7b: Possible *ERV*s and their uncertainty *U* (*k* = 2)

ERV – *T*(*TPV*) / mK		
	Value / mK	*U* (*k* = 2) / mK
Mean	-0,52	2,04 (1,30)
Weighted mean	-2,50	1,84
Median	-0,66	2,12

There is no clear preference for the choice of the *ERV*. In such a case typically the mean value is chosen. Therefore:

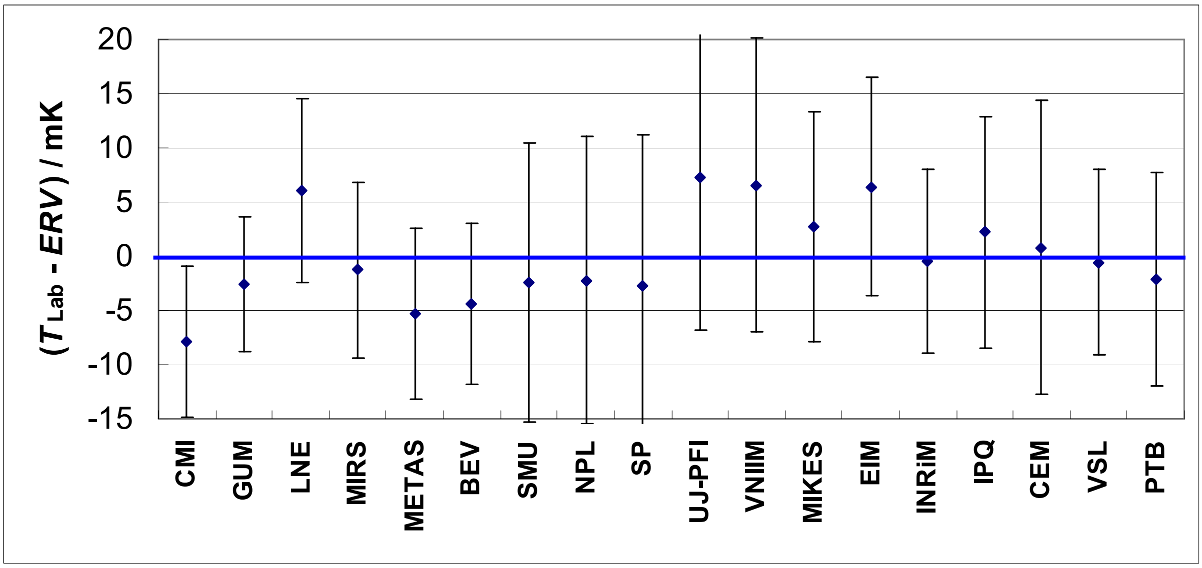
ERV(Ag, 820) = *T*(*TPV*) - (0,52 ± 1,30) mK (uncertainty for *k* = 2)

The resulting deviation of the laboratories from the *ERV* is given in Tab. 6.7c, additionally to the deviation from TPV. The results of the participants relative to the ERV are shown in Fig. 6.7b.

Tab. 6.7c: Summary of the results of EUROMET 820 for the freezing point of Ag

Participant	T_{Lab} - T(TPV)	*U* / mK	**(T_{Lab} - *ERV*) / mK**	***U* / mK**
CMI	-7,99	7,03	**-7,87**	**6,91**
GUM	-2,72	6,34	**-2,60**	**6,21**
LNE	5,90	8,57	**6,02**	**8,47**
MIRS	-1,35	8,20	**-1,23**	**8,10**
METAS	-5,45	7,97	**-5,33**	**7,86**
BEV	-4,48	7,57	**-4,36**	**7,46**
SMU	-2,55	12,97	**-2,43**	**12,90**
NPL	-2,34	13,37	**-2,22**	**13,31**
SP	-2,80	14,00	**-2,68**	**13,94**
UJ-PFI	7,17	14,11	**7,29**	**14,05**
VNIIM	6,47	13,64	**6,59**	**13,58**
MIKES	2,57	10,69	**2,69**	**10,61**
EIM	6,31	10,22	**6,43**	**10,14**
INRiM	-0,62	8,57	**-0,50**	**8,47**
IPQ	2,08	10,70	**2,20**	**10,62**
CEM	0,67	13,62	**0,79**	**13,56**
VSL	-0,70	8,67	**-0,58**	**8,57**
PTB	-2,27	9,95	**-2,15**	**9,86**

Fig. 6.7b: All participants relative to the *ERV* for Ag



6.5.1 Birge ratio test

The Birge ratio test was applied as described in chapter 5.3.1.

The data presented in Tab. 6.7c lead to a Birge ratio of $R_B = 1,63$.

Taking into account the severe problems with the instability of the HTSPRTs it is not surprising that the Birge ratio test indicates some problems with the consistency of the corrected data. Nevertheless, without an alternative the quality of the data will be considered to be sufficient for the report to the CCT.

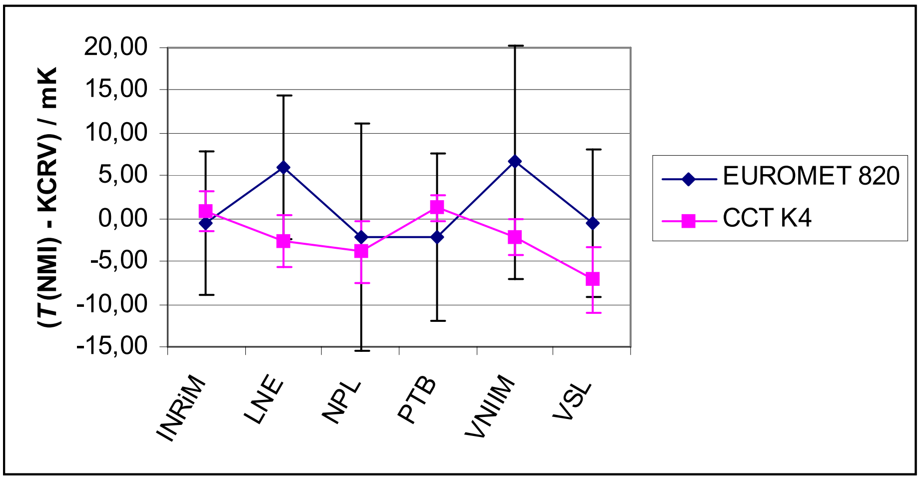
6.6 Linkage between EUROMET 820 and CCT K4

The linkage between EUROMET 820 and CCT K4 for the freezing point of Ag follows the same procedure as for the Al freezing point. Tab. 6.8 and Fig. 6.8 give the data of those NMIs that participated in EUROMET 820 and in CCT K4. The average is taken as the simple mean, and the uncertainty is the standard deviation of the mean, multiplied with *k* = 2.

Tab. 6.8: Ag freezing point: T_{Lab} – *KCRV* in mK for different interlaboratory comparisons. Uncertainties are given for *k* = 2.

(*T*(NMI) – *KCRV*) / mK for the Ag freezing point		
Laboratory	EUROMET 820	CCT K4
INRiM	-0,50± 8,47	0,85 ± 2,36
LNE-INM	6,02 ± 8,47	-2,69 ± 3,06
NPL	-2,22 ± 13,31	-3,89 ± 3,60
PTB	-2,15 ± 9,86	1,27 ± 1,46
VNIIM	6,59 ± 13,58	-2,16 ± 2,20
VSL	-0,58 ± 8,57	-7,12 ± 3,88
Mean	1,19 ± 3,29	-2,29 ± 2,55

Fig. 6.8 Results for the freezing points of Ag for participants in EUROMET 820 and CCT K4.



From the last line in Tab. 6.8 it follows:

$$\boldsymbol{ERV}\,\mathbf{(Ag, 820) =}\ \boldsymbol{KCRV}\,\mathbf{(CCT\ K4) - (3{,}48 \pm 4{,}16)\ mK}\ (\boldsymbol{k}\ \mathbf{= 2)}$$

The uncertainty of the correction is the sum in quadrature of the average values.

7. Bilateral Equivalence

The bilateral degrees of equivalence between laboratories *i* and *j* are expressed by the deviation of their results:

$$D_{ij} = T_i - T_j$$

and the related uncertainty

$$U_{ij} = 2u_{ij} = 2\sqrt{\mathrm{u}_{\mathrm{i}}^2 + u_j^2}$$

The data T_i and u_i are taken from Tab. 5.4 and Tab. 6.7c; for the uncertainty the uncertainty of the deviation from the *ERV* is taken. Note that the degrees of equivalence is independent of the choice of the *ERV* and its uncertainty depends only very little on the *ERV*. The degree of equivalence are given in Tab 7a for Al and in Tab. 7b for Ag.

In Tab. 7a and 7b below the diagonal the quantified degree of equivalence, $\mathrm{QDE}_{0.95}$, is shown, which is a one-parameter description of equivalence [8]. It is calculated as

$$QDE_{0.95}(i,j) = \left|D_{ij}\right| + \left\{1.645 + 0.3295 * \exp\left[-4.05\left|D_{ij}\right| / u_{ij}\right]\right\} u_{ij} \text{ ,}$$

with D_{ij} and u_{ij} as defined above.

The degree of equivalence of the participants relative to the ERV is also given in Tab. 7a and 7b . The data for (T_i – *ERV*) and its uncertainty are taken from Tab. 5.4 and 6.7b.

Tab. 7a: Degree of equivalence between the participating NMIs in EUROMET.T-K4 for the freezing point of Aluminum.
Upper right part of matrix: degrees of equivalence, expressed in mK
Lower left part of matrix: quantified demonstrated equivalence at the 95% level ($QDE_{0.95}$), expressed in mK

lab $_i$ ↓ lab $_j$ ⟶

	DTI		**JV**		**OMH**		**SMU**		**INM**		**UME**		**FSB**		**CMI**		**GUM**	
	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK
DTI			-0,03	7,18	4,15	6,78	-1,97	6,56	5,18	6,62	0,63	6,97	3,81	13,74	-0,15	7,06	-3,03	6,69
JV	7,08				4,18	6,00	-1,94	5,75	5,21	5,82	0,66	6,21	3,84	13,37	-0,12	6,31	-3,00	5,68
OMH	9,74		9,12				-6,12	5,24	1,03	5,31	-3,52	5,74	-0,34	13,16	-4,30	5,85	-7,18	5,16
SMU	7,46		6,73		10,43				7,15	5,03	2,60	5,47	5,78	13,05	1,82	5,59	-1,06	4,87
INM	10,63		9,99		5,58		11,29				-4,55	5,55	-1,37	13,08	-5,33	5,66	-8,21	5,19
UME	6,91		6,20		8,25		7,12		9,11				3,18	13,26	-0,78	6,06	-3,66	5,62
FSB	15,35		15,05		12,92		16,57		13,05		14,40				-3,96	13,30	-6,84	13,11
CMI	6,93		6,20		9,11		6,48		9,99		6,12		15,10				-2,88	5,52
GUM	8,56		7,68		11,42		5,20		12,48		8,29		17,66		7,43			
LNE-INM	8,73		8,07		11,49		6,00		12,21		8,40		18,08		7,42		5,70	
MIRS	7,26		6,57		6,72		7,78		7,56		5,78		13,39		6,55		8,98	
METAS	10,00		9,26		12,99		6,62		13,83		9,68		19,29		9,00		5,73	
BEV	7,74		7,05		10,75		5,36		11,62		7,44		20,46		6,81		5,92	
NPL	6,09		5,23		8,57		5,05		9,40		5,38		15,06		5,03		6,22	
SP	7,50		6,76		10,47		4,72		11,32		7,16		16,67		6,51		5,19	
UJ-PFI	7,89		7,20		10,93		5,30		11,80		7,61		16,87		6,97		5,72	
VNIIM	7,42		6,64		10,34		4,34		11,17		7,04		16,73		6,38		4,69	
MIKES	6,26		6,47		8,13		7,52		8,99		6,00		14,25		6,42		8,69	
EIM	20,03		19,53		15,04		20,98		13,88		18,70		20,99		19,55		22,14	
INRiM	7,80		7,17		7,33		8,42		8,17		6,43		13,67		7,15		9,60	
IPQ	6,90		6,17		8,53		6,94		9,40		5,90		14,62		6,06		8,10	
CEM	9,12		8,56		10,98		8,97		11,89		8,39		16,03		8,44		10,00	
VSL	7,25		6,54		10,03		5,92		10,91		6,81		15,89		6,34		6,92	
PTB	6,88		6,11		9,74		4,74		10,59		6,46		16,01		5,87		5,68	
ERV	5,45		4,48		7,63		4,59		8,42		4,49		14,57		4,28		5,83	

Tab. 7a (continued): Degree of equivalence between the participating NMIs in EUROMET.T-K4 for the freezing point of Aluminum.
Upper right part of matrix: degrees of equivalence, expressed in mK
Lower left part of matrix: quantified demonstrated equivalence at the 95% level ($QDE_{0.95}$), expressed in mK

lab $_i$ ↓ lab $_j$ →

	LNE-INM		**MIRS**		**METAS**		**BEV**		**NPL**		**SP**		**UJ-PFI**		**VNIIM**		**MIKES**	
	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK
DTI	-3,37	6,50	1,75	6,55	-4,87	6,24	-1,90	6,95	-0,56	6,13	-2,12	6,45	-2,12	6,91	-2,31	6,15	0,89	7,10
JV	-3,34	5,74	1,78	5,74	-4,84	5,38	-1,87	6,19	-0,53	5,25	-2,09	5,62	-2,09	6,14	-2,28	5,27	0,92	6,36
OMH	-7,52	4,83	-2,40	5,23	-9,02	4,83	-6,05	5,72	-4,71	4,69	-6,27	5,10	-6,27	5,67	-6,46	4,72	-3,26	5,90
SMU	-1,40	5,45	3,72	4,94	-2,90	4,51	0,07	5,45	1,41	4,37	-0,15	4,80	-0,15	5,40	-0,34	4,39	2,86	5,65
INM	-8,55	4,45	-3,43	5,01	-10,05	4,60	-7,08	5,52	-5,74	4,45	-7,30	4,88	-7,30	5,47	-7,49	4,48	-4,29	5,71
UME	-4,00	5,34	1,12	5,46	-5,50	5,08	-2,53	5,93	-1,19	4,95	-2,75	5,34	-2,75	5,88	-2,94	4,97	0,26	6,11
FSB	-7,18	13,22	-2,06	13,04	-8,68	12,89	9,56	13,25	-4,37	12,84	-5,93	12,99	-5,93	13,22	-6,12	12,85	-2,92	13,33
CMI	-3,22	5,10	1,90	5,58	-4,72	5,21	-1,75	6,04	-0,41	5,08	-1,97	5,46	-1,97	5,99	-2,16	5,10	1,04	6,22
GUM	-0,34	5,79	4,78	5,10	-1,84	4,69	1,13	5,60	2,47	4,55	0,91	4,97	0,91	5,55	0,72	4,58	3,92	5,79
LNE-INM			5,12	4,85	-1,50	12,97	2,39	5,38	2,81	4,27	1,25	4,72	1,25	5,32	1,06	4,29	4,26	5,57
MIRS	9,11				-6,62	4,50	-3,65	5,44	-2,31	4,35	-3,87	4,79	-3,87	5,39	-4,06	4,37	-0,86	5,63
METAS	13,00		10,32				2,97	5,06	4,31	3,86	2,75	4,35	2,75	5,00	2,56	3,89	5,76	5,27
BEV	6,84		8,13		7,14				1,34	4,93	-0,22	5,32	1,63	5,86	-0,41	4,95	2,79	6,09
NPL	6,33		5,90		7,49		5,48				-1,56	4,20	0,29	4,87	-1,75	3,72	1,45	5,14
SP	5,22		7,81		6,33		5,22		5,05				1,85	5,26	-0,19	4,22	3,01	5,52
UJ-PFI	5,76		8,30		6,87		6,55		4,79		6,23				-0,19	4,89	3,01	6,04
VNIIM	4,69		7,66		5,76		4,90		4,82		4,15		4,80				3,20	5,16
MIKES	8,85		5,76		10,09		7,82		5,77		7,56		8,00		7,45			
EIM	22,33		17,25		23,63		21,22		19,24		21,05		21,40		21,00		18,55	
INRiM	9,76		5,61		11,01		8,72		6,62		8,47		8,90		8,37		6,40	
IPQ	6,85		6,03		9,50		7,26		5,26		6,98		7,43		6,86		6,10	
CEM	10,19		0,24		11,49		9,19		7,77		9,02		9,34		8,97		8,58	
VSL	7,06		7,43		7,09		5,15		5,15		5,91		6,38		5,74		7,16	
PTB	5,77		7,09		6,94		0,86		4,40		4,67		5,25		4,40		6,86	
ERV	5,88		4,89		6,94		5,10		2,61		4,56		5,25		4,22		4,89	

Tab. 7a (continued): Degree of equivalence between the participating NMIs in EUROMET.T-K4 for the freezing point of Aluminum.
Upper right part of matrix: degrees of equivalence, expressed in mK
Lower left part of matrix: quantified demonstrated equivalence at the 95% level ($QDE_{0.95}$), expressed in mK

lab$_i$ ↓ lab$_j$ ⟶

	EIM		INRiM		IPQ		CEM		VSL		PTB		*ERV*	
	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK
DTI	13,37	8,10	1,76	7,15	0,39	7,01	0,08	9,26	-0,98	7,15	-1,45	6,40	-0,27	5,55
JV	13,40	7,46	1,79	6,41	0,42	6,26	0,11	8,70	-0,95	6,41	-1,42	5,57	-0,24	4,56
OMH	9,22	7,07	-2,39	5,95	-3,76	5,80	-4,07	8,37	-5,13	5,96	-5,60	5,04	-4,42	3,90
SMU	15,34	6,86	3,73	5,70	2,36	5,54	2,05	8,20	0,99	5,71	0,52	4,74	1,7	3,50
INM	8,19	6,92	-3,42	5,77	-4,79	5,61	-5,10	8,24	-6,16	5,78	-6,63	4,82	-5,45	3,61
UME	12,74	7,25	1,13	6,16	-0,24	6,01	-0,55	8,52	-1,61	6,17	-2,08	5,28	-0,9	4,21
FSB	9,56	13,89	-2,05	13,35	-3,42	13,28	-3,73	14,59	-4,79	13,35	-5,26	12,97	-4,08	12,57
CMI	13,52	7,34	1,91	6,27	0,54	6,12	0,23	8,60	-0,83	6,27	-1,30	5,40	-0,12	4,36
GUM	16,40	6,98	4,79	5,84	3,42	5,68	3,11	8,30	2,05	5,85	1,58	4,91	2,76	3,73
LNE-INM	16,74	6,80	5,13	5,63	3,76	5,46	3,45	8,14	2,39	5,64	1,92	4,65	3,1	3,38
MIRS	11,62	6,85	0,01	5,69	-1,36	5,52	-1,67	8,19	-2,73	5,70	-3,20	4,72	-2,02	3,48
METAS	18,24	6,55	6,63	5,33	5,26	5,15	4,95	7,94	3,89	5,34	3,42	4,28	4,6	2,85
BEV	15,27	7,23	3,66	6,14	2,29	5,99	1,98	8,51	0,92	4,93	0,45	5,26	1,63	4,18
NPL	13,93	6,45	2,32	5,20	0,95	5,02	0,64	7,86	-0,42	5,21	-0,89	4,12	0,29	2,61
SP	15,49	6,76	3,88	5,57	2,51	5,41	2,20	8,11	1,14	5,58	0,67	4,58	1,85	3,29
UJ-PFI	15,49	7,19	3,88	6,09	2,51	5,94	2,20	8,47	1,14	6,10	0,67	5,20	1,85	4,11
VNIIM	15,68	6,47	4,07	5,22	2,70	5,04	2,39	7,87	1,33	5,23	0,86	4,15	2,04	2,65
MIKES	12,48	7,38	0,87	6,31	-0,50	6,17	-0,81	8,63	-1,87	6,32	-2,34	5,46	-1,16	4,43
EIM			-11,61	7,42	-12,98	7,29	-13,64	9,47	-14,35	7,43	-14,82	5,90	-13,64	5,90
INRiM	17,71				-1,37	6,22	-1,68	8,67	-2,74	6,37	-3,21	5,52	-2,03	4,50
IPQ	18,98		6,66				-0,31	8,56	-1,37	6,22	-1,84	5,35	-0,66	4,29
CEM	21,43		9,11		8,40				-1,06	8,67	-1,53	8,07	-0,35	7,41
VSL	20,46		8,01		6,66		8,73				-0,47	5,52	0,71	4,51
PTB	19,67		7,76		6,29		8,45		5,47				1,18	3,19
ERV	18,49		5,75		4,39		7,28		4,63		3,83			

Tab. 7b: Degree of equivalence between the participating NMIs in EUROMET.T-K4 for the freezing point of Silver.
Upper right part of matrix: degrees of equivalence, expressed in mK
Lower left part of matrix: quantified demonstrated equivalence at the 95% level (*$QDE_{0.95}$*), expressed in mK

lab $_i$ lab $_j$ →

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	CMI		GUM		LNE-INM		MIRS		METAS		BEV		SMU		NPL		SP	
	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK
CMI			-5,27	9,29	-13,89	10,93	-6,64	10,65	-2,54	10,47	-3,51	10,17	-5,44	14,63	-5,65	15,00	-5,19	15,56
GUM	12,93				-8,62	10,50	-1,37	10,21	2,73	10,02	1,76	9,71	-0,17	14,32	-0,38	14,69	0,08	15,36
LNE-INM	22,88		17,26				7,25	11,72	11,35	11,56	10,38	11,29	8,45	15,43	8,24	15,78	8,70	16,41
MIRS	15,41		10,33		16,90				4,10	11,29	3,13	11,01	1,20	15,23	0,99	15,58	1,45	16,22
METAS	11,39		11,15		20,85		13,48				-0,97	10,84	-2,90	15,11	-3,11	15,46	-2,65	16,00
BEV	11,98		10,11		19,66		12,37		10,75				-1,93	14,90	-2,14	15,26	-1,68	15,81
SMU	17,60		14,09		21,17		15,05		15,85		15,05				-0,21	18,54	0,25	18,99
NPL	18,10		14,42		21,25		15,34		16,32		15,50		18,24				0,46	19,35
SP	18,16		15,14		22,23		16,08		16,50		15,79		18,68		19,01			
UJ-PFI	28,04		22,18		13,48		19,20		22,12		22,33		25,16		22,55		24,04	
VNIIM	26,99		21,49		15,71		20,87		24,83		23,70		24,49		24,52		25,34	
MIKES	20,98		15,46		14,80		15,10		18,90		17,74		19,09		19,18		20,02	
EIM	24,39		18,81		12,97		18,35		22,31		21,15		16,74		22,45		23,33	
INRiM	16,37		11,08		16,40		11,54		14,40		13,26		15,55		15,77		16,51	
IPQ	20,49		15,00		15,22		14,69		18,42		17,27		18,67		18,77		19,60	
CEM	21,20		16,05		18,57		15,93		19,12		18,05		19,38		19,51		20,22	
VSL	16,35		11,10		16,53		11,59		14,38		13,25		15,56		15,79		16,51	
PTB	15,67		11,44		18,87		12,59		13,82		12,86		15,96		16,33		16,76	
ERV	13,55		7,74		12,99		8,28		11,80		10,51		13,50		13,74		14,63	

Tab. 7b (continued): Degree of equivalence between the participating NMIs in EUROMET.T-K4 for the freezing point of Silver.
Upper right part of matrix: degrees of equivalence, expressed in mK
Lower left part of matrix: quantified demonstrated equivalence at the 95% level ($QDE_{0.95}$), expressed in mK

lab $_i$ ↓ lab $_j$ ⟶

	UJ-PFI		VNIIM		MIKES		EIM		INRiM		IPQ		CEM		VSL		PTB		ERV	
	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK	D_{ij}/mK	U_{ij}/mK
CMI	-15,16	15,66	-14,46	15,24	-10,56	12,66	-14,30	12,27	-7,37	10,93	-10,07	12,67	-8,66	15,22	-7,29	11,01	-5,72	12,04	-7,87	6,91
GUM	-9,89	14,93	-9,19	14,93	-5,29	12,29	-9,03	11,89	-2,10	10,50	-4,80	12,30	-3,39	14,91	-2,02	10,58	-0,45	11,65	-2,60	6,21
LNE-INM	-1,27	13,58	-0,57	16,00	3,33	13,58	-0,41	13,21	6,52	11,98	3,82	13,58	5,23	15,99	6,60	12,05	8,17	13,00	6,02	8,47
MIRS	-8,52	12,98	-7,82	15,81	-3,92	13,35	-7,66	12,98	-0,73	11,72	-3,43	13,36	-2,02	15,80	-0,65	11,79	0,92	12,76	-1,23	8,10
METAS	-12,62	11,56	-11,92	15,69	-8,02	13,20	-11,76	12,83	-4,83	11,56	-7,53	13,21	-6,12	15,67	-4,75	11,63	-3,18	12,61	-5,33	7,86
BEV	-11,65	12,98	-10,95	15,49	-7,05	12,97	-10,79	12,59	-3,86	11,29	-6,56	12,98	-5,15	15,48	-3,78	11,36	-2,21	12,36	-4,36	7,46
SMU	-9,72	18,72	-9,02	18,73	-5,12	16,70	-2,43	16,41	-1,93	15,43	-4,63	16,71	-3,22	18,72	-1,85	15,49	-0,28	16,24	-2,43	12,90
NPL	-9,51	15,83	-8,81	19,02	-4,91	17,02	-8,65	16,73	-1,72	15,78	-4,42	17,03	-3,01	19,00	-1,64	15,83	-0,07	16,56	-2,22	13,31
SP	-9,97	17,07	-9,27	19,46	-5,37	17,52	-9,11	17,24	-2,18	16,31	-4,88	17,52	-3,47	19,45	-2,10	16,36	-0,53	17,07	-2,68	13,94
UJ-PFI			0,70	19,54	4,60	27,95	7,29	17,33	7,79	16,41	5,09	17,61	6,50	19,53	7,87	16,46	9,44	17,16	7,29	14,05
VNIIM	19,18				3,90	17,23	0,16	16,95	7,09	16,00	4,39	17,24	5,80	19,19	7,17	16,06	8,74	16,78	6,59	13,58
MIKES	28,80		18,53				-3,74	14,68	3,19	13,58	0,49	15,01	1,90	17,22	3,27	13,64	4,84	14,48	2,69	10,61
EIM	21,64		16,69		16,12				6,93	13,21	4,23	14,68	6,43	16,93	7,01	13,28	8,58	14,14	6,43	10,14
INRiM	21,34		20,33		14,69		17,83				-2,70	13,58	-0,50	15,99	0,08	12,05	1,65	13,00	-0,50	8,47
IPQ	19,86		18,93		14,74		16,54		14,32				2,20	17,22	2,78	13,65	4,35	14,49	2,20	10,62
CEM	22,78		21,86		17,22		20,49		15,69		17,37				1,37	16,04	2,94	16,77	0,79	13,56
VSL	21,46		20,45		14,81		17,96		11,87		14,44		15,89				1,57	13,06	-0,58	8,57
PTB	23,59		22,58		16,91		20,23		13,11		16,48		17,40		13,13				-2,15	9,86
ERV	18,88		17,80		11,64		14,78		8,33		11,26		13,34		8,44		10,54			

8. Conclusion

The results for the measurements at the freezing point of Al are more or less satisfactory, while the measurements at the freezing point of Ag suffer from severe instabilities of the HTSPRTs. By using two HTSPRTs in each loop and an additional comparison with a travelling Ag freezing point cell results were achieved which can be used partly for scrutinizing of the CMC entries delivered by the participants. Nevertheless, the uncertainties are still higher than expected before the start of the comparison.

The following recommendations and questions may be concluded:

- All laboratories should include in their quality manual a cleaning procedure for the thermometers, fixed point cell and annealing furnaces in order to minimise the poisoning of the thermometers.
- In a future comparison at least the *W*-values of Ga and Al should also be reported. This will allow a better understanding of the history of the thermometers.
- The results with a re-sealable travelling cell were much better than the results with the HTSPRTs. It seems that the risk of damage for such a cell is acceptable. It should be considered to perform further comparisons at the freezing point of Ag with such a cell.
- HTSPRTs are used in NMIs for the interpolation of the temperature scale, but not in industry. It should be discussed if HTSPRTs are suitable as travelling instruments at all. Au/Pt thermocouples may at the end allow measurements with smaller uncertainties than HTSPRTs. Moreover, some statistical information about the real number of calibrations of HTSPRTs may be gathered to discuss if such an enormous effort as EUROMET 820 is justified.

The evaluation of the data that is suggested in this report for the measurements at the Ag freezing point is somewhat arbitrary. It is quite unlikely that another evaluation of the data would result in the same difference between the results of the participants and the *ERV*. On the other hand all data handling was clearly described and each “manipulation” (drift compensation, elimination of outliers) can be discussed and probably also be justified. Nevertheless, the large number of “manipulations” may be thought as a kind of data cosmetics. But one should have in mind that the estimation of the measurement of the uncertainties is quite conservative. All “manipulations” are thought to be within the uncertainties.

9. References

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Appendix A: Technical Protocol

PTB (Physikalisch-Technische Bundesanstalt)
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Germany

Thermometry

Agreed EUROMET Project N° 820
EUROMET-T-K4

Comparison of the realisations of the ITS-90 at the freezing points of Al (660,323 °C) and Ag (961,78 °C)

“Technical protocol”

(version 2, 2005-01-12)

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1. Introduction

The EUROMET regional key comparison was initiated during the EUROMET TC THERM meeting on 30./31. March 2004. PTB was chosen to be the pilot laboratory of EUROMET-T-K4. The procedures and instructions, which are given below, should be followed by all participants. Each laboratory should follow its common practice in realizing the aluminum and silver freezing points. The instructions are based on the Protocols given in the Guidelines for CIPM key comparisons, Appendix F to the MRA, 1 March 1999.

The procedure of EUROMET-T-K4 is not identical with the procedure of CCT-K4: While CCT-K4 was a direct comparison of Al and Ag fixed point cells, EUROMET-T-K4 requires the calibration of an SPRT (25 Ω) at the freezing point of Al and 2 HTSPRTs at the freezing point of Ag and by each participant. Participants only interested in a comparison at the Al freezing point will only calibrate the SPRT (25 Ω). The procedure follows partly the procedures used in APMP-T-K4.

During the preliminary characterization of the HTSPRTs by the pilot laboratory it was found that the thermometers are not as stable as desirable. Several improvements of the protocol have been considered and mostly rejected, because the necessary effort would have been too large. As a compromise, now 1 additional Ag fixed point cell (provided by NMi-VSL) will be compared only among the pilot and sub-pilot laboratories. This will allow for a better control of the stability of the thermometers. Moreover, the linkage of the loops can be based on more reliable measurements.

The comparison will involve the 5 EUROMET NMIs previously involved in CCT-T-K4 [BNM-INM (FR), INRiM(IT), NMi-VSL (NL), NPL (GB), PTB (DE)] as pilot or sub-pilots and additional nearly all European national laboratories. The comparison will be divided in 4 loops. Besides PTB there will be another participant of CCT-T-K4 in each of the loops of EUROMET-T-K4.

The pilot will calibrate a total of 9 HTSPRTs, and 5 SPRT, including 1 spare thermometers of each type. The thermometers were generously provided by Hart Scientific (3 HTSPRTs and 4 SPRTs), BIPM (3 HTSPRTs), MIKES (2 HTSPRTs), Metas (1 HTSPRT) and PTB (1 SPRT).

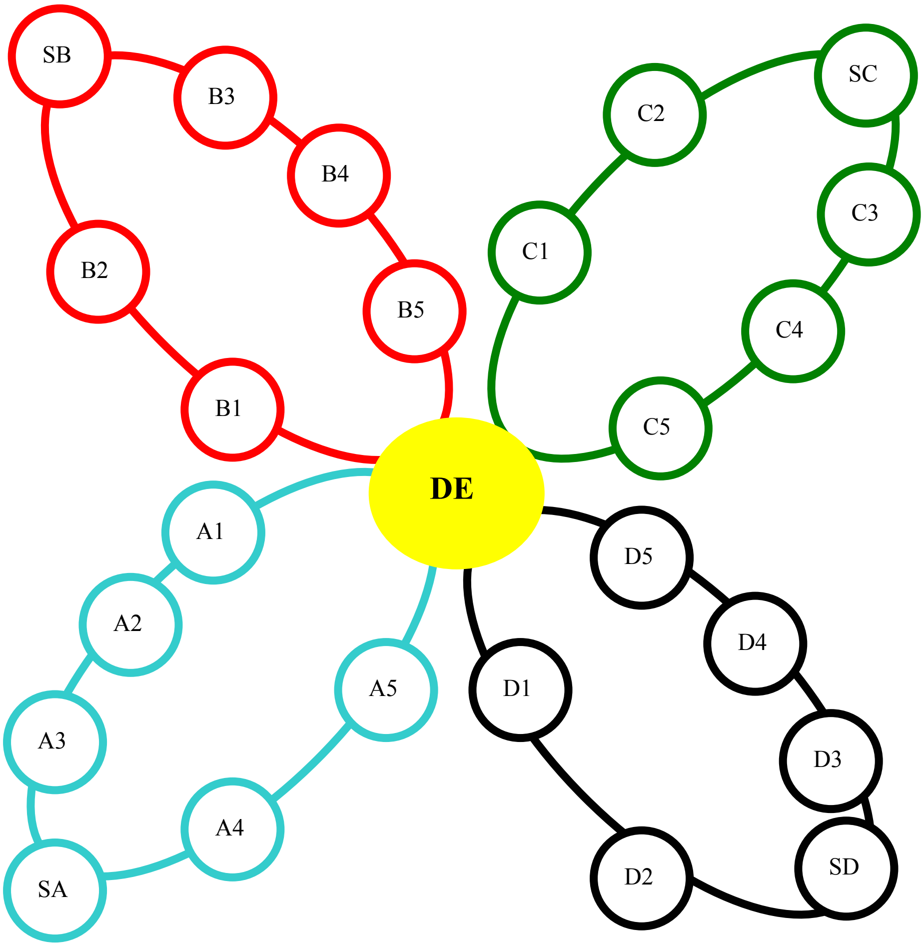
The thermometers are very fragile and must be handled with extreme care. When not in use, they should be stored in a safe place in the groove of the protecting foam. The SPRTs and the Ag fixed point cell will be hand carried from laboratory to laboratory. Each laboratory is responsible for carriage of the devices to the following laboratory in the list. The procedures required by the Department of Customs of various countries must be strictly obeyed when the thermometers are shipped outside EU. In these cases, the Carnet forms must be carefully and accurately completed. It is the responsibility of the laboratory carrying the transfer SPRT to the next laboratory to present the Carnet to Customs when leaving the country and upon arrival in the country of destination. Personnel at the receiving laboratory must check the Carnet forms very carefully upon receipt. It is the duty of the pilot laboratory to find out a solution between the different participants (in this loop) for taking in charge the ATA carnet fees.

If the thermometers have not been received in due time the pilot must be immediately informed. As a result the delayed laboratory may be excluded from the comparison, or the timetable may be revised accordingly.

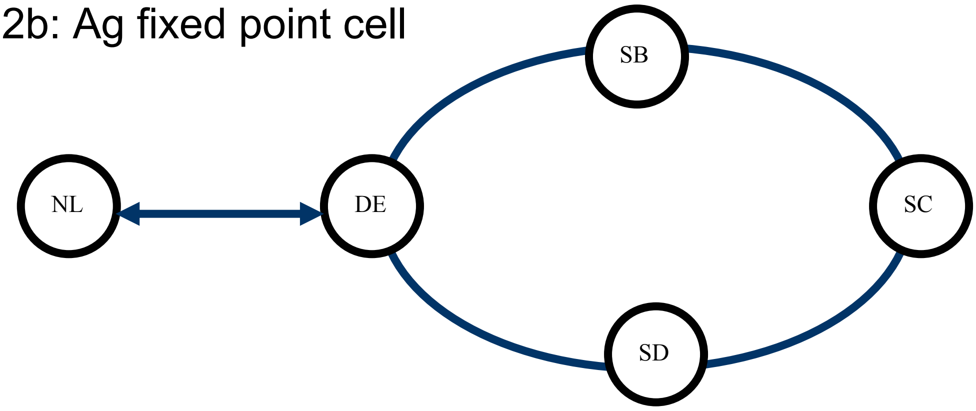
Any participant will calibrate at least one thermometer. It will establish a calibration report. This report will be sent (within 1 month) to the pilot laboratory.

2. Scheme of Organization

2a: Thermometers



2b: Ag fixed point cell



3. Provisional schedule

For a given laboratory, the time allowed for the measurements (calibration of 1 SPRT at the Al freezing point and 2 HTSPRTs at the freezing point of Ag) is estimated to be less than 4 weeks. The travelling time between two Labs could be rated at 2 weeks. In agreement with these estimations the schedule will in principle follow tables 1a and 1b.

Start of measurements	Loop A (only Pt-25)	Loop B	Loop C	Loop D
1. July 2004	DE	DE		
1. Dec. 2004	DK	CZ	DE	
1. Feb. 2005	NO	PL	AT	DE
1. Apr. 2005 [1]	HU	FR (SB) + Ag cell	SK	FI
15. May 2005	SK (SA)	CH	UK (SC) + Ag cell	GR
1. July 2005	RO	SI	SE	IT (SD) + Ag cell
1. Sep. 2005	HR	TR	LT	PT
15. Oct. 2005	DE	DE	RU	ES
1. Dec. 2005			DE	NL + Ag cell
1. Feb. 2006				DE
Draft A prepared by PTB: April 2006				

Table 1a: Provisional schedule for Thermometers in EUROMET-T-K4

The Ag fixed point cell will only be used by the pilot and sub-pilot laboratories to control the stability of the HTSPRTs. The schedule was organised in such a way that thermometers and fixed point cell should be at the same time in the laboratories of the sub-pilots, but arriving from and going to different laboratories.

Start of measurement	NMI
1. February 2005	NL
1. March 2005	DE
1. April 2005	FR (SB)
15. May 2005	UK (SC)
1. July	IT (SC)
1. September	DE
1. November	NL

Table 1b: Provisional schedule for Ag fixed point cell in EUROMET-T-K4

4. Participating Laboratories
see Chapter 3 of the report on EUROMET 820

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[1] EUROMET meeting in Vienna on 6./7. April 2005

5. Requirements to be fulfilled by participating laboratories

0. Laboratories that will only participate for the comparison of the Al fixed point, only have to do the measurements for this point. The instructions are to be modified by the laboratory accordingly.
1. All participating laboratory must have an aluminum freezing-point cell and a silver freezing-point cell whose thermometer wells have inner diameter larger than 8 mm. The freezing-point cells have to be long enough to achieve a reasonable hydrostatic head.
2. All participating laboratory should prepare one and to be safer side more than one monitoring HTSPRT and one monitoring SPRT, which will be used in the realization of the freezing points of aluminum and silver.
3. Each participating laboratory should have experience with calibration of PRTs at the freezing points of Al and Ag. The procedure shall be described in the quality manual of the laboratory, including a detailed uncertainty analysis.
4. The reproducibility of the calibration at the freezing points of Al and Ag should be demonstrated in a period close the measurements of EUROMET K4, i.e. within 3 months before the EUROMET project. For this purpose the monitor thermometers should be calibrated at least 4 times at the fixed points, and the standard deviation of the results should be in agreement with the expected uncertainty.

6. Task of the pilot and sub-pilot laboratories

The pilot laboratory prepares for each loop

- 2 pieces of HTPRT as transfer thermometer whose nominal resistance at the triple point of water is < 2,5Ω, in most cases appr. 0,25 Ω
- 1 piece of SPRT as transfer thermometer whose nominal resistance at the triple point of water appr. 25 Ω
- 1 Ag fixed point cell

The PRTs should be stable during the realization of the freezing points of aluminum or silver and the suggested short-time stability before and after the freezing-point realization, approximately within 1.0 mK at the TPW.
The pilot laboratory will in the beginning and at the end of each loop follow the same procedures as all participating laboratories, which are described in the detailed instructions.
The pilot laboratory will calibrate all thermometers of the intercomparisons according to the instruction given below twice (in the beginning and at the end of the intercomparison).

The sub-pilot laboratories will calibrate the thermometers of their loops in the same way as all other laboratories. Additionally they will compare the Ag fixed point cell to their own national standard. The procedure for this comparison follows the usual procedure. An example can be found in the technical protocol for CCT K4. Receiving of the Ag cell and the result of the comparison will be reported to the pilot laboratory.

7. DETAILED INSTRUCTIONS FOR PARTICIPATING LABORATORIES

(only calibration of thermometers)

Remark

Each participant should follow the instructions given in

(A1) (Receiving the Thermometers)

as soon as possible after receiving the thermometers.

After this, calibrate the specified thermometers for

(B): SPRT (25 Ω) at the freezing point of Al

(C): HTSPRTs at the freezing point of Ag

The sequence of (B) and (C) is not mandatory.

After the calibrations, pack securely the thermometers and transport them to the next participant. If any discussion upon this protocol and the measurements during this international comparison is necessary, the participant should share the information through e-mail to all participants.

(A) Receiving the Thermometers

Procedures

1. Upon receiving the transfer SPRT and the transfer HTSPRTs, the host laboratory must inspect the thermometers for damage. The host laboratory must report the condition of the thermometers to the pilot laboratory. If there is damage, the pilot laboratory will give instructions on how to proceed.

2. If no damage is reported to the pilot laboratory, the host must measure the resistance of the transfer PRT and the transfer HTSPRTs using two measuring currents (in order to determine the zero-power value) in a triple point of water (TPW) cell.

3. After completing measurements according to step 2, resistance values of the 3 transfer thermometers at the TPW (R_{TPW1}), which are extrapolated to a measuring current of 0 mA and which are corrected for the hydrostatic head effect, must be communicated to the pilot laboratory before proceeding further measurements. Based on this information, the pilot laboratory will advice the host laboratory on the next step to be taken.

(B) SPRT (25 Ω) to be calibrated at the freezing point of Al

Remarks

Firstly, the SPRT (25 Ω) should be annealed according to the procedures given in the following paragraphs. If the specified criteria are fulfilled, calibrate the SPRT using three plateaus of the aluminum freezing point. Measure the immersion characteristics using the transfer SPRT during one of the three plateaus or in an additional fourth plateau.

Procedures

1. Anneal the transfer SPRT before the measurement of the freezing-point temperatures of aluminum. 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, then reduce it to 500 °C over approximately 1.5 to 4 hours. When the temperature has reached 500 °C, remove slowly the SPRT from the furnace directly to the room environment.

2. After the SPRT has cooled down to room temperature, measure its resistance at the TPW (R_{TPW2}).

3. If the change of the resistance of the PRT at the TPW before and after annealing (R_{TPW2} - R_{TPW1}), as measured according to steps 2 and 3, is equivalent to 0.5 mK or smaller, proceed to step 4, otherwise repeat step 1 and 2. If the requirement is not fulfilled of the second annealing, contact the pilot or sub-pilot laboratory.

4. After the annealing and the measurements at the TPW are completed, calibrate the transfer SPRT at the aluminum freezing point. The SPRT must be preheated in an annealing furnace which is preheated to 500 °C, and then the temperature is increased up to a value between 600 °C and 660 °C over approximately 1 hour. The transfer SPRT should be removed then from the annealing

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

5. After calibration measurements at two currents at the freezing point of aluminum, measure the immersion characteristics using the transfer SPRT whenever the participating laboratory decide to measure it during this plateau. The method for measuring the immersion characteristics should follow the common procedure practiced by each participating laboratory. If the participating laboratory does not decide to measure the immersion characteristics during this plateau, proceed to step 6.

6. 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.

7. When the temperature of the annealing furnace (along with the PRT) has been dropped to 450 °C, wait for appr. 30 minutes and then remove slowly the PRT from the furnace directly to the room environment. After the SPRT has cooled down to room temperature, measure its resistance at the TPW (R_{TPW}).

8. Calibrate the thermometer three times by repeating steps 4 to 7.

9. If the participant decides to conduct the immersion characteristics measurement in an additional plateau, then repeat step 4, 5, 6 and 7.

(C) 2 HTSPRTs to be measured at the freezing point of Ag

Remarks

In this protocol only the calibration of 1 thermometer is described. The calibration of the second thermometer follows the identical procedure. The time where the thermometer is at temperatures above 500 °C should be minimized (< 8 hours).
Firstly, the HTPRT should be annealed according to the procedures given in the following paragraphs.
If the specified criteria are fulfilled, calibrate the HTPRT using two plateaus of the silver freezing point.
Perform an immersion test using the transfer HTPRT during one of the two plateaus or an additional plateau.
If desirable for reasons of organisation, the thermometer may after cooling down remain at 500 °C for several hours in all steps described below.

Procedures

1. Anneal the transfer HTPRT before the measurement of the freezing-point temperature of silver. Insert slowly the transfer HTPRT into an annealing furnace which is preheated to 500 °C, and then increase the temperature of the annealing furnace to 975 °C over approximately 2 hours. Maintain the temperature at that point for 30 minutes, then reduce it to 500 °C over approximately 4 to 8 hours. When the temperature has reached 500 °C, remove slowly the HTPRT from the furnace directly to the room environment.

2. After the transfer HTPRT has cooled down to room temperature, measure its resistance at the TPW (R_{TPW2}).

3. If the change of the resistance of the transfer HTPRT at the TPW before and after annealing (R_{TPW2} - R_{TPW1}), as measured according to steps 2 and 3, is equivalent to 1.0 mK or smaller, proceed to step 4, otherwise repeat step 1 and 2. If the requirement is not fulfilled after the second annealing, contact the pilot or sub-pilot laboratory.

4. After the annealing and the measurements at the TPW are completed, calibrate the transfer HTPRT at the Ag freezing point. The monitor HTPRT should be used in the realization of the freezing point of silver. The transfer HTPRT must be preheated in an annealing furnace which is preheated to 500 °C, and then the temperature is increased up to 960 °C over approximately 2 hour. The transfer HTSPRT should then be removed from the annealing furnace and inserted into the well of the silver freezing point cell and calibrated in a stable plateau of the freezing curve of silver, preferably between 85% and 70% of metal in the liquid phase.

5. After measurements at two currents at the freezing point of silver, perform an immersion test with the transfer HTPRT whenever the participating laboratory decides to measure it during this plateau. For the immersion test lift the thermometer by 10 mm and measure the temperature change of the HTSPRT. If the participating laboratory does not decide to measure the immersion characteristics during this plateau, proceed to step 6.

6. The transfer HTPRT should be removed and inserted into the annealing furnace at 960 °C. Increase the temperature to 975 °C, anneal the thermometer for 30 minutes and then cool down to 500 °C within approximately 4 to 8 hours.

7. When the temperature of the annealing furnace (along with the transfer HTPRT) has been dropped to 500 °C, remove slowly the HTPRT from the furnace directly to the room environment. After the transfer HTPRT has cooled down to room temperature, measure its resistance at the TPW (R_{TPW}).

8. Calibrate the thermometer a second time by repeating steps 4 to 7.

8. REPORT OF RESULTS

The participating laboratories must send the followings to the pilot laboratory by the specified schedule.

1. Resistance values of the transfer PRT in its specified freezing-point cells [= $R(T)_{\mathrm{in\ the\ freezing\text{-}point\ cell}}$] and in the TPW cell [$R$(273.16 K)], and the related resistance ratios [= $R(T)_{\mathrm{in\ the\ freezing\text{-}point\ cell}}$ / R(273.16 K)] obtained after the measurement of aluminum and silver freezing point.
   The participating laboratory report to the pilot laboratory the non-corrected $R(T)/R_{\mathrm{std}}$ data at two currents for deriving 0 mA value and the corrected values for hydrostatic head, gas pressure, and self-heating.

2. The immersion curves obtained using the transfer PRT in the Al freezing point cell used for the comparison.

3. Freezing curves of aluminum and silver cells measured by monitor PRTs.

4. Uncertainty analysis according to the "Guide to the Expression of Uncertainty in Measurement", ISO 1993, ISBN 92-67-10188-9. The uncertainty analysis must include the following terms and other items that the participating laboratory wants to include.

   Type A

   - Freeze-to-freeze repeatability with the degree of freedom

   Type B

   - Chemical impurities of Al and Ag cell
   - Hydrostatic-head errors
   - Bridge measurement errors
     effects of changes in reference resistors
     non-linearity of bridge
     quadrature effects in ac measurements
   - Uncertainty propagate from the TPW
   - SPRT self heating errors
   - Heat flux-immersion errors
   - Errors in gas pressure in the Al and Ag cell
   - Errors in the choice of freezing point value from plateau of the freezing curve
   - High-temperature insulation degradation of the transfer HTPRT

5. Details of instrumentation and experimental techniques of the participating laboratory must be reported to the pilot laboratory

Details of instrumentation

Bridge
Manufacturer and model
Frequency
Bandwidth
Gain
Quad gain
Normal measurement currents
Self-heating currents

Reference resistor

Manufacturer and model
How maintained
Temperature control stability of maintaining bath
Temperature coefficient of reference resistor

Freezing point cells

Manufacturer and model
Type of cell(open/closed)
Length and diameter of cell(cm)
Crucible materials
Source of crucible
Sample source
Purity of sample
Immersion depth of HTPRT (mm)
Thermometer well ID (mm)
Pressure in cell(kPa)

Furnace details

Manufacturer and model
Dc or Ac heat power
Furnace control type
How many zones in furnace
Uniformity in furnace with cell
Temperature stability over 16 hrs(mK)

Details of experimental techniques

Length of time that the sample is heated above the melting point before nucleating freeze
Method of nucleation freezes
Duration of freeze(h)

9. Customs declaration

TO WHOM IT MAY CONCERN

EUROMET Regional Key Comparison T-K4 (EUROMET Project 820)

EUROMET is an organisation representing the National Measurement / Standards Laboratories of a large number of countries/territories in the European region. Its broad objective is to improve the measurement capabilities in the European region by sharing facilities and experience in metrology.

As part of a major intercomparison program, EUROMET is conducting an intercomparison on International Temperature Scale of 1990 (ITS-90) in the temperature range 660 °C to 960 °C involving the participants as given in the Laboratory Schedule.

The project is co-ordinated by

Dr. Dieter Heyer

PTB

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D-10587 Berlin

Phone: +49 30 3481 595

Fax: +49 30 3481 504

Email: dieter.heyer@ptb.de

The following artefact is circulated among the participants for calibration:

Standard Platinum resistance thermometer N°:_________________________

The purchase/manufacturing cost of the artefact was_________________. However it has no commercial value (it is not for sale). It is meant solely for the calibration of national standards and will be re-exported immediately after the calibration is completed (see enclosed Laboratory Schedule).

We request that the device is not handled or removed from the container/package. If a Customs inspection is required then please contact the relevant person listed in the attached schedule so that he/she can be present and help you unpack it.

Please send to
Dieter Heyer
PTB
Fax: +49 30 3481 595
Email: dieter.heyer@ptb.de

ARTEFACT RECEIVED

EUROMET Key Comparison T-K4 (Project 820)

Name of participating lab:

The____ and its ATA Carnet was

(SPRT references)

received at_________________________

(name of laboratory)

on ______________________________

(date)

The condition when it was received was:

☐ in good physical and working order
☐ damaged *(explain)*

Signature

Please send to

Dieter Heyer

PTB

Fax: +49 30 3481 595

Email: dieter.heyer@ptb.de

ARTEFACT SHIPPED

EUROMET Key Comparison T-K4 (Project 552)

Name of participating Lab:

The____ and its ATA Carnet was hand

(SPRT reference)

delivered to_________________________

(name of laboratory)

on ______________________________

(date)

Signature

PROTOCOL APPROVAL

EUROMET Key Comparison TC-K4 (Project 820)

This approval concerns only the protocol of the measurement.
During the last EUROMET meeting held in Ljubljana (30-31 March 2004) the protocol has been globally agreed by all the participants.

We need a formal approval from any participant to the final protocol of EUROMET project 820.

Name of participating lab:

☐ We approve the Protocol (version 2005-01-04) and we agree to participate in the EUROMET Key comparison TC-K4 (Project 820)

☐ We do not approve the Protocol (version 2005-01-04) and we will not be participating in the EUROMET Key comparison TC-K4 (Project 820)

Date ______________________________

Name _____________________________

Responsibility ________________________

Signature _________________________

Appendix B: Comparison of Ag freezing point cells among the sub-pilots

The comparison among the sub-pilots and the pilot with a Ag silver freezing point cell did not follow a very strict protocol. Instead the participants were asked to follow basically the protocol of CCT K4, but applying their own procedure for their cells.

The Ag fixed point cell was provided by NMi-VSL. It is a re-sealable cell, which was hand carried in a suitcase in dismounted condition. The mounting instructions are given at the end of appendix B.

Among the sub-pilots there was a discussion about the extra measurements with the Ag freezing point cell. The summary of this discussion was distributed by email on January 12, 2005 as follows:

Comments of the sub-pilots on the protocol of EUROMET 820

Replies by Erich Tegeler are in red.

Comments from Peter Steur

Inserting a Silver Point cell, even if only partially, in the comparison, can only improve the reliability of the outcome. As such, I have no objections to it, and, in an overall sense, I agree with the modified proposal, but I do have a number of questions and a couple of remarks :

Question 1: Has it been ascertained that the travelling Ag cell is compatible with the dimensions of the sub-pilots' furnaces?
The drawing of the cell was sent to the sub-pilots on 12. Jan. 05. I hope that there are no problems.

Question 1: How is the Ag cell to be transported from one sub-pilot to the other? By hand or by courier? This information is to be specified in the protocol and preferably added at the bottom of page 2.
Hand-carrying of the cell was included on page 2.

Question 3: How does the start of measurements of the Silver cell (NL), Table 2, compare with the start of measurements of the Thermometers (NL), Table 1, November and December
respectively?
The Has been included.

Ag fixed point cell will form its own loop, independent from the thermometer loops.

Question 4: How come that loops A and B have already started when the protocol is still to be accepted by the participants?
The protocol was accepted by the participants (at least nobody had any objections that were not considered) and by CCT WG7. Now we are dealing only with modifications that became necessary due to problems of the HTSPRTs. The modified protocol will be sent to all participants when the all modifications proposed by the sub-pilots are included in the final text

Remark 1: On page 4 there are two tables, each labelled with the number 1. In the second one, thus Table 2, IT(SC) should be IT(SD).
Has been changed into Table 1a and Table 1b.

Remark 2: It would be desirable to add, on page 9, a procedure for "Receiving the Silver cell (sub-pilots only)".

Comments from Eliane Renaot

We agree the protocol of the EUROMET Project 820. Nevertheless I have some remarks:

- The inner diameter of the thermometer wells in our Al and Ag cells is 8 mm, so the diameter of the thermometers have to be smaller than 8 mm.(7.7 mm maximum).
The diameter of the thermometers is 7.7mm or less.

- What are the geometrical characteristics of the transfer Ag cell ? The sub pilots have to verify that these characteristics are compatible with their local furnaces
The drawing of the cell was sent to the sub-pilots on 12. Jan. 05. I hope that there are no problems.

- There are some contradiction between the stability of the SPRT required before and after the freezing point of **silver** page 8 (0.5 mk) and page 11 (1 mK)
Has been changed to 1 mK for all cases.

- in order to avoid a large drift of the SPRTs I suggest you recommend a maximum duration while the SPRT can stay in the fixed point.
That is a good idea. I recommend a maximum of 10 hours at temperatures >500 °C for each thermometer..

Comments from David Head

Perhaps I don't understand something - why is SK in two loops? Is it something to do with 25 Ohm PRTs?
The reason is as follows: SK participated in CCT K3, but not in CCT K4. Therefore SK can be sub-pilot for loop A, which is only a comparison at the Al fixed point. On the other hand SK is an ordinary participate for the measurements at the Ag fixed point.

We are assuming that under this protocol the sub labs are responsible also for transporting the ingot to the next sub pilot? An additional cost which we will have to bear as a bigger lab. However Jayne is concerned that it will be very tight to do both the comparison of the cells as well as the
measurement of the RT and transporting both the RT and the cell on to separate destinations, all within 6 weeks. (I suppose we will only hold up the second half of loop 4 if we are late!).
You are right, but I see no other possibility. In many cases the flight tickets are really cheap, so the costs should not really be a problem.
Maybe 6 weeks for the measurements including the comparison of the Ag fixed point are too short. What would you suggest? Would 8 weeks be enough?
I suggest we leave the timetable as it is - it might slip by the time it gets to us anyway - we will try to do it but we just want you to be aware of time problems (also in May six weeks is a UK holiday and schools off so Jayne may be out for one week with her children)

For us time is also money so re travel it is not so much the ticket but a day of my time is over £800 (1200E)! Anyway that is something we must just live with according to our full costs accounting system. Maybe I can visit Sweden - I have not been there and Jayne can go to IMGC and also look at their heat pipe set up; she has not seen it. Not to worry.

I note that the countries needing Carnets are in different loops viz Ru in C while CH and TR are in B, rather than having them all in one HTPRT group; but maybe Latvia is easy way into Russia?

I hope that the carnets will not be a problem. Most of the thermometers are provided by Hart Scientific; I suppose that they are in Europe with a carnet, but I do not have this carnet. So we have issued new carnets. Anatoly Pokhodun up to now did not decide if VNIIM will particpate in the comparison. So I decided just to wait and see.

Will the Netherlands do a before and after comparison of the travelling ingot with another? Non travelling ingot in the Netherlands? (I assume you will as it goes back to you before the NL).

This has still to be decided.

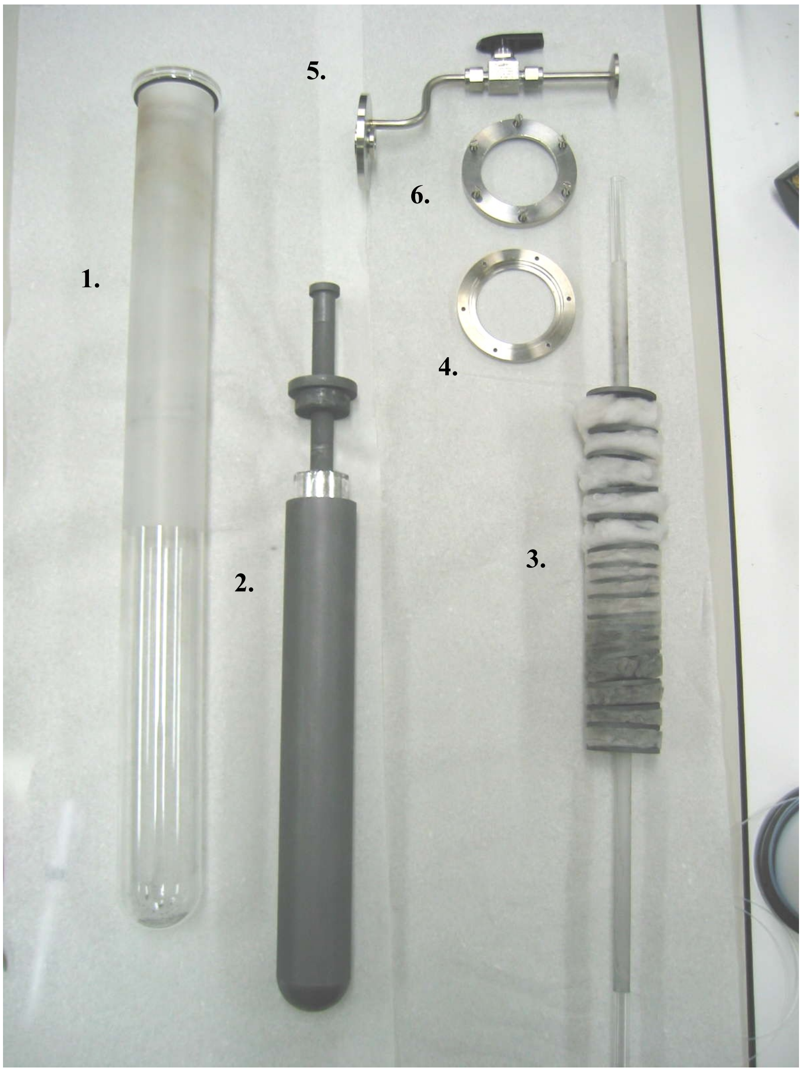
We also noted one other thing in the protocol In one point after annealing you decrease to 500 °C but everywhere else it is 450 °C - is this a deliberate difference?

500 °C is for HTSPRTs, 450 °C for SPRTs (25 Ohm).

Instruction for mounting and dismounting the VSL Ag05 cell Euromet-820 Key Comparison.

The cell is dismounted for transport into the following components:

1. Quartz outer tube / container
2. Graphite crucible containing the silver
3. Quartz thermometer well together with quartz wool isolation and graphite disks.
4. Bottom flange
5. Cell cover with gas handling tube and valve
6. Top flange with screws
7. Spare screws
8. Spare o-rings



Mounting instructions:

Step 0

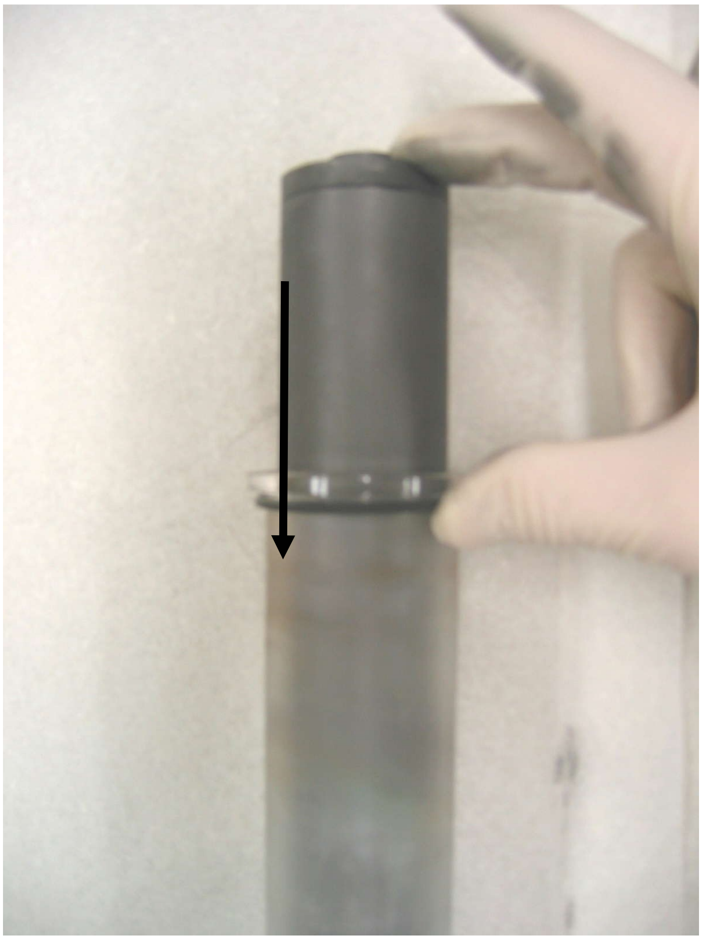
SPECIAL WARNING!!!

Be **extra careful** while unpacking and later packing of the graphite crucible with the silver ingot. As soon as you tilt this part upside down with just a very slight angle, the large weight of the silver will cause it to slide down the crucible. It will likely cause the inner graphite well to break exactly at the point where it sticks out from the silver as soon as it falls on the table (or even worse: the floor…).

Please, before transporting it to the next lab, attach a warning label on the packed crucible + silver that indicates which side is the top and pack it such that the silver ingot cannot move.

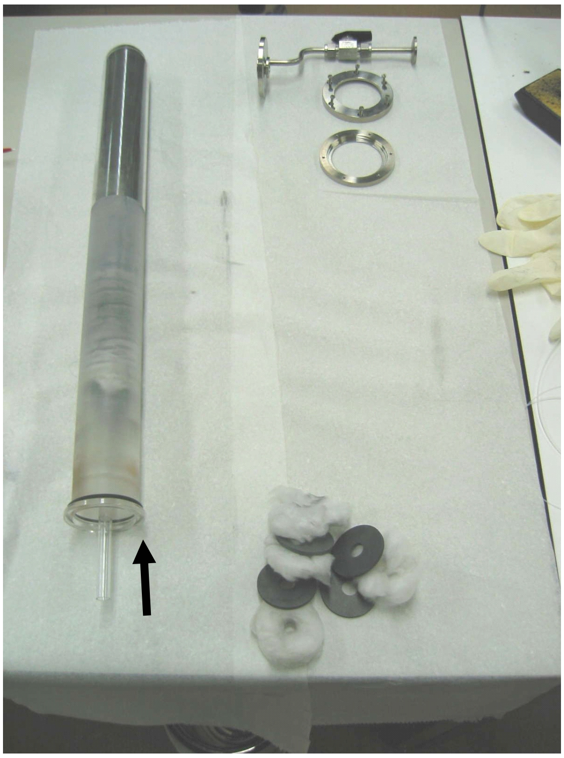
Step 1.

Insert the graphite crucible into the quartz container preferably using dust free disposable handgloves while holding the quartz container in a horizontal position.



Step 2.

Mount the thermometer well + isolation and graphite disks together into the quartz container. If you find this easier, first insert the thermometer well and insert the isolation layers one by one. Start with a graphite disk at the bottom.



Step 3.

After (or before) all the isolation and graphite disks have been stuffed in, place the cell vertically.

Step 4.

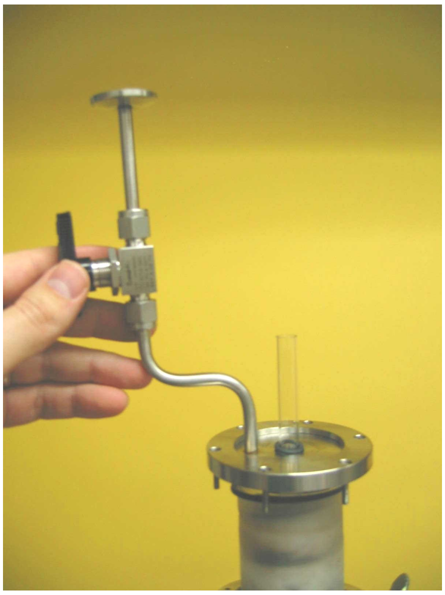
Mount the o-ring and bottom flange

Step 5.

Mount the cell cover, with the small o-ring around the quartz thermometer well

Step 6.

Mount the top flange with the crews over the cell cover.



and connect the top and bottom flange. Screw in the screws inch by inch (actually mm by mm) taking turns on the screws that are lined up opposite. Continue until the flanges are tightly together taking care not to break the quartz container.

Step 7.

Finally, screw tightly the seal for the thermometer well using a wrench.

Step 8.

Before heating the cell, check for leaks. In case of a leak, try to tighten the screws in the top flange a bit further and/or tighten the thermometer well seal. If the leak persists, check the o-rings for deformation due to excessive heating and replace them with new o-rings.

Appendix Leak testing and further preparation

The leaktest can initially be executed by simply pumping on the cell for some time and closing the valve to the pump while monitoring the pressure in the cell. The pressure in the cell should only slowly rise (outgassing) and stabilise at a few times 10-3 mbar.

If above criteria can not be met even after precautions have been taken as described in step 8., use a leak detector to find the exact position of the leak.

Further preparation:

The NMi-VSL procedure for further preparing the cell after leak testing has passed is the following:

1. Continue pumping on the cell.
2. Heat up the cell to 820 °C while pumping and keep pumping like this overnight.
3. Stop pumping and fill the cell with an overpressure (100 mbar) of very pure argon.
4. Increase the temperature further and slowly melt the silver at a few degrees above the melting point.
5. After the silver has melted, flush the cell and gas lines three times:
   Slowly pump on the cell until an underpressure of 0.9 bar has been reached and fill again up to 1.1 bar (repeat 3 times and record the end pressure at about 1.1 bar).
6. The cell is now ready for a freeze plateau to be induced according to your own procedure.

Appendic C: Instrumental details

The tables presented in the next pages correspond the experimental details Excel files sent by the participants in their reports.

Instrumentation Details

EUROMET Regional Key T-K4 Comparison (EUROMET Project 820), Loop A

Laboratory name	**DTI (DK)**	**Justervesenet (NO)**	**OMH (HU)**	**INM (RO)**	**UME (TR)**	**HR**
Bridge used for SPRT (25 Ohm)						
Manufacturer	ASL	Measurement International	Guildline	Automatic Systems	ASL - MI	ASL
Type	F18	Model 6010T	9975	F18	F18 - 6010	F700
Unity reading	1,0000000	Ohm	8 digit	1.000 000 0	1,0000001	1.000 000
AC or DC	AC	DC	DC	AC	AC -DC	AC
If AC, give Frequency	75 Hz			25 Hz	Low	50 Hz
If DC, give Period of reversal		5 s	4 sec		30 s	
Normal measurement current	1 mA	1 mA	1 mA	1 mA	1 mA	1 mA
Self-heating current	1.41 mA	1,41 mA	1,414 mA	1.414 mA	1,41 mA	1.41 mA
Evaluation of linearity of resistance				No		
bridge (yes or not)	yes	not	Yes		yes	yes
If yes, How?	resistors		from specification		by checking the equality Rx/Rs = 1/ (Rs/Rx)	RBC 400
Reference resistor 1						
Manufacturer	H.M. Sulivan Ltd	Tinsley	Cambridge	H Tinsley & Co Ltd	Tinsley	Tinsley
Type	1613	Model 5685A	10 ohm	5685 A, 100 ohm	100 ohm, 5685 A	5685A
Reference resistor temperature con	yes	yes	Yes	Yes	yes	Not
If yes, How?	termostated oilbath	Temperature controlled bath	in thermostat, measured with PRT	Oil bath and room temperature control	In a temperature-controlled oil bath	Unstirred bath, temperature correction
Al Cell						
Home made or not	no	Pyrocontrole, France	Not	Isothermal Technology Ltd	Not, NPL made	Purchased, Isotech
Closed cell or open	closed	Closed	Closed	Closed	Closed	Open
Nominal purity	6N		6N	99.999 9%	99,9999%	6.5N
Immersion depth of middle of the S	18	12,5 cm	17,5 cm	17 cm	17,4 cm	18 cm
Al Furnace						
Home or not	no	Isotech Model 17702 ITL	Not home made	Isothermal Technology Ltd	Not, Carbolite	Purchased, Isotech
Type (1 zone, 3 zones, heat pipe, ..	heat pipe	Heat pipe	3 zones	heat pipe	Heat pipe	3 zones
Typical duration of the melting plateau			3 hours	2 h	5 hours	4 hours
Typical duration of the freezing plat	16 h	4 - 7 hours, depends on load	7 hours	5 h	6 hours	8 hours
TPW Cell						
Home made or not	no	NPL type	Not home made	Laboratory of the Government Chemist (UK)	Home made	Purchased, Isotech E11
Immersion depth of middle of the S	26	20,5 cm	23 cm	18.5 cm	24,5 cm	25 cm
How are mantles maintained (ice, b	isothermail enclosure with ice/water, plexiglas jacket	Bath	bath	Ice	In a maintenance bath kept near TPW value	Ice bath

Instrumentation Details						
EUROMET Regional Key T-K4 Comparison (EUROMET Project 820), Loop B						
Laboratory name	**CMI (CZ)**	**GUM (PL)**	**LNE-INM/CNAM (FR)**	**MIRS/FE-LMK (SI)**	**METAS (CH)**	**BEV (AT)**
Bridge used for SPRT (25 Ohm)						
Manufacturer	ASL	ASL	Guildline	Measurements International	Measurements International	Measurements International
Type	F18	F18	9975	6010 B	6010T	6010B
Unity reading	1,0000000	0,0000001	1 10-7	N/A	resistance ratio	Ohm
AC or DC	AC	AC	DC	DC	DC	DC
If AC, give Frequency	75 Hz	75 Hz				-
If DC, give Period of reversal			4s	10 sec	6 s	10 sec
Normal measurement current	1 mA	1 mA	1 mA	1 mA	1 mA	1 mA
Self-heating current	1,414 mA	1,41 mA	1,414 mA	sqrt (2)	v2 mA	1.414 mA
Evaluation of linearity of resistance				Yes		
bridge (yes or not)	yes	not	yes		yes	no
If yes, How?	0,0000001		Batch of standard resistors	RBC	verification of the bridge's ratio(r) linearity with a set of standard resistors	
Reference resistor 1						
Manufacturer	Tinsley	Tinsley	Guildline	TInsley	Tinsley & Co Ltd	Fluke
Type	5685 A (100 ?)	5685 A	9330	5685 A	5685A	742A-100
Reference resistor temperature control (yes or not)	yes	yes	yes	Yes	yes	no
If yes, How?	(23 ±0,05)°C	In the bath in (23,00 ± 0,04)	Guildline bath	oil bath, stability 3 mK over	air thermostat at (36.0 ± 0.5) °C	
Bridge used for HTSPRT						
Manufacturer	ASL	ASL	ASL	Measurements International	Measurements International	Measurements International
Type	F18	F18	F18	6010 B	6010T	6010B
Unity reading	1,0000000	0,0000001	1 10-7	N/A	resistance ratio	Ohm
AC or DC	AC	AC	AC	DC	DC	DC
If AC, give Frequency	75 Hz	75 Hz	25 Hz			-
If DC, give Period of reversal				10 sec	6 s	10 sec
Normal measurement current	20 mA	10 mA	10mA	14,142 mA	10 mA	14.14 mA
Self-heating current	28,828 mA	14,1 mA	14,14mA	20 mA	v20 mA	20 mA
Evaluation of linearity of resistance				Yes		
bridge (yes or not)	yes	not	yes		yes	no
If yes, How?	0,0000001		Batch of standard resistors	RBC	verification of the bridge's ratio(r) linearity with a set of standard resistors	

Instrumentation Details

EUROMET Regional Key T-K4 Comparison (EUROMET Project 820), Loop B, continued

Laboratory name	**CMI (CZ)**	**GUM (PL)**	**LNE-INM/CNAM (FR)**	**MIRS/FE-LMK (SI)**	**METAS (CH)**	**BEV (AT)**
Reference resistor 2	Tinsley					
Manufacturer	5684 A (1 ?)	Tinsley	Tinsley	Tinsley	Tinsley & Co Ltd	Fluke
Type	yes	5685 A	5685 A	5685 A	5685A	742A-25
Reference resistor temperature control (yes or not)	(23 ±0,05)°C	yes	yes	Yes	yes	no
If yes, How?		In the bath in (23,00 ± 0,04)	Guildline bath	oil bath, stability 3 mK over	air thermostat at (36.0 ± 0.5) °C	
Al Cell	Isotech model ITL-M-17672 AL168					
Home made or not	not	ISOTECH Model ITL-M-176	Chauvin Arnoux Pyrocontrol	Not, Isotech Al 130	Isotech, type M-17672, SN	HART Scientific
Closed cell or open	closed cell	CLOSED	licence LNE-INM/CNAM	Closed	closed	closed
Nominal purity	6N	6N	closed	6 N	99,9999%	99,9999
Immersion depth of middle of the SPRT sensible element/cm	134 mm	16 cm	99,9999%	18	13,6	16,5
			14,5 cm			
Al Furnace	Isotech model ITL 17702					
Home or not	not	ISOTECH Model ITL-M-17706		Not	Isotech, type ITL-M-17706,	ISOTECH
Type (1 zone, 3 zones, heat pipe, …….)	heat pipe	heat pipe	Home	3 zones	heat pipe	heat pipe
Typical duration of the melting plateau	8 hours	2 hours	heat pipe	6 hours	4 h with setpoint 5 °C above	2-3 h
Typical duration of the freezing plateau	12 hours	10 hours	6 h	8 hours	15 h	2-3 h
			7 h			
Ag Cell	Isotech model ITL-M-17673 AG39					
Home made or not	not	ISOTECH Model ITL-M-17673 No Ag-89		Not, Isotech Ag 45	Isotech, type M-17673, SN	HART Scientific
Closed cell or open	closed cell	CLOSED	Home	closed	closed	closed
Nominal purity	6N	6N	closed	6 N	99,9999%	99,9999
Immersion depth of middle of the SPRT sensible element/cm	130 mm	16 cm	99,9999%	18	16	17,5
			14,5 cm			
Ag Furnace	Isotech model ITL 17702					
Home or not	not	ISOTECH Model ITL-M-17706		Not, Isotech	Isotech, type ITL-M-17706,	ISOTECH
Type (1 zone, 3 zones, heat pipe, …….)	heat pipe	heat pipe	Home	heat pipe	heat pipe	heat pipe
Typical duration of the melting plateau	4 hours	3 hours	heat pipe	4 hours	4 h with setpoint 5 °C above	1-2 h
Typical duration of the freezing plateau	8 hours	10 hours	8 h	6 hours	7 h	1-2 h
TPW Cell	cells NPL type 32 N° 1025					
Home made or not	not	not, glass cell NPL type 32 N° 957		Not, NMi-VSL	Hart Scientific, type 5901, S	HART Scientific
Immersion depth of middle of the SPRT sensible element/cm	250 mm	19 cm	not / UME	27	24	18
How are mantles maintained (ice, bath,….)	Water Triple Point Maintena	TPW cell in PVC box placed in the ice in Dewar vessel	23 cm	bath	ice	bath
	Isotech		Hart bath			

Instrumentation Details

EUROMET Regional Key T-K4 Comparison (EUROMET Project 820, Loop C)

Laboratory name	**PTB (DE)**	**SMU (SK)**	**NPL (UK)**	**SP (SE)**	**UJ-PFI (LT)**	**VNIIM (RU)**
Bridge used for SPRT (25 Ohm)						
Manufacturer	ASL	ASL	Automatic Systems	ASL	Measurements International	Guildline Instruments Inc.
Type	F18	F900	F900	F-18	Automatic DC bridge, model	GUILDLINE 9975
Unity reading	1	18÷30 (intervals of 10 s, time of reading 3÷5 min)	3 205 001	1.0000000 with IEEE two more figures		5 µK
AC or DC	AC	AC	AC	AC	DC	DC
If AC, give Frequency	25 Hz	75 Hz	Low (25 Hz)	75 Hz		
If DC, give Period of reversal			Not applicable	0.5 Hz	10 s	4 s
Normal measurement current	1 mA	1 mA	1 mA	1 mA	1 mA	1.0 mA
Self-heating current	1,414 mA	1,414 mA	$\sqrt{2}$ mA	v2 mA	1.414 mA	1.414 mA
Evaluation of linearity of resistance				No		6.3 • 10¯8
bridge (yes or not)	yes	not at present time	Yes, tested at 1 mA		yes	
If yes, How?	RBC 100		RBC		Check the ratios 0.1, 1 and 10 of calibrated resistors of 1 Ohm, 10 Ohms and 25 Ohms	RBC
Reference resistor 1			100 Ohm			
Manufacturer	Tinsley	Tinsley and Co	H Tinsley & Co Ltd	Tinsley	H. Tinsley & Co Ltd., UK	Association "Priborostroiteli", Russia
Type	5685A (100 ◊ ↔	100 Ohm, 5686 A	5684C	5685A 100 ohm s/n 237850	Wilkins type, model 5685A - 25 Ohms	MC3020, 10 Ohm
Reference resistor temperature control (yes or not)	yes	yes	Yes	Yes	yes, at 23 °C, +/- 0.02 °C	Yes
If yes, How?	Temperature controlled oil bath at (23 ± 0,02) °C	temperature controlled bath	Temperature controlled oil bath	In Temperature Controlled Enclosure; Tinsley 5648 s/n 64/008 at 36.0 °C; Control ratio 30 e.g. 1 °C amb. eqv. 33mK; -0.53 ppm/°C	Resistor kept in the oil bath made by Isotech, model 455	oil balh 30 ºC ± 0.005 ºC
Bridge used for HTSPRT						
Manufacturer	ASL	ASL	Automatic Systems Laboratories Ltd	ASL	Measurements International Ltd., Canada	Guildline Instruments Inc.
Type	F18	F900	F900	F-18	Automatic DC bridge, model 6010T	GUILDLINE 9975
Unity reading	1	20÷30	3 205 001	1.0000000 with IEEE two more figures		50 µK
AC or DC	AC	AC	AC	AC	DC	DC
If AC, give Frequency	25 Hz	75 Hz	Low (25 Hz)	75 Hz		
If DC, give Period of reversal			Not applicable	0.5 Hz	10 s	4 s
Normal measurement current	10 mA	10 mA	10 mA	10 mA	10 mA	10 mA
Self-heating current	14,14 mA	14,14 mA	$10\sqrt{2}$ mA	10 xv2 mA	14.14 mA	14.14 mA
Evaluation of linearity of resistance bridge (yes or not)	yes	not	Yes, tested at 1 mA	No	yes	6.3 · 10¯8
If yes, How?	RBC 100		RBC		Check the ratios 0.1, 1 and 10 of calibrated resistors of 1 Ohm, 10 Ohms and 25 Ohms	RBC

Instrumentation Details

EUROMET Regional Key T-K4 Comparison (EUROMET Project 820, Loop C), Continued

Laboratory name	PTB (DE)	SK	NPL (UK)	SP (SE)	UJ-PFI (LT)	VNIIM (RU)
Reference resistor 2						
Manufacturer	Tnsley	Tinsley and Co	H Tinsley & Co Ltd	Tinsley	H. Tinsley & Co Ltd., UK	Association "Priborostroitel", Russia
Type	5684 A (1 H)	2.5 Ohm, 5649 A	5664A	5685A 1 ohm s/n 248676	Wilkins type, model 5685A - 1 Ohm	MC3020, 1.0 Ohm
Reference resistor temperature control (yes or not)	yes	yes	Yes	Yes	yes, at 23 °C, +/- 0.02 °C	Yes
If yes, How?	Temperature controlled oil bath at (23 ± 0,02) °C	temperatur controlled bath	Temperature controlled oil bath	In Temperature Controlled Enclosure; Tinsley 5648 s/n 64/009 at 36.0 °C; Control ratio 30 e.g. 1 °C amb. eqv. 33mK; 1.2 ppm/°C	Resistor kept in the oil bath made by Isotech, model 455	oil balh 30 ºC ± 0.005 ºC
Al Cell						
Home made or not	Home made	not	Home made, Ident: Al (sealed)	ENGELHARD PYRO-CONTROLE s/n Al 056	Made by Hart Scientific, model 5907	Home made
Closed cell or open	closed	closed	Closed	long closed	closed	Open
Nominal purity	6 N	6 N	99.9999%	99.999%	99.9999%+	6N
Immersion depth of middle of the SPRT sensible element/cm	155 mm	17	See 1.2 & Table 1	13.5 cm (Hart SPRT)	17	18 cm
Al Furnace						
Home or not	ISOTECH model ITL 17702	not	Commercial - Carbolite	ISOTECH ITL 17702 s/n 111441	Made by Hart Scientific, model 9114	Home made
Type (1 zone, 3 zones, heat pipe, …….)	heat pipe	3 zone	Single zone with heatpipe	1 zone Potassium heat pipe, AC, Eurotherm 818S, Uniform. ±0.01 °C /16h: 0,04 °C	3 zones	3 zones
Typical duration of the melting plateau	4 hours	1 h	8 hours	1 h	4 hours	12 hours
Typical duration of the freezing plateau	8 hours	6 h	8 hours	6 h	8 hours	10 hours
Ag Cell						
Home made or not	PTB	not	Home made, Ident: Ag 2/97	ISOTECH ITL M 17673 s/n AG 35	Made by Isotech Ltd., UK, model ITL M 17673	Home made
Closed cell or open	closed	closed	Closed	short closed	closed	Open
Nominal purity	6 N	6 N	99.9999%	99.9999%	99.9999%+	6N
Immersion depth of middle of the SPRT sensible element/cm	155 mm	17	See 1.2 & Table 1	16.9 cm (Hart) ; 15.9 cm (Chino)	17,5	18 cm
Ag Furnace						
Home or not	Gero	not	Commercial - Carbolite	ISOTECH ITL 17705 s/n 161389-1	Made by Isotech Ltd., UK, model 465	Isotech
Type (1 zone, 3 zones, heat pipe, …….)	heat pipe	heat pipe	Single zone with heatpipe	1 zone Sodium heat pipe; AC, Eurotherm 818S; ±0.02 °C; ±0.05 °C	3 zones	heat pipe
Typical duration of the melting plateau	4 hours	1 h	8 hours	1 h	3 hours	5 hours
Typical duration of the freezing plateau	8 hours	2 ÷ 3 h	8 hours	6 h	5 hours	4 hours
TPW Cell						
Home made or not	not	not	Commercial – Isotech, Ident: B11-323	NPL 32 s/n 980	Made by Hart Scientific, model 5901	Home made
Immersion depth of middle of the SPRT sensible element/cm		23. Feb	See 1.2 & Table 1	19.7 cm (Hart) ; 18.7 cm (Chino) ;20.3 cm (Hart SPRT)	23 (for SPRT), 22 (for HTSPRT)	26 cm
How are mantles maintained (ice, bath,….)	Water triple point maintenance baths (ISOTECH and Hart	bath	Crushed ice	TPW bath ISOTECH ITL-M-18233	Cell kept in the bath made by Hart Scientific, model 7012	ice

Instrumentation Details

EUROMET Regional Key T-K4 Comparison (EUROMET Project 820), Loop D

Laboratory name	**CMA (FI)**	**EIM (GR)**	**INRiM (IT)**	**IPQ (PT)**	**C.E.M (ES)**	**VSL (NL)**
Bridge used for SPRT (25 Ohm)				AC Resistance Bridge		
Manufacturer	ASL	Measurements International Ltd,	ASL	ASL	ASL	MI
Type	F18	6010B	F18	Model F18	F18	MI6010T
Unity reading	1	1,000000000	1,0000000	ratio (0 to 1,299 999 9)	1,000 000 0	?
AC or DC	AC	DC	AC	AC	DC	DC
If AC, give Frequency	75 Hz		25 Hz	75 Hz	75 Hz	
If DC, give Period of reversal		8 s		--		3 seconds
Normal measurement current	1 mA	1mA	1 mA	1 mA	1 mA	1 mA
Self-heating current	sqrt(2) mA	1.4142 mA	1,4142 mA	1,414 mA	1,414 mA	1.41421 mA
Evaluation of linearity of resistance						
bridge (yes or not)	yes	yes	yes	No	yes	yes
If yes, How?	RBC 100	reference resistors at ratios of 1/25, 1/4, 1/1,4/1	by means of RBC unit		RTU	RBC and reciprocal measurements
Reference resistor 1						
Manufacturer	Tinsley	H. Tinsley&Co Ltd, UK	Tinsley	Tinsley	Tinsley	Tinsley
Type	5658A 25 ohm	5685A, 25 ohm	5685A	Model 5685A	model 5685 A; 1 □	5684
Reference resistor temperature control (yes or not)	yes	yes	yes	Yes	yes	yes
If yes, How?	Tinsley 5648	oil bath at 23 °C	Tinsley Enclosure 5648	Thermoregulated bath, model KB24 stability better than 0,01 ºC	isothermal enclosure at 36 ºC	Hart Scientific 7008 oil bath
Bridge used for HTSPRT				AC Resistance Bridge		
Manufacturer	ASL	Measurements International Ltd, Canada	Automatic Systems Laboratories	A□L -Automatic Systems Laboratories	ASL	MI
Type	F18	6010B	F18	Model F18	F18	MI6010T and 6015T
Unity reading	1	1,000000000	1,0000000	ratio (0 to 1,299 999 9)	1,000 000 0	?
AC or DC	AC	DC	AC	AC	DC	DC
If AC, give Frequency	75 Hz		25 Hz	25 Hz	75 Hz	
If DC, give Period of reversal		8 s		--		3 seconds
Normal measurement current	10 mA	10 mA	10 mA	10 mA	1 mA	10 mA
Self-heating current	10 mA x sqrt(2)	14.142 mA	14,142 mA	1,414 mA	1,414 mA	14.1421 mA
Evaluation of linearity of resistance		yes				
bridge (yes or not)	yes	reference resistors at ratios of 1/25, 1/4, 1/1,4/1	yes	No	yes	yes
If yes, How?	RBC 100		by means of RBC unit		RTU	RBC and reciprocal measurements

Instrumentation Details

EUROMET Regional Key T-K4 Comparison (EUROMET Project 820), Loop D, continued

Laboratory name	**CMA (FI)**	**EIM (GR)**	**INRiM (IT)**	**IPQ (PT)**	**C.E.M (ES)**	**VSL (NL)**
Reference resistor 2						
Manufacturer	Tinsley	H. Tinsley&Co Ltd, UK	Tinsley	Tinsley	Tinsley	Tinsley
Type	5685A 1 ohm	5685A, 1 ohm	5685A	Model 5685 AC	model 5685 A; 100 ÷	5684
Reference resistor temperature cor	yes	yes	yes	Yes	yes	yes
If yes, How?	Tinsley 5648	oil bath at 23 °C	Tinsley Enclosure 5648	Thermoregulated bath, model KB24 stability better than 0,01 ºC	oil bath at 23 ºC	Hart Scientific 7008 oil bath
Al Cell			Al Co3			
Home made or not	Hart Scientific	ITL-M-17672, 1995, Al49	Home made	ISOTECH	Not	Home made
Closed cell or open	closed	Closed	Open	Closed cell -Al50	open	open
Nominal purity	99.9999%+	99,9999%	6N	metal purity: 99,9999 %	99,999 9 %	6N
Immersion depth of middle of the S	15,75	18cm	15,4	18,4 cm	140 mm SPRT (s/n 1450)	22 cm
Al Furnace						
Home or not	Isotech	Isotech	Home made	ISOTECH – Model ITL – M17702	Not	Home
Type (1 zone, 3 zones, heat pipe, .	heat pipe	Very High Temperature Dual Furnace-17705 (Heat pipe)	Heat pipe	1 zone	Sodium heat pipe	heat pipe
Typical duration of the melting plat	2 h		40 hours	16 hours	8 h	few hours
Typical duration of the freezing plat	6 h		40 hours, Time sample is kept above melting point before nucleation	30 hours	8 h	40 hrs
Ag Cell			Ag JM2			
Home made or not	Isotech	ITL-M-17673, 1995, Ag48	Home made	ISOTECH	Not	Home
Closed cell or open	closed	Closed	Open	Closed cell -Ag58	closed	open
Nominal purity	100,00%	99,9999%	6N	metal purity: 99,9999 %	99,999 9 %	6N
Immersion depth of middle of the S	15,75	18cm	16,4	16 cm	130 mm HTSPRT (s/n 1041 and 1043)	17 cm
Ag Furnace						
Home or not	Isotech	181303/3	Home made	YELLOW SPRINGS – Model YSI M17702	Not	Home
Type (1 zone, 3 zones, heat pipe, .	heat pipe	Very High Temperature Dual Furnace-17705 (Heat pipe)	Heat pipe	1 zone	Sodium heat pipe	Heat pipe
Typical duration of the melting plat	4 h	Isotech	10 hours		7 h	few hours
Typical duration of the freezing plat	3 h		10 hours	10 hours	6 h	20 hours
TPW Cell			IMGC N°34	Jarrett water triple point cell, type A-11		
Home made or not	Forschungsgemeinschaft für techniches Glas - Wertheim	VNIIM, 2004, Type WTPC, ser 0/12	Home made	home	Not (s/n 2030 and 2036)	Home
Immersion depth of middle of the S	20,35	27cm	24,9	25,5 cm	260 mm HTSPRT (s/n 1041 and 1043), 260 mm SPRT (s/n 1450)	23 cm
How are mantles maintained (ice, b	crushed ice	In a water bath, set at 0.007 Degrees Celsius	Maintenance bath	HART SCIENTIFIC - Model 7312	Water stirred bath	bath

Appendix D: Uncertainty budgets

The following tables are a short version of the uncertainty Excel files sent by participants in their reports.

Uncertainty analysis

Quantity	Components	Uncertainty contribution ui in mK								
			Loop A		**SPRT**	**1444**				
Qi		PTB	DTI	JV	OMH	SMU	INM	UME	FSB	PTB
X_t	Repeatability of readings	see Wt scatter	0,20	0,15	0,29	see Wt scatter	0,13	0,02	0,31	see Wt scatter
$C_{Xt/1}$	Uncertainty linked with purity	0,39	1,50	1,50	0,43	0.35	0,66	1,50	1,50	0,39
$C_{Xt/2}$	Uncertainty linked Hydrostatic pressure correction	0,01	0,02	0,02	0,01	0.0053	0,02	0,02	0,03	0,01
$C_{Xt/3}$	Uncertainty linked with perturbing heat exchanges	0,10	0,10	0,33	0,06	0.067	0,10	0,05	3,00	0,10
$C_{Xt/4}$	Uncertainty linked with self-heating correction	0,01	0,20	0,14	0,15	0.075	0,05	0,10	0,20	0,01
$C_{Xt/5}$	Uncertainty linked with bridge linearity	0,07	0,20	0,01	0,02	0.04	0,16	0,05	0,25	0,07
$C_{Xt/6}$	Uncertainty linked with AC/DC current			0,07	0,00		0,01	0,09	5,00	
$C_{Xt/7}$	Uncertainty linked with gas pressure	0,21	1,80	0,24	0,73	0.070	0,01	0,00	0,01	0,21
$X_{0.01\ °C}$	Repeatability of readings	see Wt scatter	0,01	0,05	0,21	see Wt scatter	0,07	0,01	0,01	see Wt scatter
	Repeatability of temperature realized by cell	see Wt scatter	0,02	0,42	0,08	see scatter		0,10	0,25	see Wt scatter
	Short repeatability of calibrated SPRT	see Wt scatter	0,02	0,60	0,08	see Wt scatter		0,02	0,20	see Wt scatter
$C_{0.01°C/1}$	Uncertainty linked with purity and isotopic compositio	0,10	0,01	0,10	0,04	0.19	0,10	0,07	0,10	0,10
$C_{0.01°C/2}$	Uncertainty linked Hydrostatic pressure correction	0,00	0,04	0,01	0,04	0.008	0,03	0,01	0,01	0,00
$C_{0.01°C/3}$	Uncertainty linked with perturbing heat exchanges	0,03	0,01	0,08	0,04	0.040	0,03	0,05	0,10	0,03
$C_{0.01°C/4}$	Uncertainty linked with self-heating correction	0,04	0,05	0,22	0,24	0.067	0,07	**0,04**	0,04	0,04
$C_{0.01°C/5}$	Uncertainty linked with bridge linearity	0,24	0,03	0,02	0,02	0.067	0,13	0,05	0,25	0,24
$C_{0.01°C/6}$	Uncertainty linked with AC/DC current			0,22			0,03	0,10	0,25	
$C_{0.01°C/7}$	Uncertainty linked with internal insulation leakage	0,01	0,01	0,00			0,03	0,00	0,25	0,01
$D_{RS/1}$	Uncertainty linked with stability of RS	0,03	0,03	0,00	0,53	0.025	0,00	0,03	0,50	0,03
$D_{RS/2}$	Uncertainty linked with temperature of RS	0,17	0,05	0,01	0,53	0.0072	0,02	0,05	0,50	0,17
S_{Wt}	Wt scatter	1,08	0,02	0,22	0,01	0.58	0,64	0,25	0,41	1,08
Combined uncertainty		1,22	2,37	1,78	1,32	0.98	0,97	1,54	6,12	1,22
Expanded uncertainty		2,44	4,75	3,55	2,64	1,970	2,20	3,08	12,24	2,44

Uncertainty analysis

Quantity Qi	Components	Uncertainty contribution ui in mK							
		Loop B SPRT 1445							
		PTB	CMI	GUM	LNE-INM	MIRS	Metas	BEV	PTB
Xt	Repeatability of readings	see Wt scatter	0,20	0,40		0,03	0,06	0,06	see Wt scatter
C Xt/1	Uncertainty linked with purity	0,39	1,50	0,40	0,88	0,40	0,67	0,40	0,39
C Xt/2	Uncertainty linked Hydrostatic pressure correction	0,01	0,01	0,02	0,01	0,03	0,01	0,02	0,01
C Xt/3	Uncertainty linked with perturbing heat exchanges	0,10	0,20	0,10	0,06	0,10	0,10	0,23	0,10
C Xt/4	Uncertainty linked with self-heating correction	0,01	0,20	0,20	0,06	0,20	0,23	0,19	0,01
C Xt/5	Uncertainty linked with bridge linearity	0,07	0,10	0,20	0,10	0,03	0,06	0,32	0,07
C Xt/6	Uncertainty linked with AC/DC current			not estimated		0,00	0,05	1,20	
C Xt/7	Uncertainty linked with gas pressure	0,21	0,03	0,60		0,30	0,08	0,20	0,21
X0.01 °C	Repeatability of readings	see Wt scatter	0,17	0,30		0,02	0,08	0,41	see Wt scatter
	Repeatability of temperature realized by cell	see Wt scatter	0,17	0,58	0,09	0,05	0,02	0,11	see Wt scatter
	Short repeatability of calibrated SPRT	see Wt scatter	0,70	0,20	0,60	0,20	0,03	0,21	see Wt scatter
C 0.01°C/1	Uncertainty linked with purity and isotopic compositio	0,10	0,17	0,20	0,20	0,05	0,00	0,21	0,10
C 0.01°C/2	Uncertainty linked Hydrostatic pressure correction	0,00	0,01	0,02	0,01	0,01	0,00	0,34	0,00
C 0.01°C/3	Uncertainty linked with perturbing heat exchanges	0,03	0,07	0,17	0,10	0,01	0,04	0,21	0,03
C 0.01°C/4	Uncertainty linked with self-heating correction	0,04	0,14	0,19	0,20	0,03	0,06	0,03	0,04
C 0.01°C/5	Uncertainty linked with bridge linearity	0,24	0,14	0,19		0,03	0,01	0,27	0,24
C 0.01°C/6	Uncertainty linked with AC/DC current			not estimated		0,00	0,05	0,63	
C 0.01°C/7	Uncertainty linked with internal insulation leakage	0,01		not estimated	0,04	0,00	0,01	0,11	0,01
DRS/1	Uncertainty linked with stability of RS	0,03	0,03	0,01		0,00	0,01	0,21	0,03
DRS/2	Uncertainty linked with temperature of RS	0,17	0,35	0,05	0,03	0,00	0,17	0,12	0,17
SWt	Wt scatter	0,59	0,40	0,79	0,37	1,06	0,13	0,33	0,29
Combined uncertainty		0.81	1,81	1,42	1,18	1,22	0,77	1,70	0,66
Expanded uncertainty		1,62	3,63	2,84	2,36	2,50	1,51	3,41	1,31

Uncertainty analysis

Quantity Qi	Components	Uncertainty contribution ui in mK						
		Loop C SPRT 1446						
		PTB	SMU	NPL	SP	UJ-PFI	VNIIM	PTB
Xt	Repeatability of readings	see Wt scatter	see Wt scatter	0,02	0,05	0,02		see Wt scatter
C Xt/1	Uncertainty linked with purity	0,39	0.35	0,40	0,87	1,50	0,40	0,39
C Xt/2	Uncertainty linked Hydrostatic pressure correction	0,01	0.0053	0,01	0,01	0,00	0,005	0,01
C Xt/3	Uncertainty linked with perturbing heat exchanges	0,10	0.033	0,12	0,46	0,06	0.2	0,10
C Xt/4	Uncertainty linked with self-heating correction	0,01	0.059	0,01	0,06	0,03	0,007	0,01
C Xt/5	Uncertainty linked with bridge linearity	0,07	0.04	0,04	0,04	0,05	0,006	0,07
C Xt/6	Uncertainty linked with AC/DC current			0,00	0,12			
C Xt/7	Uncertainty linked with gas pressure	0,21	0.070	0,05	0,04	0,04	0,009	0,21
X0.01 °C	Repeatability of readings	see Wt scatter	see Wt scatter	0,03	0,10	0,03		see Wt scatter
	Repeatability of temperature realized by cell	see Wt scatter	see Wt scatter	0,00	0,19	0,20		see Wt scatter
	Short repeatability of calibrated SPRT	see Wt scatter	see Wt scatter	0,00	0,19	0,39		see Wt scatter
C 0.01°C/1	Uncertainty linked with purity and isotopic composition	0,10	0.19	0,12	0,19	0,34	0,015	0,10
C 0.01°C/2	Uncertainty linked Hydrostatic pressure correction	0,00	0.008	0,02	0,04	0,00	0,005	0,00
C 0.01°C/3	Uncertainty linked with perturbing heat exchanges	0,03	0.040	0,05	0,10	0,02	0,033	0,03
C 0.01°C/4	Uncertainty linked with self-heating correction	0,04	0.051	0,03	0,19	0,10	0,017	0,04
C 0.01°C/5	Uncertainty linked with bridge linearity	0,24	0.067	0,12	0,15	0,02	0,01	0,24
C 0.01°C/6	Uncertainty linked with AC/DC current			0,00	0,19			
C 0.01°C/7	Uncertainty linked with internal insulation leakage	0,01		0,00				0,01
DRS/1	Uncertainty linked with stability of RS	0,03	0.025	0,00	0,01			0,03
DRS/2	Uncertainty linked with temperature of RS	0,17	0.0072	0,03	0,01	0,01	0,00	0,17
SWt	Wt scatter	0,96	0.35	0,19	0,21	0,06	0,29	1,135
Combined uncertainty		1,00	0.55	0,50	1,12	1,66	0,54	1,17
Expanded uncertainty		2,01	1,100	1,00	2,25	3,33	1,11	2,34

Uncertainty analysis

Quantity Qi	Components	Uncertainty contribution ui in mK							
		Loop D SPRT 1450							
		PTB	MIKES	EIM	INRiM	IPQ	CEM	VSL	PTB
Xt	Repeatability of readings	see Wt scatter	0,20	0,01	0,62	0,02	0,04	0,04	see Wt scatter
C Xt/1	Uncertainty linked with purity	0,39	1,50	1,50	0,39	1,50	0,57	0,39	0,39
C Xt/2	Uncertainty linked Hydrostatic pressure correction	0,01	0,00	0,01	0,01	0,01	0,02	0,01	0,01
C Xt/3	Uncertainty linked with perturbing heat exchanges	0,10	0,07	0,14	0,04	0,10	0,39	0,11	0,10
C Xt/4	Uncertainty linked with self-heating correction	0,01	0,07	0,02	0,01	0,01	0,06	0,07	0,01
C Xt/5	Uncertainty linked with bridge linearity	0,07	0,00	0,06	0,00	0,01	0,00	0,18	0,07
C Xt/6	Uncertainty linked with AC/DC current		0,05	0,00	0,00		0,16	0,00	
C Xt/7	Uncertainty linked with gas pressure	0,21	0,00	0,30	0,05		0,05	0,00	0,21
X0.01 °C	Repeatability of readings	see Wt scatter	0,05	-0,01	0,02	0,01	0,01	0,10	see Wt scatter
	Repeatability of temperature realized by cell	see Wt scatter	0,10	-0,42	0,01		0,02	0,01	see Wt scatter
	Short repeatability of calibrated SPRT	see Wt scatter	0,07	-1,04	---	0,06	0,95	0,10	see Wt scatter
C 0.01°C/1	Uncertainty linked with purity and isotopic compositior	0,10	0,10	-0,42	0,01	0,10	0,00	0,12	0,10
C 0.01°C/2	Uncertainty linked Hydrostatic pressure correction	0,00	0,00	0,01	0,00	0,00	0,00	0,01	0,00
C 0.01°C/3	Uncertainty linked with perturbing heat exchanges	0,03	0,02	-0,01	0,00	0,02	0,00	0,03	0,03
C 0.01°C/4	Uncertainty linked with self-heating correction	0,04	0,00	-0,02	0,01	0,00	0,19	0,14	0,04
C 0.01°C/5	Uncertainty linked with bridge linearity	0,24	0,00	0,00	0,00	0,01	0,00	0,05	0,24
C 0.01°C/6	Uncertainty linked with AC/DC current		0,02	0,00	0,00		0,32	0,00	
C 0.01°C/7	Uncertainty linked with internal insulation leakage	0,01	0,00	-0,03	0,00		0,00	0,00	0,01
DRS/1	Uncertainty linked with stability of RS	0,03	0,04	0,01	0,00	0,07	0,00	0,00	0,03
DRS/2	Uncertainty linked with temperature of RS	0,17	0,01	0,11	0,00	0,00	0,00	0,01	0,17
SWt	Wt scatter	0,97	0,53	1,16	---	0,17	0,49	1,03	1,375
Combined uncertainty		1,01	1,62	2,49	0,73	1,52	2,55	1,42	1,41
Expanded uncertainty		2,02	3,23	5,06	1,47	3,04	6,77	3,34	2,81

Uncertainty analysis

Quantity Q_i	Components	Uncertainty contribution ui in mK							
		Loop B			**HTSPRT 1068**				
		PTB	CMI	GUM	LNE-INM	MIRS	Metas	BEV	PTB
X_t	Repeatability of readings	see Wt scatter	0,250	0,792		0,100	0,473	1,411	see Wt scatter
$C_{Xt/1}$	Uncertainty linked with purity	0,650	2,000	0,850	1,176	0,600	1,122	0,650	0,650
$C_{Xt/2}$	Uncertainty linked Hydrostatic pressure correction	0,110	0,010	0,077	0,032	0,025	0,031	0,062	0,110
$C_{Xt/3}$	Uncertainty linked with perturbing heat exchanges	0,500	0,500	0,202	0,235	0,100	0,333	0,321	0,500
$C_{Xt/4}$	Uncertainty linked with self-heating correction	0,010	0,200	0,282	0,059	0,200	0,750	0,240	0,010
$C_{Xt/5}$	Uncertainty linked with bridge linearity	0,080	0,100	0,250	0,100	0,025	0,087	0,453	0,080
$C_{Xt/6}$	Uncertainty linked with AC/DC current			not estimated		0,000	0,050	1,200	
$C_{Xt/7}$	Uncertainty linked with gas pressure	0,240	0,030	0,600		0,400	0,104	0,173	0,240
$X_{0.01\ °C}$	Repeatability of readings	see Wt scatter	0,210	0,377		0,100	0,262	0,409	see Wt scatter
	Repeatability of temperature realized by cell	see Wt scatter	0,210	0,946	0,108	0,050	0,017	0,754	see Wt scatter
	Short repeatability of calibrated SPRT	see Wt scatter	0,650	0,253	0,759	0,480	0,166	1,390	see Wt scatter
$C_{0.01°C/1}$	Uncertainty linked with purity and isotopic composition	0,130	0,210	0,257	0,253	0,050	0,003	0,301	0,130
$C_{0.01°C/2}$	Uncertainty linked Hydrostatic pressure correction	0,010	0,009	0,031	0,018	0,005	0,004	0,493	0,010
$C_{0.01°C/3}$	Uncertainty linked with perturbing heat exchanges	0,040	0,090	0,214	0,126	0,010	0,038	0,301	0,040
$C_{0.01°C/4}$	Uncertainty linked with self-heating correction	0,050	0,170	0,253	0,253	0,030	0,166	0,048	0,050
$C_{0.01°C/5}$	Uncertainty linked with bridge linearity	0,340	0,170	0,247		0,025	0,014	0,391	0,340
$C_{0.01°C/6}$	Uncertainty linked with AC/DC current			not estimated		0,000	0,050	1,420	
$C_{0.01°C/7}$	Uncertainty linked with internal insulation leakage	0,010		not estimated	0,051	0,000	0,067	0,752	0,010
$D_{RS/1}$	Uncertainty linked with stability of RS	0,120	0,040	0,009		0,001	0,011	0,602	0,120
$D_{RS/2}$	Uncertainty linked with temperature of RS	0,170	0,430	0,064	0,003	0,001	0,231	0,174	0,170
S_{Wt}	Wt scatter	2,500	0,500	1,182	0,490	5,391	2,846	0,713	5,000
Combined uncertainty		**2,680**	**2,328**	**2,154**	**1,558**	**5,467**	**3,235**	**3,314**	**5,100**
Expanded uncertainty		**5,360**	**4,657**	**4,307**	**3,117**	**11,000**	**6,341**	**6,629**	**10,200**

Uncertainty analysis

Quantity Q_i	Components	Uncertainty contribution ui in mK							
		Loop B		HTSPRT 93103					
		PTB	CMI	GUM	LNE-INM	MIRS	Metas	BEV	PTB
X_t	Repeatability of readings	see Wt scatter	0,250	0,891		0,100	0,492	1,404	
$C_{Xt/1}$	Uncertainty linked with purity	0,650	2,000	0,850	1,176	0,600	1,122	0,650	
$C_{Xt/2}$	Uncertainty linked Hydrostatic pressure correction	0,110	0,010	0,077	0,032	0,025	0,031	0,062	
$C_{Xt/3}$	Uncertainty linked with perturbing heat exchanges	0,500	0,500	0,289	0,235	0,100	0,333	0,321	
$C_{Xt/4}$	Uncertainty linked with self-heating correction	0,010	0,200	0,282	0,047	0,200	0,888	0,211	
$C_{Xt/5}$	Uncertainty linked with bridge linearity	0,080	0,100	0,250	0,100	0,025	0,087	0,453	
$C_{Xt/6}$	Uncertainty linked with AC/DC current			not estimated		0,000	0,050	1,200	
$C_{Xt/7}$	Uncertainty linked with gas pressure	0,240	0,030	0,600		0,400	0,104	0,173	
$X_{0.01\ °C}$	Repeatability of readings	see Wt scatter	0,210	0,377		0,100	0,239	0,409	
	Repeatability of temperature realized by cell	see Wt scatter	0,210	0,711	0,108	0,050	0,017	0,754	
	Short repeatability of calibrated SPRT	see Wt scatter	1,100	0,253	2,529	0,190	0,202	1,384	
$C_{0.01°C/1}$	Uncertainty linked with purity and isotopic composition	0,130	0,210	0,257	0,253	0,050	0,003	0,301	
$C_{0.01°C/2}$	Uncertainty linked Hydrostatic pressure correction	0,010	0,009	0,031	0,018	0,005	0,004	0,493	
$C_{0.01°C/3}$	Uncertainty linked with perturbing heat exchanges	0,040	0,090	0,214	0,202	0,010	0,038	0,301	
$C_{0.01°C/4}$	Uncertainty linked with self-heating correction	0,050	0,170	0,253	0,202	0,030	0,202	0,048	
$C_{0.01°C/5}$	Uncertainty linked with bridge linearity	0,340	0,170	0,247		0,025	0,014	0,391	
$C_{0.01°C/6}$	Uncertainty linked with AC/DC current			not estimated		0,000	0,050	1,204	
$C_{0.01°C/7}$	Uncertainty linked with internal insulation leakage	0,010		not estimated	0,051	0,000	0,067	0,752	
$D_{RS/1}$	Uncertainty linked with stability of RS	0,120	0,040	0,009		0,001	0,011	0,602	
$D_{RS/2}$	Uncertainty linked with temperature of RS	0,170	0,430	0,064	0,003	0,001	0,231	0,174	
S_{Wt}	Wt scatter	1,640	0,800	0,121	2,450	3,375	3,180	0,619	
Combined uncertainty		**1,905**	**2,569**	**1,754**	**3,743**	**3,468**	**3,569**	**3,200**	
Expanded uncertainty		**3,810**	**5,137**	**3,507**	**7,486**	**6,935**	**6,994**	**6,400**	

Uncertainty analysis

Quantity Q_i	Components	Uncertainty contribution ui in mK							
		Loop C HTSPRT 1065							
		PTB	SMU	NPL	SP	UJ-PFI	PTB	VNIIM	PTB
X_t	Repeatability of readings	see Wt scatter	see Wt scatter	0,044	0,100	0,200	0,473		see Wt scatter
$C_{Xt/1}$	Uncertainty linked with purity	0,650	0.45	0,577	2,309	4,000	1,122	0,670	0,650
$C_{Xt/2}$	Uncertainty linked Hydrostatic pressure correction	0,110	0.018	0,031	0,031	0,016	0,031	0,016	0,110
$C_{Xt/3}$	Uncertainty linked with perturbing heat exchanges	0,500	0.19	0,433	1,732	0,289	0,333	0,300	0,500
$C_{Xt/4}$	Uncertainty linked with self-heating correction	0,010	0.10	0,006	0,115	0,066	0,750	0,013	0,010
$C_{Xt/5}$	Uncertainty linked with bridge linearity	0,080	0.10	0,041	0,043	0,242	0,087		0,080
$C_{Xt/6}$	Uncertainty linked with AC/DC current			0,000	0,115		0,050		
$C_{Xt/7}$	Uncertainty linked with gas pressure	0,240	0.060	0,045	0,035	0,300	0,104	0,008	0,240
$X_{0.01\ °C}$	Repeatability of readings	see Wt scatter	see Wt scatter	0,114	0,214	0,129	0,262		see Wt scatter
	Repeatability of temperature realized by cell	see Wt scatter	see Wt scatter	0,000	0,247	0,257	0,017		see Wt scatter
	Short repeatability of calibrated SPRT	see Wt scatter	see Wt scatter	0,000	0,247	0,619	0,166		see Wt scatter
$C_{0.01°C/1}$	Uncertainty linked with purity and isotopic composition	0,130	0.24	0,175	0,247	0,429	0,003	0,015	0,130
$C_{0.01°C/2}$	Uncertainty linked Hydrostatic pressure correction	0,010	0.010	0,024	0,049	0,005	0,004	0,006	0,010
$C_{0.01°C/3}$	Uncertainty linked with perturbing heat exchanges	0,040	0.81	0,072	0,124	0,066	0,038	0,043	0,040
$C_{0.01°C/4}$	Uncertainty linked with self-heating correction	0,050	0,770	0,048	0,247	0,285	0,166	0,040	0,050
$C_{0.01°C/5}$	Uncertainty linked with bridge linearity	0,340	0.086	0,175	0,186	0,124	0,014		0,340
$C_{0.01°C/6}$	Uncertainty linked with AC/DC current			0,000	0,247		0,050		
$C_{0.01°C/7}$	Uncertainty linked with internal insulation leakage	0,010		0,000	2,475		0,067		0,010
$D_{RS/1}$	Uncertainty linked with stability of RS	0,120	0.0043	0,000	0,007		0,011		0,120
$D_{RS/2}$	Uncertainty linked with temperature of RS	0,170	0.0039	0,037	0,010	0,017	0,231	0,01	0,170
S_{Wt}	Wt scatter	1,070	0,500	1,660	0,924	0,268	8,000	0,740	11,020
Combined uncertainty		**1,440**	**1,350**	**1,835**	**3,969**	**4,145**	**8,060**	**1,040**	**11,070**
Expanded uncertainty		**2,880**	**2,700**	**3,669**	**7,939**	**8,290**	**16,090**	**2,890**	**22,130**

Uncertainty analysis

Quantity Q_i	Components	Uncertainty contribution ui in mK							
		Loop C			**HTSPRT 944RS13**				
		PTB	SMU	NPL	SP	UJ-PFI	PTB	VNIIM	PTB
X_t	Repeatability of readings	see Wt scatter	see Wt scatter	0,039	0,150	0,200	0,473		see Wt scatter
$C_{Xt/1}$	Uncertainty linked with purity	0,650	0.45	0,577	2,309	4,000	1,122	0,670	0,650
$C_{Xt/2}$	Uncertainty linked Hydrostatic pressure correction	0,110	0.018	0,031	0,031	0,016	0,031	0,016	0,110
$C_{Xt/3}$	Uncertainty linked with perturbing heat exchanges	0,500	0.19	0,433	1,732	0,664	0,333	0,300	0,500
$C_{Xt/4}$	Uncertainty linked with self-heating correction	0,010	0.10	0,006	0,115	0,061	0,750	0,013	0,010
$C_{Xt/5}$	Uncertainty linked with bridge linearity	0,080	0.10	0,041	0,043	0,233	0,087		0,080
$C_{Xt/6}$	Uncertainty linked with AC/DC current			0,000	0,115		0,050		
$C_{Xt/7}$	Uncertainty linked with gas pressure	0,240	0.060	0,045	0,035	0,300	0,104	0,008	0,240
$X_{0.01\,°C}$	Repeatability of readings	see Wt scatter	see Wt scatter	0,084	0,214	0,257	0,262		see Wt scatter
	Repeatability of temperature realized by cell	see Wt scatter	see Wt scatter	0,000	0,247	0,257	0,017		see Wt scatter
	Short repeatability of calibrated SPRT	see Wt scatter	see Wt scatter	0,000	0,742	0,619	0,166		see Wt scatter
$C_{0.01°C/1}$	Uncertainty linked with purity and isotopic composition	0,130	0.24	0,175	0,247	0,429	0,003	0,015	0,130
$C_{0.01°C/2}$	Uncertainty linked Hydrostatic pressure correction	0,010	0.010	0,024	0,049	0,005	0,004	0,006	0,010
$C_{0.01°C/3}$	Uncertainty linked with perturbing heat exchanges	0,040	0.81	0,072	0,124	0,153	0,038	0,043	0,040
$C_{0.01°C/4}$	Uncertainty linked with self-heating correction	0,050	0,086	0,078	0,247	0,334	0,166	0,040	0,050
$C_{0.01°C/5}$	Uncertainty linked with bridge linearity	0,340	0.086	0,175	0,186	0,124	0,014		0,340
$C_{0.01°C/6}$	Uncertainty linked with AC/DC current			0,000	0,247		0,050		
$C_{0.01°C/7}$	Uncertainty linked with internal insulation leakage	0,010		0,000	2,475		0,067		0,010
$D_{RS/1}$	Uncertainty linked with stability of RS	0,120	0.0043	0,000	0,007		0,011		0,120
$D_{RS/2}$	Uncertainty linked with temperature of RS	0,170	0.0039	0,037	0,010	0,017	0,231	0,010	0,170
S_{Wt}	Wt scatter	2,060	1,310	3,220	1,039	0,779	9,920	1,130	18,140
Combined uncertainty		**2,280**	**1,640**	**3,313**	**4,060**	**4,263**	**9,970**	**1,350**	**18,170**
Expanded uncertainty		**4,450**	**3,290**	**6,626**	**8,120**	**8,525**	**19,930**	**8,770**	**36,340**

Uncertainty analysis

Quantity Q_i	Components	Uncertainty contribution ui in mK							
		Loop D HTSPRT 1041							
		PTB	MIKES	EIM	INRiM	IPQ	CEM	VSL	PTB
X_t	Repeatability of readings	see Wt scatter	0,973	0,211	2,266	0,123	0,070	0,277	see Wt scatter
$C_{Xt/1}$	Uncertainty linked with purity	0,650	4,000	3,600	0,650	4,000	2,889	0,648	0,650
$C_{Xt/2}$	Uncertainty linked Hydrostatic pressure correction	0,110	0,001	0,031	0,015	0,031	0,054	0,031	0,110
$C_{Xt/3}$	Uncertainty linked with perturbing heat exchanges	0,500	0,800	0,289	0,290	0,022	2,889	0,300	0,500
$C_{Xt/4}$	Uncertainty linked with self-heating correction	0,010	0,046	0,231	0,015	0,014	0,049	0,555	0,010
$C_{Xt/5}$	Uncertainty linked with bridge linearity	0,080	0,146	0,013	0,001	0,007	0,163	0,208	0,080
$C_{Xt/6}$	Uncertainty linked with AC/DC current		0,050	0,000	0,001		0,558	0,000	
$C_{Xt/7}$	Uncertainty linked with gas pressure	0,240	0,001	0,300	0,040		1,445	0,002	0,240
$X_{0.01\ °C}$	Repeatability of readings	see Wt scatter	0,095	-0,121	0,249	0,069	0,060	0,423	see Wt scatter
	Repeatability of temperature realized by cell	see Wt scatter	0,100	-0,602	0,013	0,050	2,414	0,009	see Wt scatter
	Short repeatability of calibrated SPRT	see Wt scatter	0,027	-0,521	---		2,258	0,423	see Wt scatter
$C_{0.01°C/1}$	Uncertainty linked with purity and isotopic composition	0,130	0,100	-0,602	0,014	0,100	0,199	0,150	0,130
$C_{0.01°C/2}$	Uncertainty linked Hydrostatic pressure correction	0,010	-0,001	0,013	0,100	0,001	0,022	0,009	0,010
$C_{0.01°C/3}$	Uncertainty linked with perturbing heat exchanges	0,040	0,020	-0,021	0,100	0,022	0,036	0,051	0,040
$C_{0.01°C/4}$	Uncertainty linked with self-heating correction	0,050	0,009	-0,104	0,600	0,000	0,209	1,396	0,050
$C_{0.01°C/5}$	Uncertainty linked with bridge linearity	0,340	0,011	0,334	0,001	0,002	0,697	0,634	0,340
$C_{0.01°C/6}$	Uncertainty linked with AC/DC current		0,040	0,000	0,001		0,880	0,000	
$C_{0.01°C/7}$	Uncertainty linked with internal insulation leakage	0,010	0,437	-0,043	0,100		0,000	0,000	0,010
$D_{RS/1}$	Uncertainty linked with stability of RS	0,120	0,140	0,015	0,100	0,081	0,000	0,000	0,120
$D_{RS/2}$	Uncertainty linked with temperature of RS	0,170	0,032	0,152	0,100	0,004	0,000	0,000	0,170
S_{Wt}	Wt scatter	3,530	2,779	2,464	---	0,118	3,395	1,584	13,280
Combined uncertainty		**3,660**	**5,057**	**4,643**	**2,473**	**4,007**	**7,483**	**2,486**	**13,310**
Expanded uncertainty		**7,330**	**10,115**	**9,609**	**4,947**	**8,014**	**15,414**	**5,420**	**26,630**

Uncertainty analysis

Quantity Q_i	Components	Uncertainty contribution ui in mK							
		Loop D		**HTSPRT 1043**					
		PTB	MIKES	EIM	INRiM	IPQ	CEM	VSL	PTB
X_t	Repeatability of readings	see Wt scatter	0,320	0,226	1,237	0,118	0,084	0,352	see Wt scatter
$C_{Xt/1}$	Uncertainty linked with purity	0,650	4,000	3,600	0,650	4,000	2,889	0,648	0,650
$C_{Xt/2}$	Uncertainty linked Hydrostatic pressure correction	0,110	0,001	0,031	0,015	0,031	0,054	0,031	0,110
$C_{Xt/3}$	Uncertainty linked with perturbing heat exchanges	0,500	0,800	0,173	0,290	0,149	2,889	0,300	0,500
$C_{Xt/4}$	Uncertainty linked with self-heating correction	0,010	0,000	0,061	0,015	0,011	0,049	0,493	0,010
$C_{Xt/5}$	Uncertainty linked with bridge linearity	0,080	0,146	0,011	0,001	0,007	0,163	0,208	0,080
$C_{Xt/6}$	Uncertainty linked with AC/DC current		0,050	0,000	0,001		0,410	0,000	
$C_{Xt/7}$	Uncertainty linked with gas pressure	0,240	0,001	0,300	0,040		1,445	0,002	0,240
$X_{0.01\,°C}$	Repeatability of readings	see Wt scatter	0,090	-0,136	0,289	0,106	0,139	0,387	see Wt scatter
	Repeatability of temperature realized by cell	see Wt scatter	0,100	-0,602	0,013	0,711	2,414	0,009	see Wt scatter
	Short repeatability of calibrated SPRT	see Wt scatter	0,242	-0,608	---		3,474	0,387	see Wt scatter
$C_{0.01°C/1}$	Uncertainty linked with purity and isotopic composition	0,130	0,100	-0,602	0,014	0,100	0,199	0,150	0,130
$C_{0.01°C/2}$	Uncertainty linked Hydrostatic pressure correction	0,010	-0,001	0,013	0,100	0,001	0,022	0,009	0,010
$C_{0.01°C/3}$	Uncertainty linked with perturbing heat exchanges	0,040	0,020	-0,021	0,100	0,022	0,036	0,051	0,040
$C_{0.01°C/4}$	Uncertainty linked with self-heating correction	0,050	0,011	-0,261	0,600	0,000	0,209	0,223	0,050
$C_{0.01°C/5}$	Uncertainty linked with bridge linearity	0,340	0,011	0,341	0,001	0,002	0,697	0,644	0,340
$C_{0.01°C/6}$	Uncertainty linked with AC/DC current		0,040	0,000	0,001		1,237	0,000	
$C_{0.01°C/7}$	Uncertainty linked with internal insulation leakage	0,010	0,437	-0,043	0,100		0,000	0,000	0,010
$D_{RS/1}$	Uncertainty linked with stability of RS	0,120	0,140	0,015	0,100	0,081	0,000	0,000	0,120
$D_{RS/2}$	Uncertainty linked with temperature of RS	0,170	0,032	0,152	0,100	0,004	0,000	0,000	0,170
S_{Wt}	Wt scatter	2,710	2,605	0,704	---	3,026	2,449	0,950	5,400
Combined uncertainty		**2,880**	**4,884**	**4,006**	**1,591**	**5,072**	**7,620**	**1,617**	**5,490**
Expanded uncertainty		**5,760**	**9,768**	**8,021**	**3,182**	**10,144**	**15,697**	**3,411**	**10,970**

Appendix E: Drift compensation applying Matthiessen's rule

Instability of HTSPRTs is caused mainly by two reasons: mechanical stress of the sensor and poisoning of the sensor by impurities. Metallic and other impurities can diffuse at high temperatures (for instance the freezing temperature of Ag) through the thermometer sheath and into the sensor material.

It is known for a long time that the change in the resistance of metals due to impurities or stress follows a simple rule and is described in many text books: The resistance change ΔR is constant and does not depend on temperature (Matthiessen's rule or Mattiessen-Nernst-rule). The reason is that scattering of the electron at lattice defects is not temperature dependent, while scattering at phonons is temperature dependent. This rule allows a compensation of the drift: the change in resistance can easily be derived from the resistance measured at the TPW. It is known from the measurements with SPRTs at the Al freezing point that all participants of EUROMET 820 agree in the measurement at the TPW by better than 1 mK. It therefore may be assumed that the resistance measured at the TPW can be used for a compensation also for HTSPRTs.

Although the drift compensation method for SPRTs is known [6], it is only seldom applied. The reasons may be as follows:

- Whenever you have a drift of a thermometer, something is going wrong and you should not use the thermometer anymore.
- The resistance at the TPW is due to oxidation / reduction effects dependent on the history of the thermometer and therefore not a good reference.

These arguments are true, but in the case of high drifts during an interlaboratory comparison it may be worthwhile to consider a compensation of the drift of the thermometer applying Matthiessens's rule.

The compensation is simple:

$$W = \frac{R(t)}{R(TPW)} \rightarrow W^* = \frac{R(t)+\Delta R}{R(TPW)+\Delta R}$$

or

$$\Delta W = W^* - W \approx \frac{\Delta R(H_2O)}{R(H_2O)}(1-W)$$

The uncertainty of the compensation can be estimated [7] to be

$$u(\Delta W) = \frac{u(\Delta R)}{R(H_2O)}(1-W)$$

The uncertainty $u(\Delta \mathrm{R})$ due to the scatter of the measurements is already included in the uncertainties of W. So the additional uncertainty of the correction is mainly the uncertainty of the resistance measurement. The information about this component is not fully available from all participants, but it is clear that the relative uncertainty of the resistance measurement is of the order of 10^{-6} corresponding to a temperature uncertainty of 0,3 mK. Without going into detail the uncertainty $u(\Delta \mathrm{W})$ of the compensation is therefore estimated to be 1 mK (k = 1).

Appendix F: Comments on the stability of Pt-25 thermometers

It has been noticed that all SPRTs (Pt-25) used in EUROMET 820 showed a shift in the same direction: The *W*-value for the final PTB measurement was higher by an equivalent of appr. 4 mK for all thermometers. It is very strange that for the thermometers with SN 1445, 1446 and 1450 the increase is nearly identical (4,3 mK). It is therefore worthwhile to check if the reason may be a shift in the PTB standards. A possible explanation may be the replacement of the Al fixed point cell due to a damage of the first cell.

The replacement of the cell was controlled by a check thermometer and by comparison with a third cell. There is a difference in the fixed point temperature of the cell, but this difference was corrected. The check thermometer does not give any indication for a drift of the reference cell. Therefore there is a high probability that there really was a parallel drift of all 4 thermometers. This may be possible, as all thermometers are new and from the same production batch. The history of the thermometers may be better understood from the changes in *R(*TPW), W and *W**, where *W** is the *W*-value corrected for a possible drift as described in Appendix E.

Fig. F1a: *R*(TPW) for SPRT SN 1444

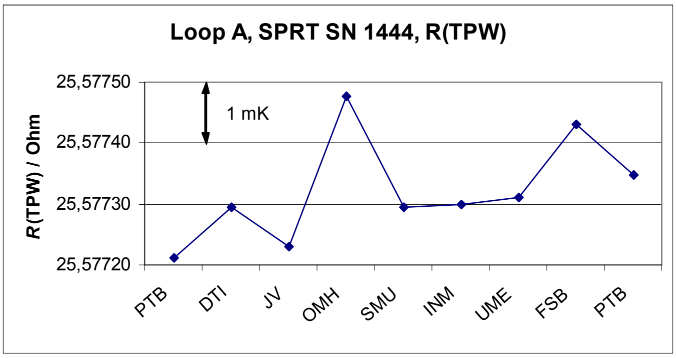
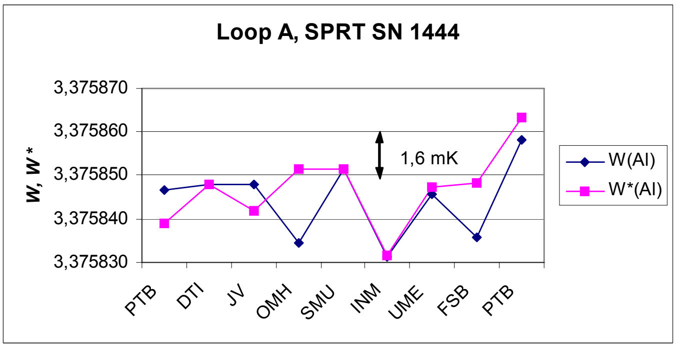


Fig. F1B: *W* and *W** for SPRT SN 1444



SPRT SN 1444: The largest drift of the thermometer occurred between the last 2 NMIs (FSB and PTB).

Fig. F2a: *R*(TPW) for SPRT SN 1445

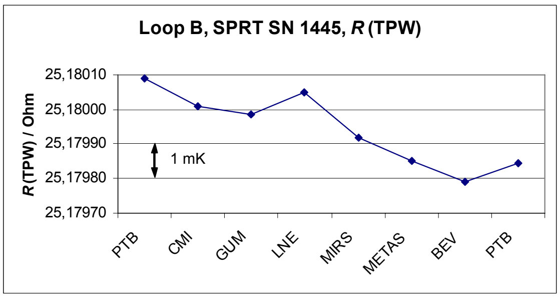
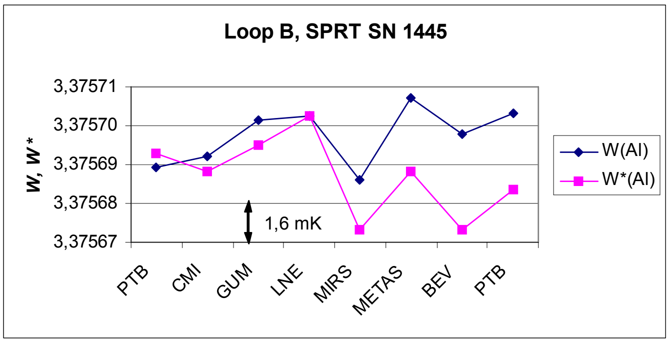


Fig. F2b: *W* and *W** for SPRT SN 1445



SPRT SN 1445: There is a continuous drift of the thermometer as can be seen from *R*(TPW) and to a smaller amount also from *W*(Al). The drift can be partly (over)compen-sated according to Matthiesen’s rule. For *W**(Al) there is no increase, but a decrease between the initial and final measurements of PTB.

Fig. F3a: *R*(TPW) for SPRT SN 1445

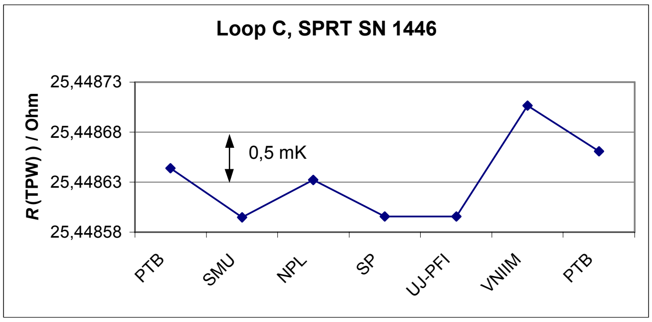
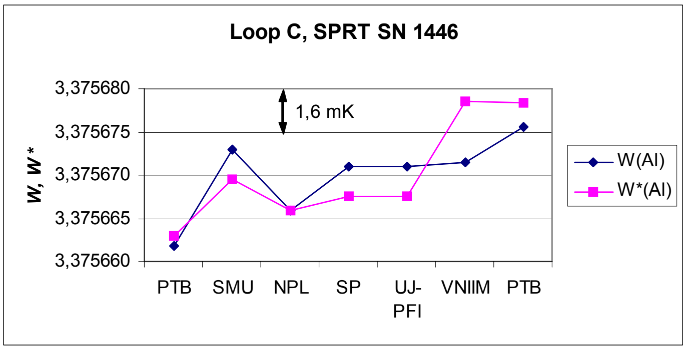


Fig. F3B: *W* and *W** for SPRT SN 1446



SPRT SN 1446: There is a jump between the measurements at UJ-PFI and VNIIM. The thermometers was transported from UJ-PFI to VNIIM via PTB, but unfortunately no measurements were made at PTB.

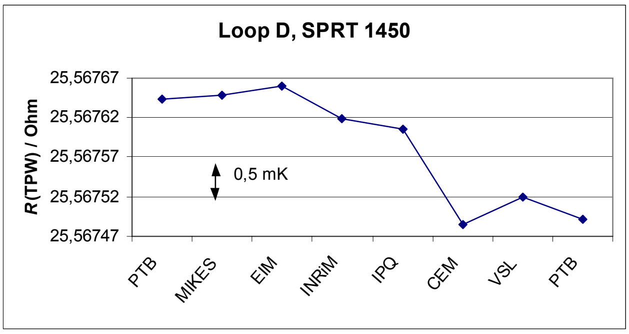


Fig. F4a: *R*(TPW) for SPRT SN 1445

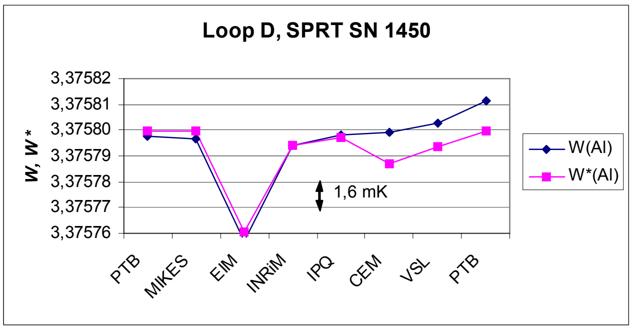


Fig. F4B: *W* and *W** for SPRT SN 1450

SPRT SN 1450: The deviation in the measurement of EIM seems to be due to problems with measurements at the Al fixed points. There is a continuous shift of the thermometers after the measurements at EIM, which can be fully compensated using Matthiesens's rule. The decrease of *R*(TPW) seems to indicate an annealing of lattice defects, but there is not enough information for a full explanation of what is happening.

It can be concluded that the drift of the thermometers is different for the 4 thermometers. The more or less identical drift of the thermometers between the initial and the final measurement at PTB is therefore probably accidental.

It should be noted that the deviation for the measurement of the TPW between PTB and the sub-pilots is in all cases smaller than 1 mK. Therefore within this uncertainty the NMIs are equivalent.

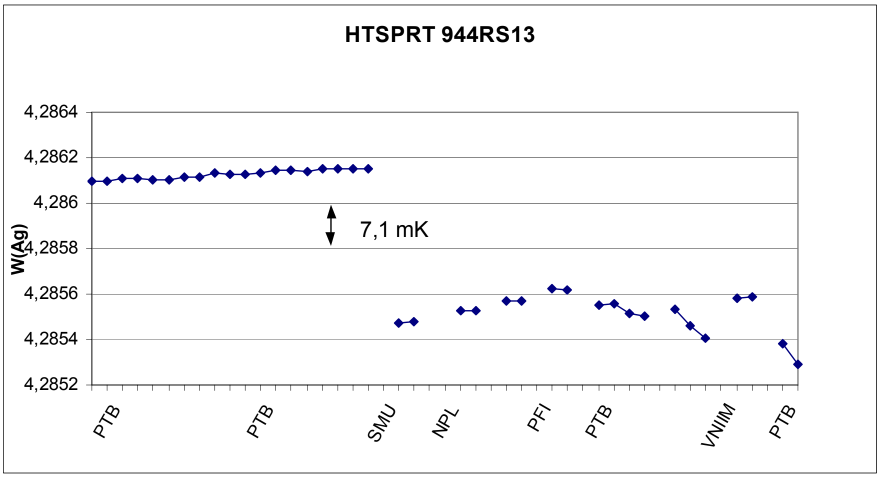
Appendix G: Comments on the stability of the HTSPRTs

The HTSPRTs used for EUROMET showed in most cases an insufficient stability. The reason for this instability is not clear in all cases: it may be diffusion of impurities, mechanical stress or other reasons. Looking for the behaviour in detail may give some information about the reason for the instability.

This analysis will be done exemplary for the HTSPRT SN 944RS13. The thermometer was produced by Chino and provided by MIKES.

Detailed results are given in the following figures, this time not for the average values, but for the individual results.

Fig. G1: *W*(Ag) for HTSPRT SN 944RS13. Data points are no averages.



There is already a small drift noticed for the initial measurements at PTB. The next 2 figures gives results for *R(*TPW) and *R(*Ag) for the initial measurements at PTB.

Fig. G2: *R*(TPW) for the initial measurements at PTB

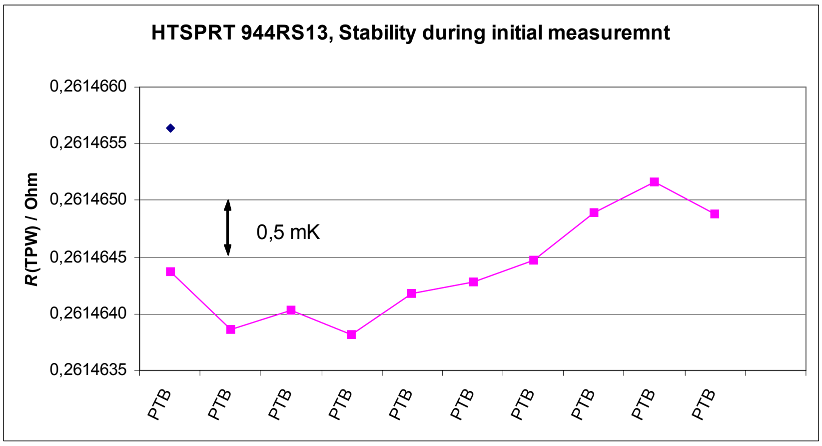
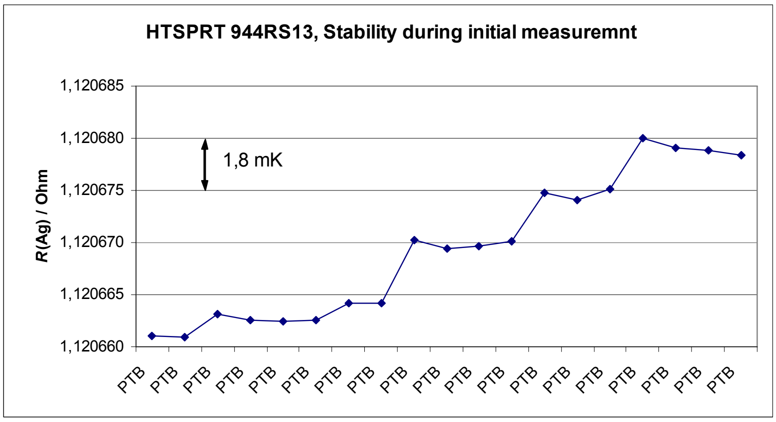


Fig. G3: *R*(Ag) for the initial measurements at PTB



The drift of *R(*TPW) was only about 1 mK, while for *R(*Ag) it was about 7 mK. The measurements were performed at 2 different Ag freezing point cells, and the thermometer was not cooled down between the measurements. The steps in *R(*Ag) occurred when the thermometer was operated at the Al freezing point or was annealed in a furnace.

During the measurements at PTB a steady increase of *W*(Ag) was noticed. This is not unusual for quite new thermometers, while the effect of poisoning by impurities is just in the opposite direction.

Between the measurements at PTB and SMU a large jump occurred.

Fig. G4: Jump in *R(*TPW) between the measurements at PTB and SMU

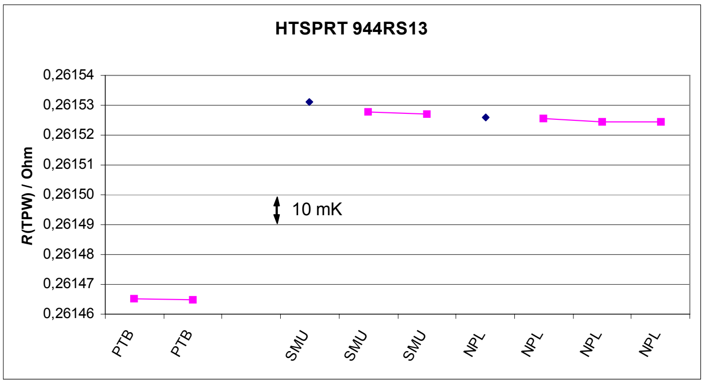
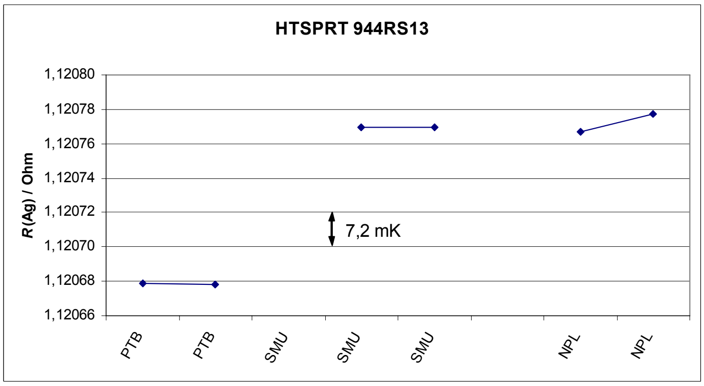


Fig. G4: Jump in *R(*Ag) between the measurements at PTB and SMU



The jump between PTB and SMU occurred in *R*(TPW) and *R*(Ag), but the effects do not cancel. They cancel better (not perfectly) when a drift compensation is applied (Fig. G5). So there is a high probability that that mechanical stress was the reason for the jump. It is not clear how this happened: the thermometer was handcarried from PTB to SMU, and did not fall to the floor, and no similar affairs are known.

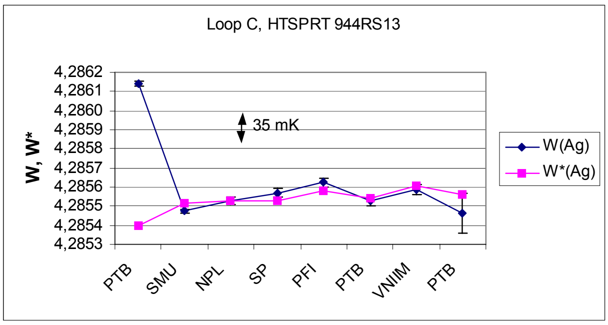


Fig. G5

After the measurements at SMU an increasing drift for the thermometer was noticed, for *R*(TPW) as well as for *R*(Ag) (see Fig. G6 and G7). Nevertheless, there is a different behaviour for *R*(TPW) and *R*(Ag). The effect may be caused by impurities.

Fig. G6: *R*(TPW) for the measurements after the jumps

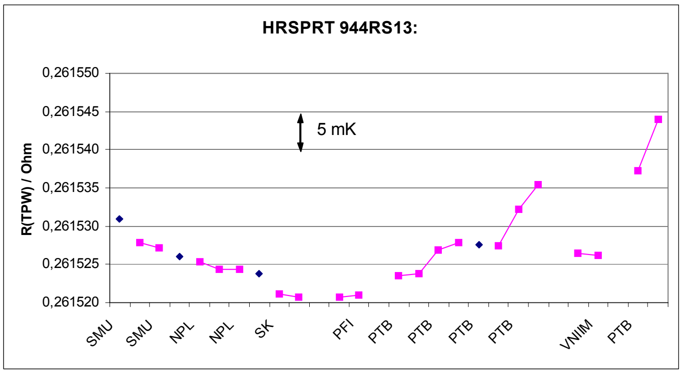
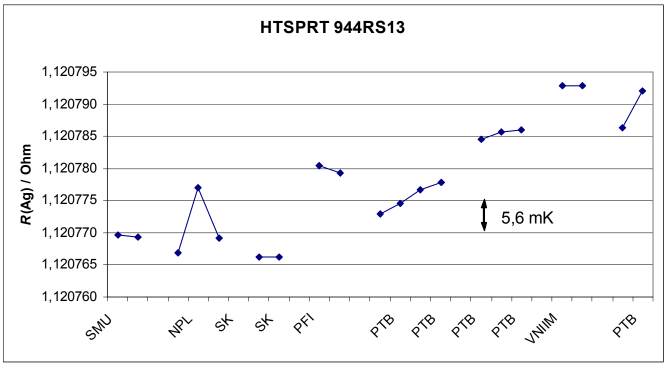


Fig. G6: *R*(Ag) for the measurements after the jumps



For HTSPRT also *W*(Al) was measured during the initial and final measurements at PTB. Also for *W*(Al) an enormous drift to smaller values was found, similar as for *W*(Ag). This may support the assumption of an increasing poisoning of the thermometer by impurities.