RMO Key Comparison EURAMET.EM-K2.1
Comparison of Resistance Standards at 10 MΩ and 1 GΩ

Final Report

Beat Jeckelmann[1], Hani Saad Abdel Aziz[2], Edyta Dudek[3], Damir Ilic[4], Ivan Lenicek[4], Nadia Nassif Tadros[2], Marcin Orzepowski[3], Andrey Tenev[5]

[1]Federal Institute of Metrology METAS, Lindenweg 50, 3003 Bern-Wabern, Switzerland
beat.jeckelmann@metas.ch

[2]National Institute for Standards (NIS), Egypt
[3]Central Office of Measures (GUM), Poland
[4]Croatian Metrology Institute - Primary Electromagnetic Laboratory (HMI/FER-PEL), Croatia
[5]Bulgarian Institute of Metrology (BIM), Bulgaria

Abstract

Four National Metrology Institutes, among them three EURAMET members, participated in the follow-up comparison EURAMET.EM-K2.1. The comparison aimed at evaluating the degrees of equivalence of the measurements of 10 MΩ and 1 GΩ resistance standards. Through the pilot laboratory, the results are linked to comparisons EUROMET.EM-K2 and CCEM-K2 respectively. At 1 GΩ, all results supplied by the participants agreed with the comparison reference value within the expanded uncertainty. At 10 MΩ, a slight disagreement with the KCRV for three of the four participants was observed.

Table of Contents

Annexes

1. Introduction

After approval of the draft B report of the RMO key comparison EUROMET.EM-K2, it was decided to organise a follow-up comparison to allow new participants to join in and to allow some participants of EUROMET.EM-K2 to improve their results. The Federal Institute of Metrology METAS, already pilot laboratory and co-ordinator of EUROMET.EM-K2, coordinated this follow-up and assures the link to CCEM-K2.

The comparison protocol is essentially equivalent to the protocol of EUROMET.EM-K2. It was prepared following the CCEM guidelines for planning, organizing, conducting and reporting key, supplementary and pilot comparisons.

2. Participants and organisation of the comparison

2.1 Co-ordinator and members of the support group

The pilot laboratory for the comparison was the Federal Institute of Metrology (METAS).

Co-ordinator:

Dr Beat Jeckelmann
Tel.: +41 58 387 02 97; e-mail: beat.jeckelmann@metas.ch

Support group, appointed by the EURAMET technical committee for electricity and magnetism:

Dr Bernd Schumacher, Physikalisch-Technische Bundesanstalt (PTB), DE;
e-mail: bernd.schumacher@ptb.de

Dr Gert Rietveld, VSL, NL:
e-mail: grietveld@vsl.nl

2.2 List of participants

Five EURAMET NMIs and one non-EURAMET NMI participated in the comparison. IPQ performed measurements but withdrew afterwards from the comparison before the analysis of the comparison results was carried out.

No	Country	Institute	Acronym
1	Switzerland	Federal Institute of Metrology	METAS*)
2	Portugal	Portuguese Institute for Quality	IPQ**)
3	Poland	Central Office of Measures	GUM
4	Croatia	Croatian Metrology Institute - Primary Electromagnetic Laboratory	HMI/FER-PEL
5	Bulgaria	Bulgarian Institute of Metrology	BIM
6	Egypt	National Institute of Standards	NIS

Table 1: Participants
*) METAS participated in CCEM-K2 and EUROMET.EM-K2 and assures the link to the CCEM key comparison.
**) IPQ performed the measurements but withdrew from the comparison before the draft A report was issued.

2.3 Organisation and comparison schedule

The comparison was carried out in one measurement loop. The circulation of the standards started in March 2010 and was completed in February 2011. The detailed time schedule for the comparison is given in Table 2.

A period of four weeks was allowed for the measurements in each laboratory, including the time necessary for transportation. The standards were re-measured in the middle and at the end of the loop by the pilot laboratory to establish a drift rate for the standards and to detect resistance changes related to transport.

Loop A

No *p*	Institute	Country	Dates: arrival to dispatch of standards
1	**Pilot (METAS)**	Switzerland	
2	IPQ	Portugal	30 March to 3 May 2010
3	GUM	Poland	12 May to 10 June 2010
4	HMI/FER-PEL	Croatia	29 June to 16 July 2010
5	BIM	Bulgaria	23 July to 10 Sept 2010
	Pilot (METAS)	Switzerland	15 Sept to 1 Nov 2010
6	NIS	Egypt	23 Nov to 16 Feb 2011
	Pilot (METAS)	Switzerland	

Table 2: Comparison schedule

2.4 Unexpected incidents

No travel incidents or problems were reported by the participants. When the standards returned to the pilot in September 2010 after the first comparison sequence, it was realized that at least one of the 10 MΩ standards (Serial number 47225) must have been used in an oil bath by one of the participants. As a consequence, the resistance value measured for this standard was not as expected. The standard was opened, carefully cleaned and reassembled. This procedure induced another step-change in the value (see Sect. 5 below).

3. Travelling standard and measurement instructions

3.1 Description of the standards

10 MΩ

Two different types of travelling standards (one resistor each) were used:

1. MI 9331, SN 1050109
   Standard manufactured by Measurements International (CA), Model 9331. The resistance element is hermetically sealed in a metal container. The four resistor terminations of the standards are tellurium copper binding posts. A separate ground terminal is included for screening.
2. Guildline 9930, SN 47225
   Standard manufactured by Guildline Instruments, model 9330. The resistance element is suspended in oil in a hermetically sealed metal container. This container is mounted inside a metal box. The two resistor terminations of the standard are coaxial N-type connectors mounted on the top panel of the enclosure. The resistor container, the outer box and the shields of the coaxial N-connectors are joined together.

1 GΩ
Two travelling standards of the same type were used:

1. MI 9331S, SN 1010802 and MI 9331S, SN 1100036
   Standards manufactured by Measurements International (CA), Model 9331S (based on NIST design). The resistance elements are housed in a double shielded enclosure. The two resistor terminations of the standards are N-type coaxial connectors mounted directly on the outer enclosure. The inner enclosure containing the resistive element is connected to the guard terminal. For one of the standards, this terminal is isolated from the outer enclosure and may be operated either in floating mode, in a grounded mode, or driven at a guard potential. For the 2nd standard, the guard terminal is connected to the outer enclosure.

The standards 10 MΩ, SN 1050109, and 1 GΩ, SN 1100036, were already used in the comparison EUROMET.EM-K2. Their values were deliberately offset by means of trim resistors after this comparison. In this way, an extrapolation of their value based on the results of EUROMET.EM-K2 is not possible.

R	Std-ind. *a*	Standards
10 MΩ	1	MI 9331, SN 1050109
	2	Guildline 9330, SN 47225
1 GΩ	3	MI 9331S, SN 1010802
	4	MI 9331S, SN 1100036

Table 3: List of travelling standards

3.2 Quantities to be measured and conditions of measurement

- Resistance of the 10 MΩ standards at the following conditions:
  test voltage: $V_{test} \leq 100$ V; preferably 10 V
  ambient temperature: (23 ± 0.2) °C
  relative humidity: (50 ± 10) %

- Resistance of the 1 GΩ standards at the following conditions:
  test voltage: $V_{test} \leq 100$ V; preferably 100 V
  ambient temperature: (23 ± 0.2) °C
  relative humidity: (50 ± 10) %

3.3 Measurement instructions

Pre- conditioning: The standards were to be installed in a thermostatic air bath, regulated at the chosen working temperature, at least 24 h before starting the measurements.

Measurements: It was expected that the measurements would be repeated several times during the whole period allocated to the participating laboratory.

Method: The measurement method was not specified. It was assumed that every participant uses its normal measurement method. The method and the traceability

scheme had to be described in the measurement report.
The choice of the ground/guard configuration was left to the participants.

3.4 Deviations from the protocol

The comparison was carried out as described in the protocol. Except to the modifications in the comparison schedule, no adjustments of the protocol were necessary.

4. Methods of measurement

The following measurement methods and step-up procedures were applied by the participants:

METAS (see also [1])

- 10 MΩ: Potentiometric resistance bridge (MI 6000B). Reference standards up to 1 MΩ calibrated in terms of the quantized Hall resistance (QHR) using a cryogenic current comparator (CCC). Ratio accuracy of potentiometric bridge checked with Hamon devices up to 100 MΩ.
- 1 GΩ: Active arm Wheatstone bridge; reference standards at 1 MΩ, 10 MΩ and 100 MΩ calibrated with potentiometric system and CCC resp., traceable to QHR.

GUM

- 10 MΩ and 1 GΩ: Potentiometric resistance bridge (MI 6000B). Step-up from 100 Ω using the same bridge. 100 Ω standards calibrated against QHR using a CCC.

HMI/FER-PEL

- Active arm Wheatstone bridge. 1:1 comparison against reference standards calibrated at PTB

BIM-NCM

- 10 MΩ and 1 GΩ: Potentiometric resistance bridge (MI 6000B). 10:1 comparisons against 1 MΩ and 100 MΩ standards resp. The traceability is provided by calibration of 10 kΩ resistor SR 104, SN J1-0824605 at BIPM with Certificate No 78/19 Oct 2009. Step-up from 10 kΩ using the potentiometric resistance bridge (MI 6000B).

NIS

- 10 MΩ: Potentiometric resistance bridge (home-made). 1:1 comparisons against reference standard; step-up from 10 kΩ standard calibrated at BIPM using Hamon networks.

5. Repeated measurements of the pilot institute, behaviour of the travelling standards

5.1 Temperature and voltage dependence

Before starting the measurement loops, the temperature and voltage dependences of the travelling standards were determined at the pilot laboratory. The temperature was varied around 23 °C. The voltage was varied between 5 V and 90 V for the 10 MΩ standards and between 10 V and 1000 V for the 1 GΩ standards respectively.

The temperature (T) dependence around 23 °C and the voltage (V) dependence can be described by the following model:

$$R_a(T,V) = R_a(T_{nom}, V_{nom}) \cdot \left(1 + \alpha_a (T - T_{nom}) + \gamma_a (V - V_{nom})\right), \quad (5.1)$$

where a is the index for the standard.

The temperature coefficients (α) and the voltage coefficient (γ) were determined by a least-squares fit to the data. The fit results are listed in Table 4.

Standard Index a		T_{nom} (°C)	α_a (ppm/K)	V_{nom} (V)	γ_a (ppm/V)
10 MΩ					
1	1050109	23	0.74± 0.05	10	(-1.2 ± 1.0) 10^{-3}
2	47225		1.27 ± 0.04		(-1.2 ± 2.4) 10^{-4}
1 GΩ					
3	1010802	23	14.7± 3.3	100	(3.9 ± 1.0) 10^{-3}
4	1100036		1.2 ± 0.3		(2.9 ± 0.5) 10^{-3}

Table 4: Temperature and voltage coefficients of the travelling standards. The uncertainties are one-standard-deviations.

5.2 Drift behaviour of the standards

The measurements carried out at the pilot laboratory before starting the comparison, in the middle of the loop and at the end were used to establish the drift behaviour of the standards.

Due to relaxation effects in the metal used to fabricate a standard, its resistance value changes in time. Step-like resistance changes are observed after temperature shocks or mechanical shocks. After a long stabilization time and over short or medium-term time periods, a polynomial fit up to order two is usually sufficient to describe the resistance change over time.

Following these considerations, the following model was used to fit the measurements:

$$R_a(t) = R_{nom}\left(1 + p_{a,0} + p_{a,1}(t - t_0) + p_{a,2}(t - t_0)^2\right) = R_{nom}(1 + f(t)) \quad (5.2)$$

The reference date t_0 was chosen as 1 January 2010, 00:00 h. The fit results are listed in Table 5 and plotted in Figures 1 to 4. With one exception (10 MΩ standard no 2), the fit residuals are randomly distributed and the scatter around zero corresponds well with the type A standard deviation attributed to the individual measurement points.

For the 10 MΩ standard no 2, a rapid change of its value was observed by the pilot laboratory after the first part of the measurement loop (see Fig. 2a). The values were far off the expected drift line. In addition, it was realized that the standard must have been used in an oil bath (see Sect. 2.4) by one of the participants. As a consequence, the standard was opened and cleaned, and then assembled in a slightly modified way. The subsequent measurements by the pilot before and after the second part of the loop showed a satisfactory and stable behaviour of the standard after this operation (see Fig. 2b). As described in Sect. 6.2.4 below, the data for this standard measured in the *first part* of the loop (participants 3 to 5) were not used in the evaluation of the degrees of equivalence.

Standard Index *a*		$P_{a,0}$ (ppm)	$P_{a,1}$ (ppm/y)	$P_{a,2}$ (ppm/y^2)
10 MΩ				
1	1050109	29.605 ± 0.043	4.80 ± 0.12	-1.014 ± 0.062
2a	47225	237.14 ± 0.14	8.61 ± 1.12	0, fixed
2b	47225	107.07 ± 0.62	10.2 ± 1.2	-3.10 ± 0.54
1 GΩ				
3	1010802	-851.9 ± 0.4	2.44 ± 0.36	0, fixed
4	1100036	-0.57 ± 0.18	1.15 ± 0.22	0, fixed

Table 5: Fit parameters describing the drift behaviour of the travelling standards
Reference date t_0: 1 January 2010, 00:00 h

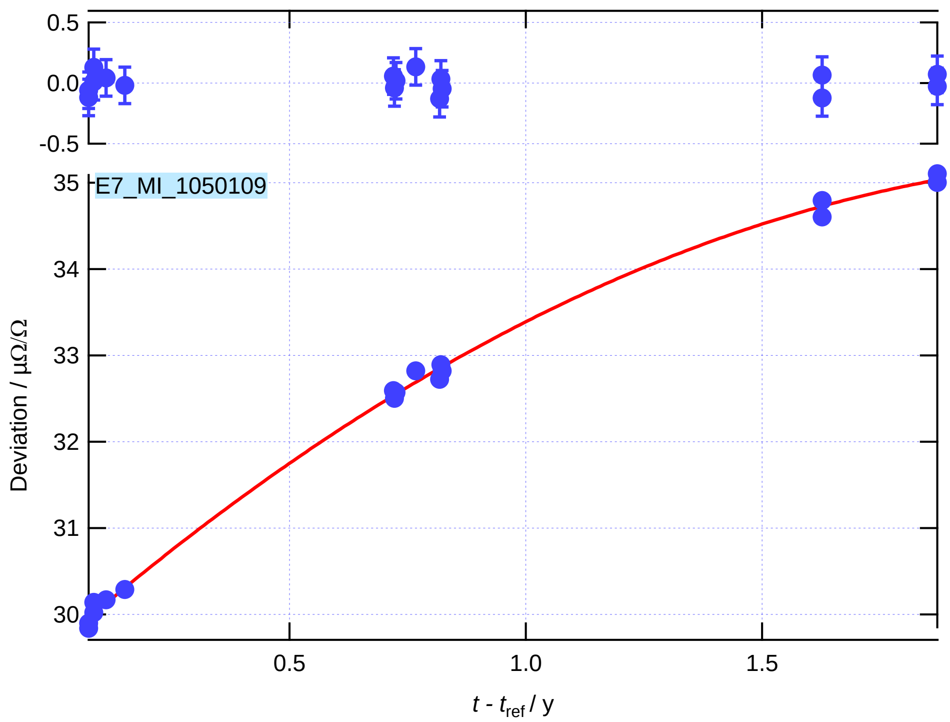

Figure 1: Drift behaviour of the 10 MΩ standard *a* = 1. The residuals to the fit are shown in the upper part of the figure.

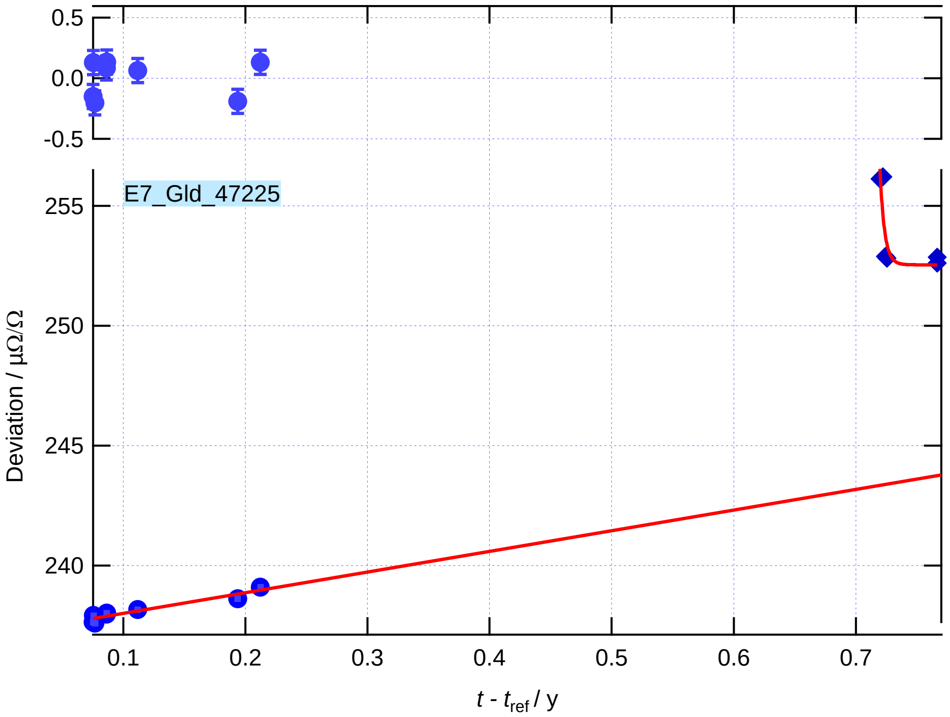

Figure 2a: Drift behaviour of the 10 MΩ standard *a* = 2 for the first part of the loop.

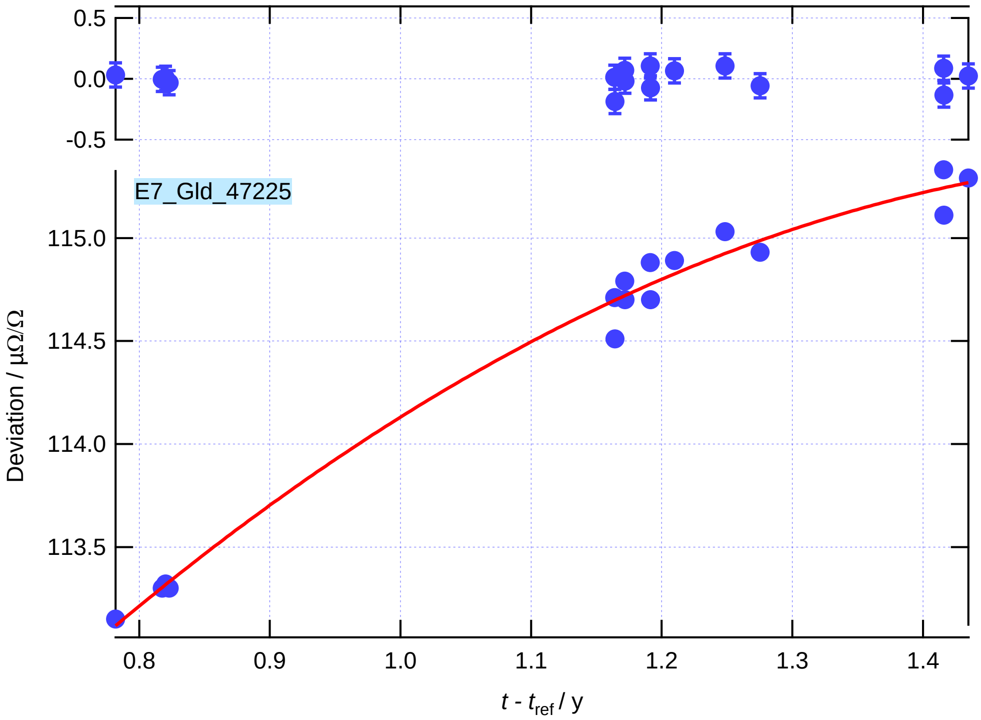

Figure 2b: Drift behaviour of the 10 MΩ standard *a* = 2 for the second part of the loop.

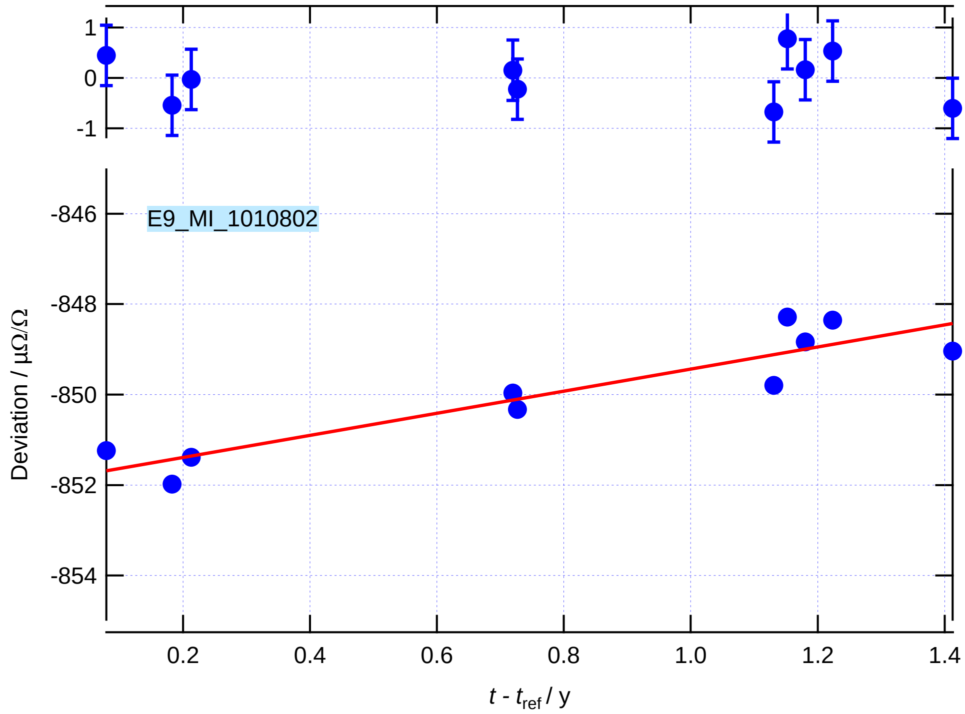

Figure 3: Drift behaviour of the 1 GΩ standard *a* = 3. The residuals to the fit are shown in the upper part of the figure.

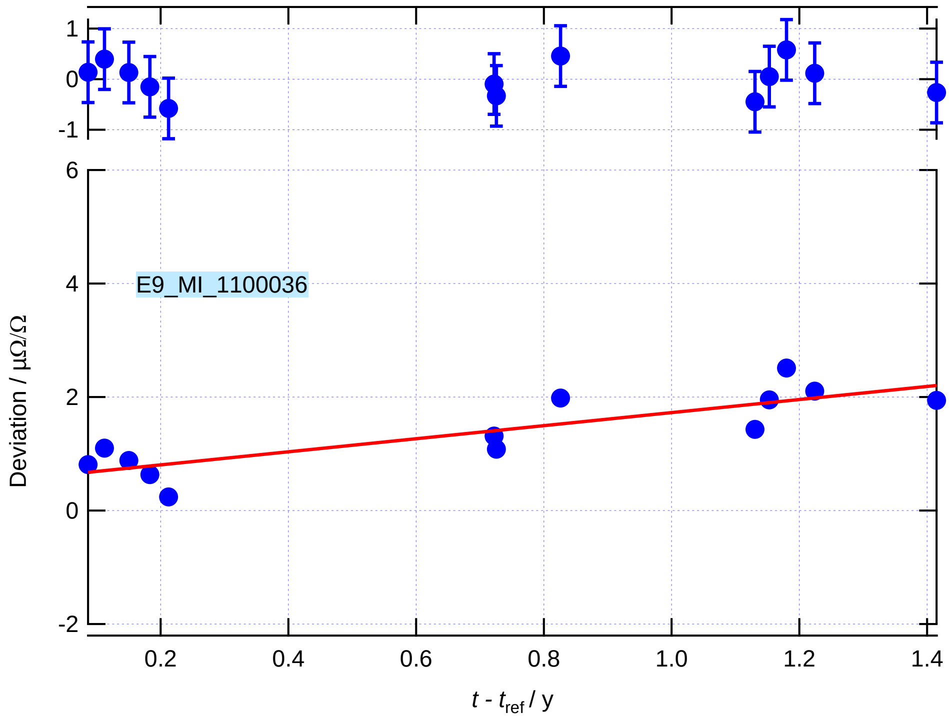

Figure 4: Drift behaviour of the 1 GΩ standard *a* = 4.

6. Analysis of comparison data set

6.1 Results of the participating institutes

The participants were asked to do as many measurements as deemed reasonable distributed in time over the whole period allocated to the laboratory. This should allow to detect a departure of the drift behaviour from the overall drift model fitted by the pilot laboratory. For each measurement point the following information was reported:

- Date of the measurement
- Resistance value
- Repeatability of the result (type A standard deviation of the measurement)
- Temperature including its uncertainty
- Test voltage

Each result reported by the participants can be expressed as:

$$R_{p,a,m} = R_{nom}\left(1+O_{p,a,m}\right) = R_{nom}\left(1+O\left(t_{p,a,m}, T_{p,a,m,} V_{p,a,m}\right)\right), \text{ with} \qquad (6.1)$$

- p: Index for the participant
- a: Index for the artefact
- m: Index for the measurement of artefact a at participant p
- $O_{p,a,m}$: Deviation from the nominal value, reported for time $t_{p,a,m}$, temperature $T_{p,a,m}$ and test voltage $V_{p,a,m}$

Furthermore, the following nomenclature is used, unless otherwise noted: $N_{p,a}$ is the number of measurements done by participant p with artefact a, N_p is the number of all measurements done by participant p.

The values $O_{p,a,m}$ and the associated standard deviations, $u_{r\text{-}p,a,m}$, are given in Annex A.

In addition to the individual results, mean values for every resistor and combined standard uncertainties were reported. The mean values were not used in the analysis (see Sect. 6.2). The reported combined uncertainties, $u_{c\text{-}p,a}$, for participant p and artefact a can be expressed as:

$$u_{c-p,a}{}^2 = u_{s-p}{}^2 + u_{r-p,a}{}^2, \text{ where:} \qquad (6.2)$$

- $u_{s\text{-}p}$: Combined standard uncertainty of the measurement set-up (step-up procedure, bridge…)
- $u_{r\text{-}p,a}$: Component related to the repeatability of the measurement; typically the standard deviation of the mean of the series of measurements performed.

The reported uncertainty values are listed in Table 6.

p	**Laboratory**	10 MΩ			1 GΩ		
		$u_{c\text{-}p,a}$ (ppm)	$u_{r\text{-}p,a}$ (ppm)	$u_{s\text{-}p}$ (ppm)	$u_{c\text{-}p,a}$ (ppm)	$u_{r\text{-}p,a}$ (ppm)	$u_{s\text{-}p}$ (ppm)
3	GUM	0.62	0.50	0.37	1.6	1.3	1.0
4	HMI/FER-PEL	0.88	0.14	0.87	4.4	1.6	4.1
5	BIM	1.45	0.26	1.43	8.7	2.8	8.2
6	NIS	47.00	3.80	46.85	-	-	-

Table 6: Combined uncertainties reported by the laboratories

6.2 Normalization of the results

6.2.1 Correction to standard ambient conditions

In a ***first step***, temperature and voltage corrections were applied to the reported results. The corrected results (expressed as deviation from the nominal resistance value) are given by:

$$O_{c-p,a,m} = O_{p,a,m} - \alpha_a \left(T_{p,a,m} - T_{nom}\right) - \gamma\left(V_{p,a,m} - V_{nom}\right) \tag{6.3}$$

The uncertainty of the mean correction term for every participant and standard may be expressed as:

$$u_{TV-p,a}{}^2 = \left(\alpha \cdot u\left(T_{p,a}\right)\right)^2 + \left(u(\alpha)\cdot\left(T_{p,a} - T_{nom}\right)\right)^2 + \left(u(\alpha)\cdot u\left(T_{p,a}\right)\right)^2 + \left(u(\gamma)\cdot\left(\overline{V}_{p,a} - V_{nom}\right)\right)^2 \tag{6.4}$$

Most of the measurements were carried out close to the nominal temperature. For this reason, also the second order term of the Taylor expansion was taken into account in the uncertainty expression. The resulting uncertainty components are listed in Tables 7 and 8.

p	**Lab**	***T* (°C)**	**V (V)**	***u*(*T*) (°C)**	**$u_{TV-p,1}$ (ppm)**	**$u_{TV-p,2}$ (ppm)**
1	METAS	23.00	10.0	0.03	0.022	0.038
3	GUM	23.02	9.1	0.10	0.074	0.127
4	HMI/FER-PEL	23.03	50.0	0.05	0.055	0.064
5	BIM	22.98	90.0	0.01	0.080	0.023
6	NIS	22.88	20.0	0.10	0.075	0.127

Table 7: Averaged measurement conditions for the 10 MΩ standards. Uncertainty contributions due to the temperature/voltage correction.

p	**Lab**	***T* (°C)**	**V (V)**	***u*(*T*) (°C)**	**$u_{TV-p,3}$ (ppm)**	**$u_{TV-p,4}$ (ppm)**
1	METAS	23.00	100.0	0.05	0.753	0.062
3	GUM	23.00	90.9	0.10	1.507	0.124
4	HMI/FER-PEL	23.01	100.0	0.05	0.754	0.062
5	BIM	22.98	90.0	0.01	0.165	0.015
6	NIS	Not measured				

Table 8: Averaged measurement conditions for the 1 GΩ standards. Uncertainty contributions due to the temperature/voltage correction.

6.2.2 Drift correction

In a ***second step***, the time dependence of the standards and an offset term, taken from the results of the pilot laboratory, are removed from the results:

$$M_{p,a,m} = O_{c-p,a,m} - f\left(t_{p,a,m}\right) \tag{6.5}$$

f(t) is the model function fitted to the results of the pilot laboratory (see Sect. 5.2.)

The normalized results $M_{p,a,m}$ are given in Annex A.

The mean value for every participant and every standard is calculated as:

$$M_{p,a} = \frac{1}{N_{p,a}} \sum_m M_{p,a,m} \tag{6.6}$$

6.2.3 Repeatability of results

In a ***third step***, the uncertainties $u_{\text{r-p,a,m}}$, which are related to the repeatability and which were indicated by the participants for each measured value were checked against the variation of the normalized results. If necessary, a corrected value based on the observed scatter of the data was determined. This was done the following way:

For every participant and artefact, the internal standard deviation of the arithmetic mean was calculated as

$$s^2_{\text{int}-p,a} = \frac{1}{N^2_{p,a}} \sum_m u^2_{r-p,a,m} \tag{6.7}$$

This value can be compared to the external standard deviation calculated from the scatter of the individual results as

$$s^2_{ext-p,a} = \frac{1}{(N_{p,a}-1)N_{p,a}} \sum_m (M_{p,a,m} - M_{p,a})^2 \; . \tag{6.8}$$

The standard deviation $u^*_{\text{r-p,a}}$ for the mean value was chosen as:

$$u^*_{r-p,a} = \max(s_{\text{int}-p,a}, s_{ext-p,a}, u_{r-p,a}). \tag{6.9}$$

The combined uncertainty component $u_{rs\text{-}p,a}$ linked to the reproducibility of the result for a particular standard can finally be expressed as:

$$u_{rs-p,a}{}^2 = u^*_{r-p,a}{}^2 + u_{TV-p,a}{}^2 + u_{tr-a}{}^2 \; . \tag{6.10}$$

The last component ($u_{\text{tr-a}}$) describes the uncertainty contribution due to transport effects. Based on the experience made during the comparison EUROMET.EM-K2 [1] with similar standards and a large number of participants and standards, the values listed in Table 9 were attributed to the standards.

Standard	a	u_{tr-a} (ppm)	Standard	a	u_{tr-a} (ppm)
10 MΩ	1	0.50	1 GΩ	3	1.50
	2	0.50		4	1.50

Table 9: Base transport variability attributed to the artefacts.

The normalized results $M_{\text{p,a}}$ and the corresponding uncertainty components linked to reproducibility are listed in Tables 10 and 11.

p	Laboratory	a =1 $N_{\text{p,1}}$	$M_{\text{p,1}}$ (ppm)	$u^*_{\text{r-p,1}}$ (ppm)	$u_{\text{rs-p,1}}$ (ppm)	a =2 $N_{\text{p,2}}$	$M_{\text{p,2}}$ (ppm)	$u^*_{\text{r-p,2}}$ (ppm)	$u_{\text{rs-p,2}}$ (ppm)
1	METAS	17	0.00	0.04	0.50	8	0.00	0.08	0.51
3	GUM	6	2.20	0.50	0.71	10	5.02	0.50	0.72
4	HMI/FER-PEL	9	4.00	0.14	0.52	9	4.37	0.14	0.52
5	BIM-NCM	5	1.99	1.55	1.63	4	1.62	1.59	1.66
6	NIS	21	-8.08	3.80	3.83	20	-69.02	3.80	3.83

Table 10: Uncertainty contributions due to the reproducibility of the measurements for the 10 MΩ standards.

p	Laboratory	*a* =3 $N_{p,3}$	$M_{p,3}$ (ppm)	$u^*_{r-p,3}$ (ppm)	$u_{rs-p,3}$ (ppm)	*a* =4 $N_{p,2}$	$M_{p,4}$ (ppm)	$u^*_{r-p,4}$ (ppm)	$u_{rs-p,4}$ (ppm)
1	METAS	10	0.0	0.2	1.7	13	0.0	0.2	1.5
3	GUM	7	4.9	1.3	2.5	12	-3.9	1.3	2.0
4	HMI/FER-PEL	9	-24.5	1.4	2.2	9	14.2	1.6	2.2
5	BIM-NCM	8	213.2	92.0	92.0	5	-6.3	44.7	44.7
6	NIS								

Table 11: Uncertainty contributions due to the reproducibility of the measurements for the 1 GΩ standards.

The normalized results for the two 10 MΩ standards and the two 1 GΩ standards are shown in Figures 5 and 6 respectively. For every participant, the two values measured for the same nominal value should agree within the uncertainty component $u_{rs\text{-}p,a}$. This is not the case for lab number 6 at 10 MΩ and lab number 4 at 1 GΩ. Reasons for discrepancies may be:

- Differences in ground-guard configurations and/or leakage effects between the standards which are not properly accounted for in the set-ups of the participants.
- Step-like changes in the value of a transport standard which recovered before the start of the measurement period carried out by the next participant in the loop. In such a case, a clear time dependence of the individual measurement values $M_{p,a,m}$ from the overall drift behaviour should be visible. This is not the case (see Appendix A).

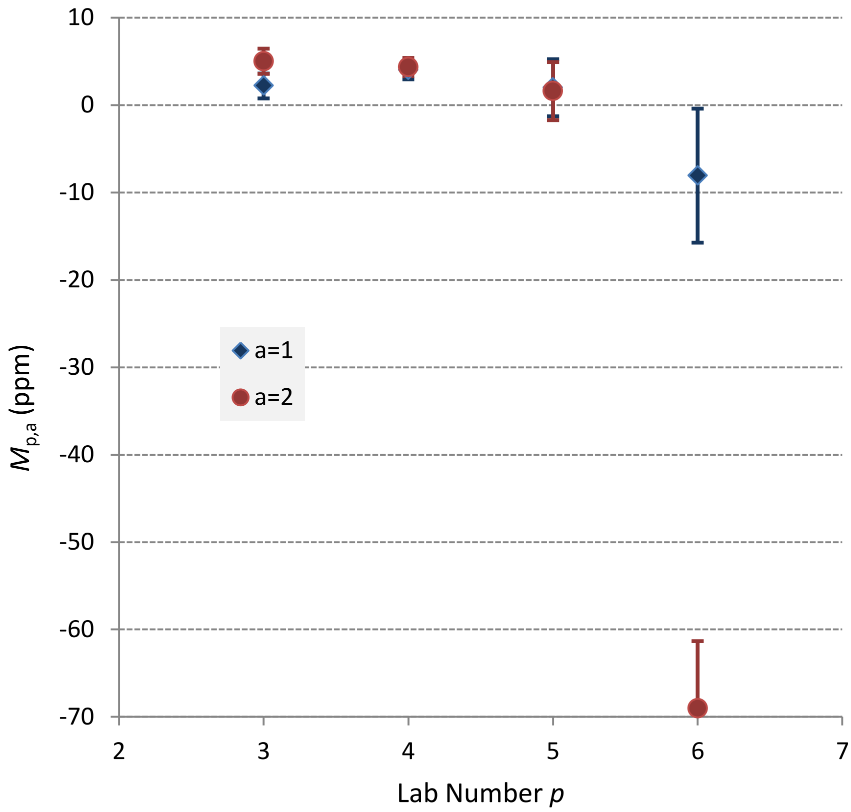

Figure 5: Normalized results for the 10 MΩ standards. The uncertainty bars represent the expanded reproducibility component $2u_{rs-p,a}$

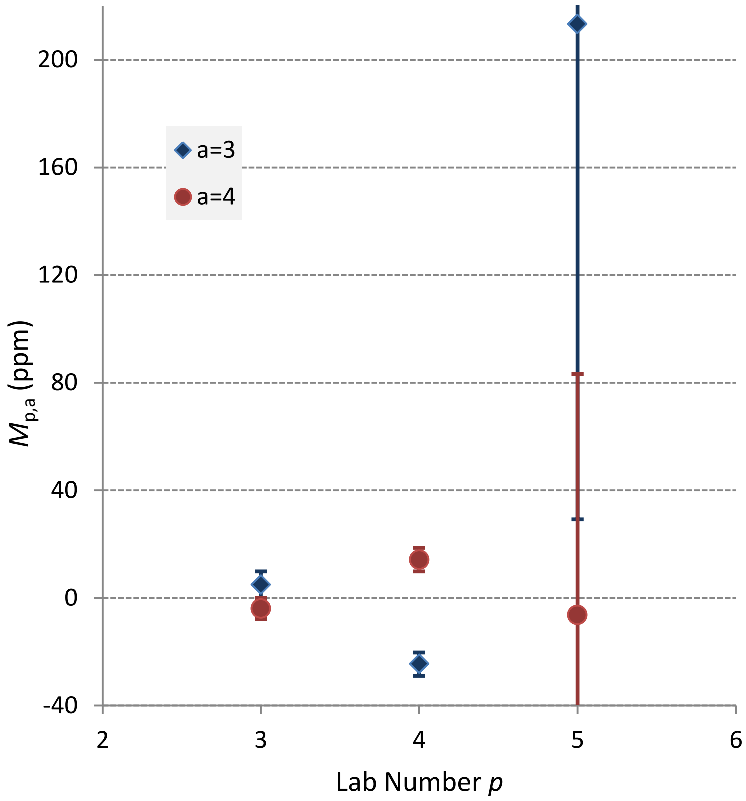

Figure 6: Normalized results for the 1 GΩ standards. The uncertainty bars represent the expanded reproducibility component $2u_{rs-p,a}$

6.2.4 Combination of results for the same nominal value

In the final step, the two results obtained for the same nominal resistance value have to be combined. The following procedure was applied:

10 MΩ

Due to the step change of the standard number two in the first part of the comparison loop, two separate cases have to be considered:

- Participants number 3, 4 and 5: The results in Figure 5 for standard number 2 are based on a linear extrapolation of the values measured by the pilot laboratory before the start of the loop. As can be seen, these results agree quite well with the results of standard number one. This may indicate that the step change of the value of standard number 2 may have occurred during the transport form participant number 5 to the pilot laboratory. Despite this observation, it is not safe to base comparison results on extrapolated fit results and it was, thus, decided to exclude the results of standard number two from the calculation for the first part of the loop. The combined normalized result and its uncertainty component due to reproducibility at 10 MΩ for participants *p*= 3, 4, 5 is then given by:

$$M_p^{10M} = M_{p,1}$$
$$u_{rs-p}^{10M} = u_{rs-p,1} \tag{6.11}$$

- Participants number 6: In this case, no anomaly in the drift behaviour of standard number two can be observed (see Fig. 2b). The combined result is thus calculated as the mean value from the normalized results from the standards number one and two:

$$M_p^{10M} = \frac{1}{2}\left(M_{p,1} + M_{p,2}\right) \tag{6.12}$$

To check the consistency of the two values $M_{p,1}$ and $M_{p,2}$, a t-test is performed. The two results are based on $N_{p,1}$ and $N_{p,2}$ individual results. The uncertainty components describing the reproducibility of the results are given by $u_{rs\text{-}p,1}$ and $u_{rs\text{-}p,2}$. Then the t-value is given by:

$$t_p = \frac{\left|M_{p,1} - M_{p,2}\right|}{\sqrt{u_{rs-p,1}{}^2 + u_{rs-p,2}{}^2}} \tag{6.13}$$

The number of degrees of freedom is: $\nu = N_{p,1} + N_{p,2} - 2$.

A common procedure is to declare the two mean values as consistent if the probability (calculated from the t-distribution) for a t-value greater than the calculated one is at least 5%. The results for the t-test are summarized in Table 12. As expected (see Sect. 6.2.3) the t-test is not passed for participant 6 at 10 MΩ. As it is not possible to decide, if this inconsistency is caused by an unknown systematic error in the measurement set-up or by an undiscovered step-change in one of the travelling standards, it is decided to increase the uncertainty component in such a way that the t-test is passed. The corresponding multiplication factor is denoted by k in Table 12. The uncertainty component of the combined result (6.12) due to reproducibility is thus:

$$u_{rs-p}^{\mathrm{10M}} = k \cdot \left(\frac{1}{2}\sqrt{u_{rs-p,1}{}^2 + u_{rs-p,2}{}^2}\right). \tag{6.14}$$

p	Laboratory	*t*-test t_p	ν	L	k	Mean values M_p (ppm)	$u_{rs\text{-}p}$
3	GUM	Not applicable				2.20	0.71
4	HMI/FER-PEL					4.00	0.52
5	BIM-NCM					1.99	1.63
6	NIS	11.24	39	2.02	5.56	-38.55	15.06

Table 12: Combined normalized results at 10 MΩ and t-test values. L is the t-value where the probability to have a t-value with a higher value is equal to 5%. The t-test is passed if $t_p \le L$.

1 GΩ

No anomalies are present in the drift behaviour of the two 1 GΩ standards. For this reason, the results of both standards are taken into account for all participants, analogue to (6.12) to (6.14):

$$\begin{aligned} M_p^{1G} &= \frac{1}{2}\left(M_{p,3} + M_{p,4}\right) \\ u_{rs-p}^{1G} &= k \cdot \left(\frac{1}{2}\sqrt{\left(u_{rs-p,3}{}^2 + u_{rs-p,4}{}^2\right)}\right) \end{aligned} \tag{6.15}$$

p	Laboratory	*t*-test t_p	ν	L	k	Mean values M_p (ppm)	$u_{rs\text{-}p}$
3	GUM	2.79	17	1.02	2.72	0.5	4.3
4	HMI/FER-PEL	12.59	16	1.03	12.27	-5.2	18.9
5	BIM-NCM	2.15	11	1.04	2.06	103.5	105.4

Table 13: Combined normalized results at 1 GΩ and t-test values. L is the t-value where the probability to have a t-value with a higher value is equal to 5%. The t-test is passed if $t_p \leq L$.

6.3 Degrees of equivalence DoE

As this comparison is a follow-up of EUROMET.EM-K2 with a small number of participants, no comparison reference value is calculated. The results of the participants are linked to the Comparison Reference Value (CRV) of EUROMET.EM-K2 through the results of the pilot laboratory. For METAS, the degrees of equivalence to the CRV in this comparison are [1]:

10 MΩ: $d_1^{10\mathrm{M}}$ = 0.49 µΩ/Ω, expanded uncertainty (k=2): $U(d_1^{10\mathrm{M}})$ = 0.57 µΩ/Ω

1 GΩ: $d_1^{1\mathrm{G}}$ = -1.4 µΩ/Ω, expanded uncertainty (k=2): $U(d_1^{1\mathrm{G}})$ = 5.6 µΩ/Ω

DoE at 10 MΩ

The DoEs are calculated as follows:

$$d_p = M_p^{10M} + d_1^{10\mathrm{M}}$$
$$u(d_p)^2 = \left(u_{rs-p}^{10M}\right)^2 + \left(u_{s-p}^{10\mathrm{M}}\right)^2 + u_{rs-1}{}^2 + u\left(d_1^{10\mathrm{M}}\right)^2 \qquad (6.16)$$

The combined uncertainty of the DoE contains the following contributions:

$u_{rs\text{-}p}$: Reproducibility component; see eq. (6.14)

$u_{s\text{-}p}$: Combined standard uncertainty of the measurement set-up (step-up procedure, bridge…) for participant p; see eq. (6.2)

$u_{rs\text{-}1}$: Reproducibility component of pilot laboratory

$u(d_1^{10\mathrm{M}})$: Uncertainty of the DoE of the pilot lab to the CRV in EUROMET.EM-K2. This uncertainty also includes $u_{s\text{-}1}$.

DoE at 1 GΩ

Analogue to (6.14), we may write:

$$d_p = M_p^{1G} + d_1^{1\mathrm{G}}$$
$$u(d_p)^2 = \left(u_{rs-p}^{1G}\right)^2 + \left(u_{s-p}^{1\mathrm{G}}\right)^2 + u_{rs-1}{}^2 + u\left(d_1^{1\mathrm{G}}\right)^2 \qquad (6.17)$$

The DoEs are summarized in Table 14 and Figures 7 and 8 resp.

p	Laboratory	**10 MΩ** d_p=DoE (ppm)	U_{DoE} (ppm)	**1 GΩ** d_p=DoE (ppm)	U_{DoE} (ppm)
3	GUM	2.7	2.0	-0.9	10.7
4	HMI/FER-PEL	4.5	2.3	-6.6	39.1
5	BIM-NCM	2.5	4.5	102.0	211.5
6	NIS	-38.1	98.4		

Table 14: The degree of equivalence DoE is the difference between a laboratory result and the comparison reference value. The uncertainty U_{doe} is the combined expanded uncertainty with a coverage factor of k = 2.

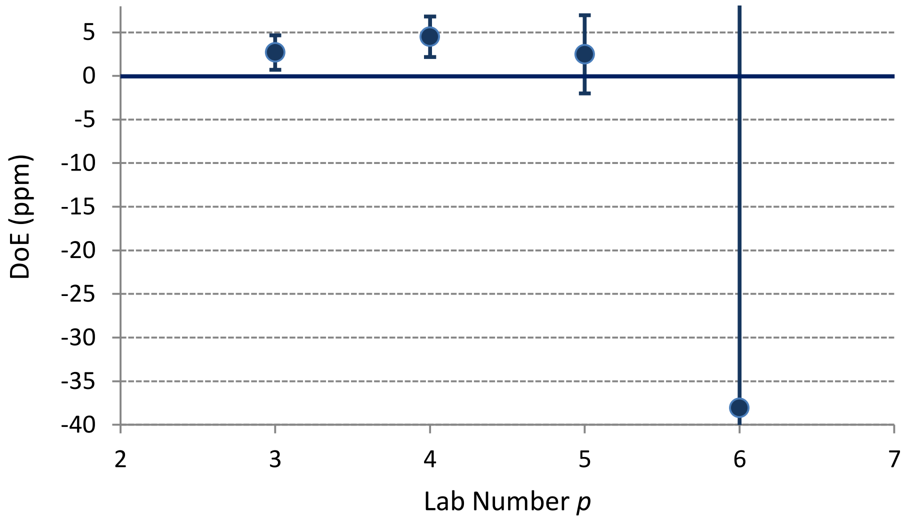

Figure 7: Unilateral degrees of equivalence with respect to the CRV at 10 MΩ.

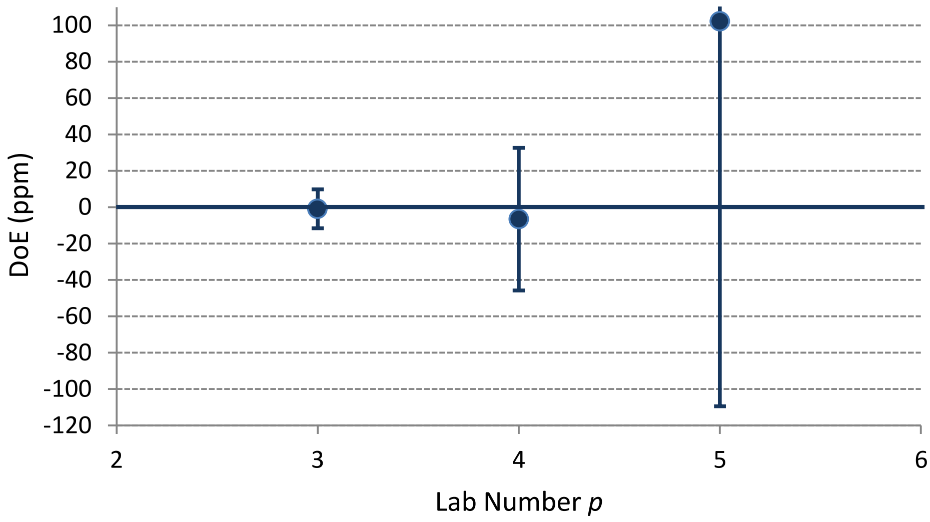

Figure 8: Unilateral degrees of equivalence with respect to the CRV at 1 GΩ.

6.4 Linking the results of EURAMET.EM-K2.1 with CCEM-K2 and degrees of equivalence

The purpose of this linking step is to determine unilateral degrees of equivalence with respect to the Key Comparison Reference Value (KCRV). The values should represent best estimates of what would have been the results of the laboratories had they actually participated in the CCEM comparison.

The linking procedure is the same as applied in the comparison EUROMET.EM-K2 (see [1] for details). It is assumed that the linking laboratories (LNE, METAS, NPL, PTB, VSL, VNIIM) performed similarly in the CCEM and in the RMO comparison. The difference between the unilateral DoE d_i in the CCEM and RMO comparison can thus be taken as the correction Δ_i which needs to be applied to the RMO values.

$$\Delta_i = d_i^{CCEM} - d_i^{RMO} \tag{6.18}$$

with i indicating the linking laboratory.

The correction to the DoEs of those who participated exclusively in the RMO comparison can then be written as

$$d_p^{CCEM} = d_p^{RMO} + \Delta$$

with uncertainties

$$u^2\left(d_p^{CCEM}\right) = u^2\left(d_p^{RMO}\right) + u^2(\Delta) \tag{6.19}$$

In the analysis made in [1], the following correction factors Δ with their standard uncertainties were determined:

10 MΩ:	**Δ = (0.54 ± 0.81) µΩ/Ω**
1 GΩ:	**Δ = (-1.43 ± 2.97) µΩ/Ω**

Using these values, the unilateral degrees of equivalence with respect to the KCRV are calculated as listed in Table 15.

p	Laboratory	10 MΩ d_p^{CCEM}=DoE (ppm)	U_{DoE} (ppm)	1 GΩ d_p^{CCEM} =DoE (ppm)	U_{DoE} (ppm)
3	GUM	3.2	2.6	-2.4	12.2
4	HMI/FER-PEL	5.0	2.8	-8.0	39.6
5	BIM-NCM	3.0	4.8	100.6	211.5
6	NIS	-37.5	98.4		

Table 15: Degree of equivalence with respect to the KCRV (CCEM-K2). The uncertainty U_{doe} is the combined expanded uncertainty with a coverage factor of k = 2.

7. Summary and conclusions

Four National Metrology Institutes, among them three EURAMET members, participated in the follow-up comparison EURAMET.EM-K2.1. The comparison aimed at evaluating the degrees of equivalence of the measurements of 10 MΩ and 1 GΩ resistance standards. At 1 GΩ, all results supplied by the participants agreed with the comparison reference value within the expanded uncertainty. At 10 MΩ, a slight disagreement with the KCRV for three of the four participants was observed.

In several cases it was observed that the normalized results for the two standards of the same nominal value were not consistent with each other. This may be an indication for imperfections in the measurement set-up and/or the measurement procedures. Based on this finding, the corresponding participants should check their set-up and the uncertainty analysis.

The analysis of the comparison results with respect to the CMC claims of the participating institutes and the measures to be taken in the case of inconsistencies are described in a separate executive report.

8. References

[1] B. Jeckelmann and M. Zeier, Final report on RMO key comparison EUROMET.EM-K2: Comparison of resistance standards at 10 MΩ and 1 GΩ, Metrologia 47 Tech. Suppl. 01006, 2010.

Annexes

A. Measurement results reported by the participants
B. Uncertainty budgets as declared by the participants
C. Technical protocol