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Intercomparison of the Realizations of the ITS-90 from 83.8058 K to 692.677 K among European NMIs

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Abstract The EUROMET.T-K3 comparison is the regional extension of CCT-K3. The comparison involved the six European national metrology institutes (NMIs) previously involved in CCT-K3 (LNE-INM/CNAM, SMU, INRiM, NMi-VSL, NPL, PTB) and 18 additional European national laboratories. The comparison was divided into five different loops, each coordinated by a co-pilot chosen from the laboratories having participated in the CCT-K3 comparison. LNE-INM/CNAM played the role of pilot in linking the five loops. In each loop, an artifact in the form of a standard platinum resistance thermometer (SPRT, 25 Ω) was circulated among the participating

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laboratories. To have sufficient information about the possible drift of the SPRTs, the co-pilots performed a calibration over the full temperature range at the beginning and at the end of the loop. A EUROMET reference value (ERV), taking into account the whole comparison, was defined, and the differences ($T_{\mathrm{Lab}} - T_{\mathrm{ERV}}$) were calculated with the associated uncertainties. The method for establishing the link between the participants in CCT-K3 and in EUROMET.T-K3 is described.

Keywords Argon · Bilateral equivalence · Comparison · EUROMET · Gallium · Indium · ITS-90 · Mercury · SPRT · Tin · Zinc

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1 Introduction

The Mutual Recognition Arrangement of the International Committee of Weights and Measures (CIPM) [1] requires that the equivalence of national metrology institutes (NMIs) be supported by interlaboratory comparisons. EUROMET Project 552 was initiated in March 2000. It was defined as the comparison of the European local realizations of the International Temperature Scale of 1990 (ITS-90) from the triple point of argon (83.8058 K) to the freezing point of zinc (692.677 K) using long-stem standard platinum resistance thermometers (SPRTs). This project was intended to be the Regional Key Comparison corresponding to the Consultative Committee for Thermometry Key Comparison 3 (CCT-K3) [2]. CCT-K3 covered the range from the triple point of argon to the freezing point of aluminum. EUROMET.T-K3 did not include the aluminum freezing point to limit the effect of the SPRT's instability.

The technical comparison protocol (together with an amendment) was sent to the participating laboratories and to the CCT Working Group 7 (WG7) chairman in May 2001. These documents received the approval of all participants. EUROMET Project 552 was agreed by the CCT-WG7 as a regional key comparison corresponding to CCT-K3 and was named EUROMET.T-K3.

The full report with the individual results and the associated uncertainties has been published in a Technical Supplement of Metrologia [3]. Consequently, this article presents only a summary of the results and the method employed to establish the link with CCT-K3.

2 Organization of EUROMET.T-K3

The comparison involved 24 European NMIs. It was divided into five loops, each one coordinated by a co-pilot chosen from the laboratories that had participated in CCT-K3 (BNM-INM/CNAM, SMU, IMGC, NMi-VSL, NPL, PTB). BNM-INM/CNAM played the role of pilot in linking the five loops. The schedule of the comparison is given below.

Loop 1: co-pilot SMU, participants: BNM-INM/CNAM, OMH.
Loop 2: co-pilot INRiM, participants: BNM-INM/CNAM, CEM, METAS, BEV, MIRS/FE-LMK, IPQ.
Loop 3: co-pilot NMI-VSL, participants: BNM-INM/CNAM, MIKES, DTI, SP, VMT/PFI.
Loop 4: co-pilot PTB, participants: BNM-INM/CNAM, GUM, CMI, INM, UME.
Loop 5: co-pilot NPL, participants: BNM-INM/CNAM, JV, EIM, SMD, NML.

In preparation for the comparison, the co-pilots selected and calibrated two SPRTs. One of these SPRTs was sent to BNM-INM/CNAM for calibration. BNM-INM/CNAM received the five circulating SPRTs in Spring 2001 and calibrated them. The SPRTs were returned to the co-pilots, who then determined again the reduced resistance values at the gallium and zinc points to check the SPRT stability. Unfortunately, substantial differences were observed at the zinc point for two SPRTs. Due to practical difficulties, and not to unduly delay the comparison, two new SPRTs were calibrated in two different runs. The BNM-INM/CNAM 1825320 thermometer was included in the three calibration runs.

3 Uncertainties

Each participating laboratory provided resistance ratios measured in their local fixed points. Each participant was requested to supply the uncertainty budget associated with the calibration at the different points and to complete the "Uncertainty Excel file" that was included in the protocol and agreed by all the participants. Table 1 lists the components and the range of the values of these components as given by the participants for the calibration at the zinc fixed point. The combined uncertainties were computed by the root-sum-of-squares of the types A and B contributions. For all the laboratories, a coverage factor $k = 2$ was used to calculate the combined expanded uncertainties. Whatever the component, the values by the participants are remarkably different. Nevertheless, the combined standard uncertainties seem consistent.

3.1 Additional Uncertainties

In addition to the uncertainties reported by each laboratory, an uncertainty for possible instabilities of the circulated SPRT over the course of the comparison must be taken into account. The measurements at BNM-INM/CNAM link the five loops. Nevertheless, the five circulated SPRTs were not calibrated during the same run at BNM-INM/CNAM, so an additional uncertainty associated with the repeatability of BNM-INM/CNAM calibration has to be added.

3.1.1 Uncertainty Due to the Instability of the Circulated SPRTs

The co-pilot calibrations of the traveling instrument carried out during the time of the loop are used to establish the uncertainty component associated with the stability of the traveling SPRT. For the SPRT and the fixed point considered, the uncertainty u_{stabSPRT_j} is calculated using a type B method and the hypothesis of a rectangular asymmetrical distribution;

$$u_{\text{stabSPRT}_j} = \frac{\left|\left(W_{\text{CP}_j}\right)_{\text{end}} - \left(W_{\text{CP}_j}\right)_{\text{beginning}}\right|}{\sqrt{3}} \times \left(\frac{\delta T}{\delta W}\right)_T \tag{1}$$

with W_{CP_j}, being the value of W measured by the co-pilot of the loop $j\,(j = 1, \ldots, 5)$

3.1.2 Uncertainty Due to the Repeatability of the BNM-INM/CNAM Calibration

As some SPRTs were not stable, the five circulating SPRTs were not calibrated in the same run at BNM-INM/CNAM (three runs were needed). The BNM-INM/CNAM's

Table 1 List of the components included in the uncertainty budget

Fixed point	Component	Range of values Min/Max (μK)
Zinc	Repeatability of readings	6/200
	Uncertainty linked with purity of fixed-point metal	100/710
	Uncertainty linked with hydrostatic pressure correction	5/39
	Uncertainty linked with perturbing heat exchanges	3/460
	Uncertainty linked with SPRT self-heating correction	2/344
	Uncertainty linked with resistance bridge linearity	1/400
	Uncertainty linked with AC/DC current	Negligible/200
	Uncertainty linked with gas pressure	1/981
TPW[a]	Repeatability of readings	5/211
	Repeatability of temperature realized by cell	20/642
	Short-term repeatability of calibrated SPRT	20/560
	Uncertainty linked with purity and isotopic composition	6/337
	Uncertainty linked with hydrostatic pressure correction	1/40
	Uncertainty linked with perturbing heat exchanges	5/76
	Uncertainty linked with SPRT self-heating correction	1/134
	Uncertainty linked with resistance bridge linearity	2/340
	Uncertainty linked with AC/DC current	Negligible/90
	Uncertainty linked with internal insulation leakage	Negligible/40
	Uncertainty linked with stability of standard resistor	Negligible/367
	Uncertainty linked with temperature of standard resistor	Negligible/367
Wt scatter		20/906
	Combined standard uncertainty	430/1,800

[a] Triple point of water

SPRT 1825320 was calibrated during each run, so it is possible to estimate the repeatability of the BNM-INM/CNAM calibration at these fixed points. The additional uncertainty due to the repeatability of the BNM-INM/CNAM calibration is calculated from

$$u_{\text{reproducibility}T_{\text{p}}} = \frac{|(W_{1825320})_{\max} - (W_{1825320})_{\min}|}{2\sqrt{3}} \times \left(\frac{\delta T}{\delta W}\right)_T \qquad (2)$$

with the hypothesis of a rectangular symmetrical distribution. It appears that this repeatability is very good (from (40 to 110) μK depending to the fixed point considered) and does not introduce a significant uncertainty component.

4 EUROMET Reference Value

From the beginning, it was expected that a EUROMET reference value would be established based on the data from all the European participant laboratories to conform to the CCT-K3 data processing and thereby follow the MRA rules.

Let $T_{\mathrm{Lab}_{ij}}$ be the value measured by lab i in loop j, where $i = 1, \ldots, N_j$ lab index in loop j, $j = 1, \ldots, 5$ loop index, N_j = number of labs in loop j, and $i = 1$ corresponds to the co-pilot of the loop. So, $T_{\mathrm{Lab}_{1j}} = T_{\mathrm{CP}_j}$ with T_{CP_j} being the value measured by the co-pilot (CP_j) on SPRT j.

$i = 2$ corresponds to the second laboratory of the loop, which was the comparison pilot BNM-INM/CNAM, so $T_{\mathrm{Lab}_{2j}} = T_{\mathrm{P}_j}$ with T_{P_j} being the value measured by the pilot on SPRT j.

As BNM-INM/CNAM was involved in all loops, it is possible to calculate the differences $T_{\mathrm{Lab}_{ij}} - T_{\mathrm{P}_j}$:

$$T_{\mathrm{Lab}_{ij}} - T_{\mathrm{P}_j} = \left(W_{\mathrm{Lab}_{ij}} - W_{\mathrm{P}_j}\right) \left(\frac{\delta T}{\delta W}\right)_T \tag{3}$$

It is expected that, taking the uncertainty associated with the repeatability of the BNM-INM/CNAM calibration into account,

$T_{\mathrm{P}_j} = T_{\mathrm{P}}$ (same cell, same furnace, same bridge, …).

$$T_{\mathrm{Lab}_{ij}} - T_{\mathrm{P}_j} = T_{\mathrm{Lab}_{ij}} - T_{\mathrm{P}} \tag{4}$$

4.1 Designation of the EUROMET Reference Value (ERV)

Three possible reference values were considered: the simple mean, $T_{\mathrm{m}552}$, the weighted mean, $T_{\mathrm{wm}552}$, and the median, $T_{\mathrm{med}552}$. The differences among these values, obtained from a classical statistical analysis, are very small. The lowest uncertainty is associated with the median. Nevertheless, the participant laboratories decided by vote during a workshop to be conservative and to adopt the weighted mean, $T_{\mathrm{wm}552}$, at each fixed point as the EUROMET Reference Value.

4.2 ($T_{\mathrm{Lab}_{ij}} - T_{\mathrm{wm}552}$) Computation

The value of $T_{\mathrm{wm}552}$ is not known a priori and has no physical meaning, but the difference between a laboratory calibration value and the reference value can be computed. For example,

$$\left(T_{\mathrm{Lab}_{ij}} - T_{\mathrm{wm}552}\right) = \left(T_{\mathrm{Lab}_{ij}} - T_{\mathrm{P}}\right) - \left(T_{\mathrm{wm}552} - T_{\mathrm{P}}\right) \tag{5}$$

The value of T_{P} of the pilot (BNM-INM/CNAM) cancels out and does not affect the result of the comparison, assuming that the BNM-INM/CNAM value is stable during the period of the comparison. However, T_P is the link between all the laboratories and its uncertainty is therefore included in the uncertainties of all the inter-laboratory differences.

The W-values of the co-pilots are the means of the W-values measured at the beginning and at the end of the loop;

$$T_{\mathrm{CP}_j} = \frac{\left(T_{\mathrm{CP}_j}\right)_{\mathrm{beginning}} + \left(T_{\mathrm{CP}_j}\right)_{\mathrm{end}}}{2} \tag{6}$$

The uncertainty of $\left(T_{\mathrm{Lab}_{ij}} - T_{\mathrm{wm552}}\right)$ is obtained by:

$$u_{\left(T_{\mathrm{Lab}_{ij}} - T_{\mathrm{wm552}}\right)} = \sqrt{u^2_{\left(T_{\mathrm{Lab}_{ij}} - T_\mathrm{P}\right)} + u^2_{(T_{\mathrm{wm552}} - T_\mathrm{P})}} \tag{7}$$

The uncertainty associated with $T_{\mathrm{Lab}_{ij}} - T_\mathrm{P}$ is obtained from:

- the uncertainty of the measurement data supplied by Lab_{ij},
- the uncertainty associated with the stability of the circulating SPRT during the period of the loop measurements (depends on the loop), and
- the repeatability of the BNM-INM/CNAM calibration is

$$u_{(T_{\mathrm{Lab}_{ij}} - T_\mathrm{P})} = \sqrt{u^2_{\mathrm{Lab}_{ij}} + u^2_{\mathrm{stabSPR}T_j} + u^2_{\mathrm{reproducibility}T\mathrm{p}}} \tag{8}$$

All the significant components are obtained from a type B evaluation. These components were assumed to have an infinite number of degrees of freedom. Finally, the expanded uncertainties are computed by using a coverage factor of two, $U = 2u$.

The details of the results are not presented in this article. The histogram of the results for each fixed point is given in Figs. 1–6. The value of $(T_{\mathrm{Lab}_j} - T_{\mathrm{wm552}})$ is generally less than $U_{(T_{\mathrm{Lab}_{ij}} - T_{\mathrm{wm552}})}$, even when $(T_{\mathrm{Lab}_j} - T_{\mathrm{wm552}})$ is substantial. If this is not the case, the result of the participating laboratory is considered an outlier.

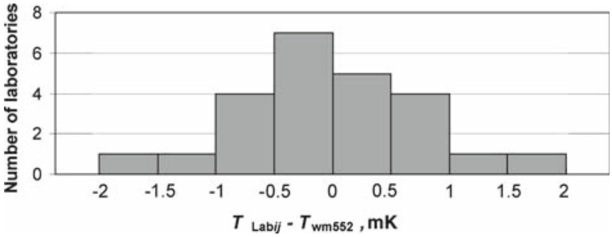

Fig. 1 Histogram associated with the zinc fixed point (24 participants): no outliers

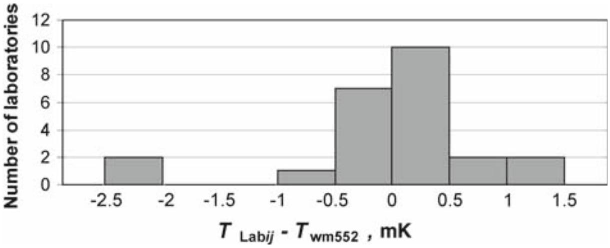

Fig. 2 Histogram associated with the tin fixed point (24 participants): one outlier

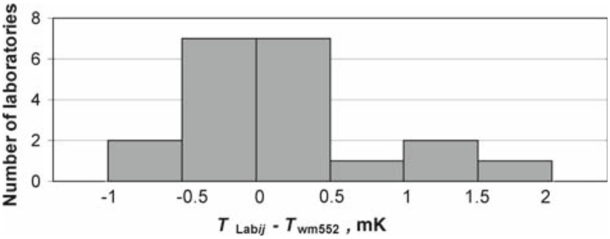

Fig. 3 Histogram associated with the indium fixed point (21 participants): two outliers, one laboratory (not shown): −3.9 mK ± 4.9 mK

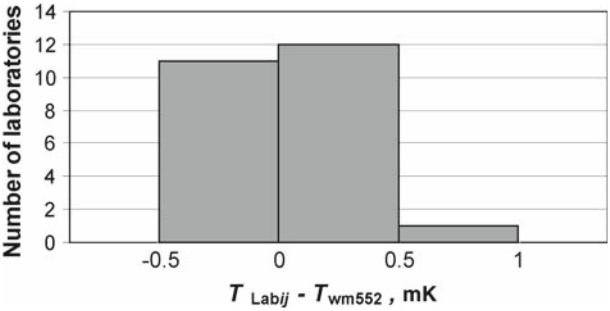

Fig. 4 Histogram associated with the gallium fixed point (24 participants): no outliers

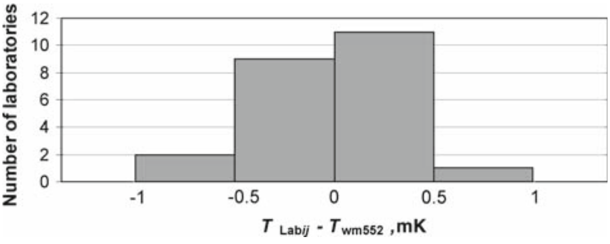

Fig. 5 Histogram associated with the mercury fixed point (24 participants): no outliers, one laboratory (not shown): +2.7 mK ± 3.1 mK

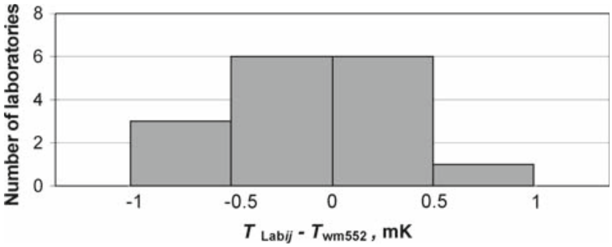

Fig. 6 Histogram associated with the argon fixed point (16 participants): two outliers

5 Linkage Between EUROMET 552 and CCT-K3

The pilot and the co-pilot laboratories have been used to link EUROMET 552 to the CCT-K3 average reference value, *ARV-K3*. The hypothesis is that the mean temperature of the pilot and co-pilot laboratories is the same in EUROMET 552 as it was in CCT-K3:

$$\left(\frac{T_{\text{BNM-INM}}+T_{\text{PTB}}+T_{\text{IMGC}}+T_{\text{NPL}}+T_{\text{NMi}}+T_{\text{SMU}}}{6}\right)_{\text{CCT-K3}} = \left(\frac{T_{\text{BNM-INM}}+T_{\text{PTB}}+T_{\text{IMGC}}+T_{\text{NPL}}+T_{\text{NMi}}+T_{\text{SMU}}}{6}\right)_{\text{EUROMET552}} \quad (9)$$

It has been shown [3] that even when the results given by one or several members of the “linking laboratories” are not exactly the same in EUROMET 552 and CCT-K3, the mean calculated for the group is highly reproducible.

The difference between $(ARV-K3-T_{(\text{P\&CP}j)\text{mean}})$ and $(T_{(\text{wm552})}-T_{(\text{P\&CP}j)\text{mean}})$ is generally equal to or smaller than 0.1 mK except at the zinc (0.23 mK) and tin (0.14 mK) fixed points.

6 Conclusion

EUROMET Project 552 compares the various European local realizations of the ITS-90 from the triple point of argon (83.8058 K) to the freezing point of zinc (692.677 K) using long-stem SPRTs. This project was agreed by the CCT-WG7 as a Regional Key Comparison corresponding to CCT-K3 and was named EUROMET.T-K3. Given that the protocol of the comparison contains a detailed description of how the uncertainties are to be calculated, the uncertainty budgets established by the participants seem consistent, or at least homogeneous.

A EUROMET reference value taking into account the whole comparison was defined for each fixed point.

The results of the measurements at all the considered fixed points (Ar, Hg, Ga, In, Sn, Zn) are satisfactory. It is surprising to note that the worst results are associated with the fixed point of indium, a metal that one easily finds with a high purity. The laboratories were probably too optimistic when they evaluated the uncertainty associated with the realization of this fixed point. The degree of equivalence resulting from this comparison can be used to validate the CMCs presented by the participating laboratories.

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