

Intercomparison of the Realization of the ITS-90 at the Freezing Points of Al and Ag among European NMIs

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Abstract The EUROMET.T-K4 comparison is the regional extension of CCT-K4, an intercomparison of the realizations of the freezing points of Al (660.323°C) and

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Ag (961.78°C). The intercomparison was organized in four loops. Long-stem standard platinum resistance thermometers (SPRTs) were used as traveling standards: 25 Ω thermometers to be used only at the Al freezing point and two high-temperature standard platinum resistance thermometers (HTSPRTs) to be used only at the Ag freezing point in each loop. Parallel to the measurements with thermometers, the pilots and sub-pilots organized an internal intercomparison using an Ag fixed-point cell. Most HTSPRTs showed a strong drift which is mainly due to mechanical stress and poisoning of the sensor by impurities. This drift can be partially compensated by a correction based on Matthiessen's rule. An evaluation of the data taking into account both HTSPRTs in each loop, the linkage of the sub-pilots by measurements at the Ag freezing point, and a possible compensation according to Matthiessen's rule, allows calculation of the results of the participants' measurements at the Ag freezing point. The results of the participating laboratories are summarized, and proposals for key comparison reference values and linking of the results to CCT-K3 and CCT-K4 are presented.

Keywords Al freezing point · Ag freezing point · CCT · EUROMET · Fixed points · High-temperature standard platinum resistance thermometers · Key comparison · Matthiessen's rule

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1 Introduction

The Mutual Recognition Arrangement of the International Committee of weights and Measures (CIPM) [1] requires that the equivalence between national metrology institutes (NMIs) be supported by interlaboratory comparisons. At the highest level, such comparisons in the field of thermometry are organized by the Consultative Committee for Thermometry (CCT) of the CIPM. These comparisons have to be repeated as regional comparisons to include all NMIs that could not participate in the CCT key comparisons. Within the regional organization EUROMET, until now only EUROMET.T-K3 [2] was organized as an analog to CCT-K3, using standard platinum resistance thermometers (Pt-25) as traveling standards. While CCT-K3 covered the range from the triple point of argon to the freezing point of aluminum, EUROMET.T-K3 did not include the Al freezing point.

CCT-K4 [3] was an intercomparison of Al and Ag freezing-point cells. Due to problems with the transportation of fixed-point cells, EUROMET.T-K4 was organized as a comparison of SPRTs at the freezing points of Al (660.323°C) and Ag (961.78°C). Together with EUROMET.T-K3, the full temperature range covered by CCT-K3 and CCT-K4 is now also covered by EUROMET.T-K3 and T-K4.

The measurements of EUROMET.T-K4 are finished, and draft A report has been sent by the pilot laboratory to the participants. This report is still under discussion. The CIPM MRA requires that the results of key comparisons be kept unattributable to specific participants until the report is accepted as the so-called draft B. Therefore, this article mainly concerns the organization of the comparison and the method to evaluate the data; the results for the NMIs are presented only in anonymous form. All data given in this article are preliminary. The full report with the individual results will be published in the Technical Supplement of Metrologia.

2 Organization of EUROMET.T-K4

The EUROMET regional key comparison EUROMET.T-K4 (accepted by EUROMET as project EUROMET 820) was initiated during the EUROMET TC THERM meeting on March 30/31, 2004 in Ljubljana, Slovenia. PTB was chosen to be the pilot laboratory. 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 of two high temperature standard platinum resistance thermometers (HTSPRTs) at the freezing point of Ag by each participant. Participants interested in a comparison at the Al freezing point only, calibrated the SPRT (25 Ω).

During the preliminary characterization of the HTSPRTs by the pilot laboratory, it was found that the thermometers were less stable than desired. Several improvements to the protocol were considered and (mostly) rejected, because the effort required would have been too large. As a compromise, one additional Ag fixed-point cell (provided by NMI/VSL) was compared between the pilot and sub-pilot laboratories. This allows for better control of the thermometer stability and allows the linking of the loops to be based on more reliable measurements.

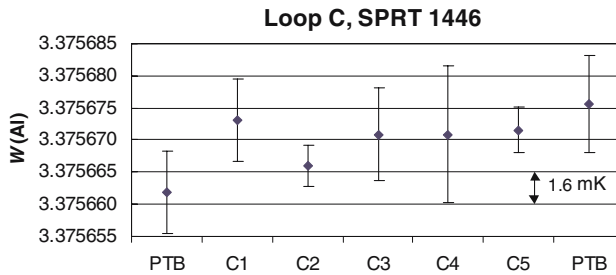


Fig. 1 Results of the participants in loop C for the calibration of a SPRT at the freezing point of Al. Uncertainties are given for $k=2$

The comparison involves the five EUROMET NMIs that participated in CCT-K4 [BNM-INM (FR), INRiM (IT), NMi/VSL (NL), NPL (GB), PTB (DE)] as pilot or sub-pilots and nearly all additional European national laboratories (see list of authors). The comparison is divided into four loops. Besides PTB, there is another participant of CCT-K4 in each of the loops of EUROMET.T-K4. A total of 24 NMIs participated in the comparison. Six of these NMIs measured only at the Al freezing point.

The pilot laboratory calibrated a total of seven HTSPRTs and five SPRTs, including one spare thermometer of each type. The thermometers were generously provided by Hart Scientific (three HTSPRTs and four SPRTs), BIPM (three HTSPRTs), MIKES (two HTSPRTs), METAS (one HTSPRT), and PTB (one SPRT). Some additional thermometers were rejected as insufficiently stable. Unfortunately, the selected HTSPRTs were still less stable than desired.

The project began in December 2004, and the measurements were completed in July 2006.

3 Results at the Freezing Point of Aluminum

The comparison at the freezing point of Al was carried out in four different loops. The first and the last measurements were performed by the pilot laboratory PTB. As an example, the results for loop C are presented in Fig. 1. Only the average values of at least three realizations of the Al freezing points are given. The uncertainties were estimated by the participants.

The results presented in Fig. 1 are typical of the measurements at the freezing point of Al. Uncertainties reported by the participants vary between 1.5 and 12 mK, with the majority between 2.5 and 4 mK. As known from CCT-K3, uncertainties below 2 mK are quite ambitious, and it is therefore not surprising that not all measurements agree within their uncertainties. It was found in all loops that the final measurement at PTB was higher than the initial measurement by 3–4 mK. Careful checking has disclosed no evidence of a shift in the standards of PTB, so it is therefore assumed that the drift of the thermometers is responsible for the limitation in closing the measurement loop. This is strange, but not entirely unlikely, because all thermometers were new and from the same batch.

Table 1 Possible *ERVs* and their uncertainty for the freezing point of Al

	$(ERV - T_{\text{mean}}(\text{PTB}))$ (mK)	
	Value (mK)	U (mK) ($k=2$)
Mean	-1.180	1.03 (0.99)
Weighted mean	-0.865	0.795
Median	-1.295	0.636

3.1 Linking the Loops at the Al Freezing Point and the ERV

The procedure for linking the loops in EUROMET.T-K4 follows a similar method used for EUROMET.T-K3 (EUROMET 552). In the report of EUROMET 552 [2], all formulae are given in detail, and they will not be repeated in full here.

The pilot (PTB) is the only laboratory with measurements in all loops. Therefore, results will be given relative to the PTB measurements. The reference for each loop is the simple mean of the first and the last measurements at PTB. All differences in resistance ratio (W) are converted to differences in temperature using the reference function of the ITS-90.

The uncertainty of the temperature difference with respect to PTB is given by

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

where $U_{T_{\text{Lab}}}$ is the uncertainty reported by the participants, $u_{\text{rep}(\text{PTB})}$ is the reproducibility of the PTB measurements, $u_{\text{inst}(\text{PTB})}$ is caused by changes in the instrumentation of PTB between the initial and final measurements, and the contribution u_{Stab} describing the instability of the thermometers has been calculated from the initial and final calibration at PTB according to

$$u_{\text{Stab}} = \frac{|W_{\text{final}} - W_{\text{initial}}|}{2\sqrt{3}} \left(\frac{dT}{dW}\right)_{\text{Al}}$$

The designation of the EUROMET Reference Value (*ERV*) for EUROMET.T-K4 follows the procedure proposed in EUROMET 552: the *ERV* is calculated from the differences between all laboratories and the pilot PTB. Three possible values are considered: the simple mean, the weighted mean, and the median calculated from the data of all participants (excepting one potential outlier). The possible *ERVs* are presented in Table 1 relative to the result of the pilot laboratory, PTB. For the simple mean, two uncertainties are given; the first calculated from the standard deviation of the results, the second (in brackets) calculated from the uncertainty of the measurement results. The possible *ERVs* are close together and agree within their uncertainties. The simple mean is suggested as the *ERV*, with its uncertainty calculated from the uncertainties of the participants.

Figure 2 presents the deviations of all participants from the *ERV*. The uncertainty of this deviation was calculated according to Cox [4], and includes the uncertainty of the measurements at the NMIs and the uncertainty in linking the loops. The participants

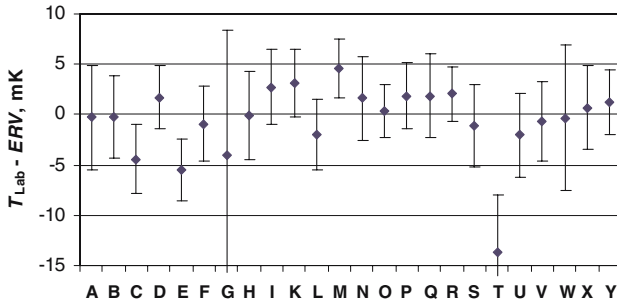


Fig. 2 Deviation of the results of the participating laboratories from the *ERV* at the freezing point of Al. Uncertainties are given for $k=2$

Table 2 Al freezing point: $(T_{Lab} - ERV)$ or $(T_{Lab} - KCRV)$ in mK for different interlaboratory comparisons. Uncertainties are given for $k=2$

Laboratory	EUROMET.T-K4	CCT-K3	CCT-K4
Average (INRIM, LNE, PTB, SMU, VNIIM, VSL)	1.12 ± 1.42	0.58 ± 1.33	
Average (INRIM, LNE, NPL, PTB, VNIIM)	0.90 ± 1.74		-0.65 ± 1.16

For CCT-K3, the “unofficial” ARV was used

are labeled with letters from A to Y. Participant T seems to be an outlier; its data were not used for the calculation of the *ERV*.

3.2 Linkage between EUROMET.T-K4 and CCT-K3 and CCT-K4

The linkage of EUROMET.T-K4 to CCT-K3 and CCT-K4 is made via those NMIs that 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 relative to the reference values is caused by a difference in the reference values themselves. Table 2 gives an overview for the relevant key comparisons.

The standard deviation of the results of the participating NMIs was used as the uncertainty of the deviation of the group of laboratories from the reference values of the comparisons. This seems to be too optimistic, in particular for linking EUROMET.T-K4 to CCT-K4.

The linking of the comparisons is straightforward and follows immediately from Table 2:

$$ERV(Al, E.T-K4) = ARV(Al, CCT-K3) - (0.54 \pm 1.96) \text{ mK} \quad (k = 2)$$

$$ERV(Al, E.T-K) = KCRV(Al, CCT-K4) + (1.55 \pm 2.09) \text{ mK} \quad (k = 2)$$

Table 3 Results for the comparison of the Ag freezing point cell

Laboratory	$(T_{\text{Lab}} - T_{\text{PTB}})$ (mK)	U ($k = 2$) (mK)
Sub-pilot B	-0.46	1.71
Sub-pilot C	-2.34	2.06
Sub-pilot D	0.59	1.15

The agreement between EUROMET.T-K4 and CCT-K3 is slightly better than between EUROMET.T-K4 and CCT-K4. Nevertheless, within the uncertainties of EUROMET.T-K4, the *KCRVs* for Al in CCT-K3 and CCT-K4 are consistent.

4 Results for Measurements at the Ag Freezing Point

4.1 Intercomparison with a Ag Freezing-point Cell

An additional interlaboratory comparison between the pilot and the sub-pilots was organized for the freezing point of silver, which basically followed the protocol of CCT-K4. The traveling instrument was a re-sealable Ag freezing-point cell provided by NMi-VSL. During transportation, the crucible containing the silver ingot was removed from the assembly to minimize the risk of damage. The participating NMIs measured the temperature difference between their cell used in EUROMET.T-K4 and the traveling cell of NMi-VSL.

To evaluate the HTSPRT measurement, the differences between the sub-pilots and PTB must be calculated. For the estimation of the uncertainty, a contribution for the stability of the traveling cell was estimated from the difference between two measurements at NMi-VSL:

$$u_{\text{Stab}} = \frac{|T_{\text{NMi1}} - T_{\text{NMi2}}|}{2\sqrt{3}} = 0.28 \text{ mK}$$

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

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

The results are given in Table 3. These values will be used to link the measurements of the sub-pilots with the HTSPRTs to the corresponding measurements at PTB.

4.2 Intercomparison with HTSPRTs: Loop Results

The comparison at the freezing point of Ag was carried out in three different loops. The first and the last measurements were performed by the pilot laboratory, PTB. In each loop, a sub-pilot that participated in CCT-K4 was included. Following the procedure applied to the freezing point of Al, the loops will be linked using the measurements of PTB. Because the sub-pilots are linked to PTB by the comparison with the Ag freezing

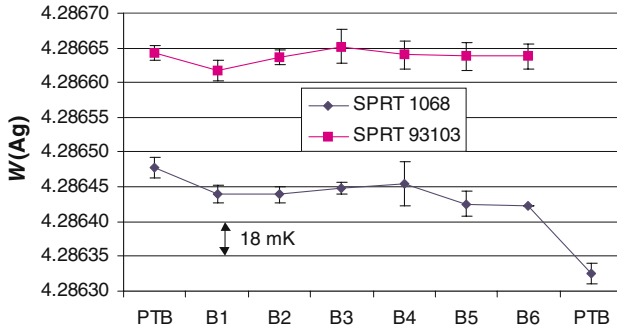


Fig. 3 $W(\text{Ag})$ for both thermometers by the laboratories in loop B. The difference in the absolute value of W is not relevant. Uncertainties are given for $k = 2$

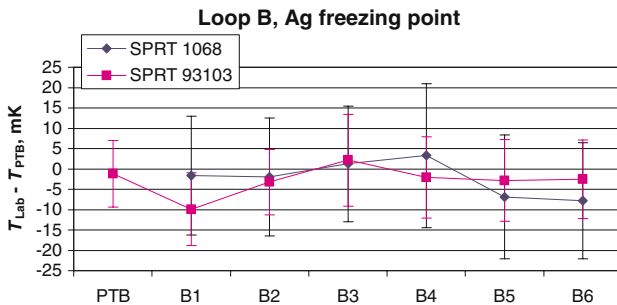


Fig. 4 Results of the NMIs for both HTSPRTs of loop B relative to the PTB measurements. Uncertainties are given for $k = 2$

point cell, the measurements of the sub-pilots can also be used to link the loops, if their values are corrected according to their deviation from PTB.

As an example, the procedure is described in some detail for loop B. The results for both HTSPRTs in this loop are presented in Fig. 3. For HTSPRT S/N 1068, a mechanical shock or similar event seems to have occurred between the measurements of PTB and participant B1. When the thermometer came back to PTB, the sensor was contaminated. Therefore, both PTB measurements will be ignored and only the measurement of the sub-pilot will be used to link the loops and the thermometers.

HTSPRT SN 93103 was the most stable of all thermometers in EUROMET.T-K4. Unfortunately, it was destroyed at the end of the measurements at participant B6. Therefore, no final measurement at PTB could be made. To link the thermometers and loops, the average of the first PTB measurement and the result of sub-pilot B (corrected for deviation from PTB) will be used.

For both thermometers, the deviation from the PTB measurement is calculated. This is just the difference in $W(\text{Ag})$, converted to a temperature difference using the relation given by the reference function of the ITS-90. Therefore, both thermometers of the loop can be combined as shown in Fig. 4. The results for both HTSPRTs are then summarized by calculating the weighted mean for $(T_{\text{Lab}} - T_{\text{PTB}})$ of both thermometers.

4.3 Compensation of the Drift of the Thermometers

Due to the strong drift, an individual evaluation of the data for each thermometer may be necessary. Here, only some examples for the evaluation procedure are given; the full procedure will be described in the final report on EUROMET.T-K4.

It is known that the instability of HTSPRTs is caused mainly by two mechanisms: mechanical stress of the sensor and poisoning of the sensor by impurities. Metallic and other impurities can diffuse at high temperatures (for instance, at 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 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.

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

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

The drift of a HTSPRT caused by contamination can be partly compensated [5]. Basically, the change in the resistance is only slightly temperature-dependent (Matthiessen's rule). By comparing the resistance $R(\text{TPW})$ at the triple point of water measured by the laboratory with the resistance measured by the pilot or sub-pilot, compensation can be applied:

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

The shift ΔR in the resistance can be easily determined from the measured resistance $R(\text{TPW})$ at the triple point of water. It was found for the measurements at the Al freezing point that all measurements at the TPW agreed within 1 mK or better. The reference value for $R(\text{TPW})$ is more or less arbitrary and will only lead to the same shift for all compensated W^* . For convenience, the measurements of the sub-pilots were chosen for reference, because these measurements are in the middle of the loops.

The effect of such compensation was calculated for all HTSPRTs in the comparison. As an example, in Fig. 5 the effect of the compensation is shown for HTSPRT S/N 1041 in loop D. For this thermometer, the compensated W^* -values will be used for further evaluation. The change between the initial PTB measurement and the participant D1 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 will also be ignored. The reference

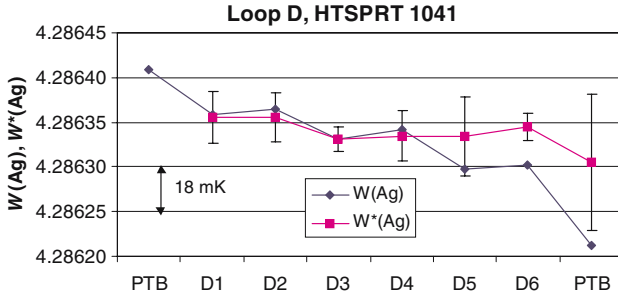


Fig. 5 Original W and compensated W^* . The uncertainty ($k=2$) is only given for W^*

Table 4 Possible *ERV*s and their uncertainty for the freezing point of Ag

	$(ERV - T_{\text{mean}}(\text{PTB}))$ (mK)	
	Value (mK)	U ($k=2$) (mK)
Mean	-0.52	2.04 (1.30)
Weighted mean	-2.50	1.84
Median	-0.66	1.06

value to link loop D to the other loops is calculated from the measurement of the sub-pilot D3 (corrected to the PTB value with the results from the comparison of the Ag fixed-point cells).

4.4 Linking of the Loops for the Ag Freezing Point and *ERV*

The procedure for linking the loops and calculating the *ERV* is similar to the procedure used for the measurements at the Al freezing point: all measurements will be given as differences with respect to the PTB measurement, because only the pilot PTB participated in all loops. The *ERV* is then calculated from the deviation of the results of the participants from the PTB measurements. Three possible values for the *ERV* are shown in Table 4. For the simple mean, two uncertainty values are given, the first calculated from the standard deviation of the results, the second (in brackets) calculated from the uncertainty of the measurement results.

The simple mean of all participants is suggested as the *ERV*:

$$ERV(\text{Ag, E.T-K4}) = T_{\text{mean}}(\text{PTB}) - (0.43 \pm 2.18) \text{ mK} \quad (k = 2)$$

The deviation of all participants from the *ERV* is shown in Fig. 6. The uncertainty of the deviation was calculated in the same way as for the freezing point of Al.

4.5 Linkage between EUROMET.T-K4 and CCT-K4

The linkage between EUROMET.T-K4 and CCT-K4 at the freezing point of Ag follows the same procedure as for the Al freezing point. Table 5 gives the data of those NMIs

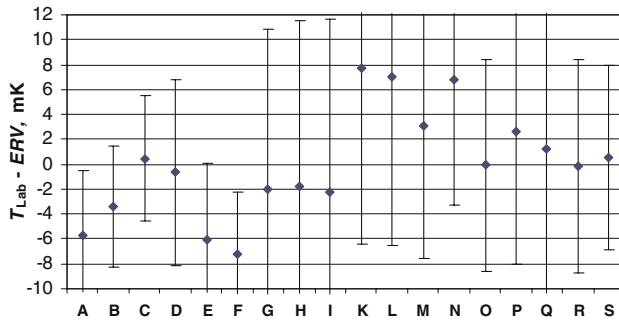


Fig. 6 Deviation of the participants from the *ERV* for the Ag freezing point. Uncertainties are given for $k=2$

Table 5 Ag freezing point: ($T_{\text{Lab}}-ERV$) or ($T_{\text{Lab}}-KCRV$) in mK for different interlaboratory comparisons

Laboratory	EUROMET.T-K4	CCT-K4
Mean (INRiM, LNE, NPL, PTB)	-0.24 ± 1.09	-1.12 ± 1.28

Uncertainties are given for $k=2$

that participated in EUROMET.T-K4 and in CCT-K4. For the calculation of the mean values, the VNIIM and NMI-VSL are neglected because their results for EUROMET.T-K4 or CCT-K4 may be outliers. From Table 5 it follows immediately:

$$ERV(\text{Ag, E-T-K4}) = KCRV(\text{CCT-K4}) - (0.88 \pm 1.68) \text{ mK} \quad (k = 2)$$

5 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 traveling Ag freezing-point cell, results were achieved which can be used in part to scrutinize the CMC entries delivered by the participants. Nevertheless, the uncertainties are still higher than anticipated prior to 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 minimize poisoning of the thermometers.
- In a future comparison, 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 traveling cell were much better than the results with the HTSPRTs. It seems that the risk of damage to such a cell is acceptable. Further comparisons at the freezing point of Ag should be considered with such a cell.

- HTSPRTs are used in NMIs for the interpolation of the temperature scale, but not in the industry. It should be discussed (by the CCT) whether HTSPRTs are suitable transfer standards. Au/Pt thermocouples may, in the end, allow measurements with smaller uncertainties than HTSPRTs. Moreover, some statistical information about the number of calibrations of HTSPRTs (but only those performed for clients) should be gathered to discuss if the enormous effort of EUROMET.T-K4 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 justified.

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