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EM-K13.a and b DC electrical Resistance Key Comparisons between INRIM and BIPM on 1 Ω and 10 kΩ Resistors

EM-K13.a et b Comparaisons clés de résistance électrique en courant continu entre l'INRIM et le BIPM sur des résistances de 1 Ω et 10 kΩ

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I.N.RI.M. TECHNICAL REPORT

Contents

Abstract

EM-K13.a and b DC electrical Resistance Key Comparisons between INRIM and BIPM on 1 Ω and 10 kΩ Resistors

The technical report deals with the comparison BIPM.EM-K13.a (1 Ω standards) and BIPM.EM-K13.b (10 kΩ standards) between the Bureau International des Poids et Mesures (BIPM), acting as pilot laboratory, and the National Research Institute (INRIM). The comparison took part from October 2022 to February 2023, with initial and return measurements at the BIPM. The differences between the results of INRIM and of BIPM were within their expanded uncertainties. In particular these differences were: $(-0.074 \pm 0.134)\times10^{-6}$ and $(-0.045 \pm 0.174)\times10^{-6}$ respectively at 1 Ω and 10 kΩ at 2σ confidence level.

Resumé

EM-K13.a et b Comparaisons clés de Résistance électrique en courant continu entre l'INRIM et le BIPM sur des résistances de 1 Ω et 10 kΩ

Le rapport technique traite de la comparaison BIPM.EM-K13.a (étalons de 1 Ω) et BIPM.EM-K13.b (étalons de 10 kΩ) entre le Bureau International des Poids et Mesures (BIPM), faisant office de laboratoire pilote, et l'Institut National de Recherche Métrologique (INRIM). La comparaison s'est déroulée d'octobre 2022 à février 2023, avec des mesures initiales et de retour au BIPM. Les différences entre les résultats de l'INRIM et du BIPM se situaient dans les limites de leurs incertitudes élargies. En particulier, ces différences étaient les suivantes $(-0.074 \pm 0.134)\times10^{-6}$ et $(-0.045 \pm 0.174)\times10^{-6}$ respectivement à 1 Ω et 10 kΩ au niveau de confiance 2σ.

1. Introduction

National Measurement Institutes (NMIs), signatories of the CIPM Mutual Recognition Arrangement (CIPM MRA), assure the equivalence of their measurement standards and of their calibration certificates. The Calibration and Measurement Capabilities (CMCs) of the signatories NMIs are internationally recognized as peer-reviewed and approved. To maintain the condition of signatories of the CIPM MRA, NMIs must participate at key and supplementary comparisons and establish a quality system according to the requirements of the ISO/IEC 17025:2017 standard. In this framework, the comparison

BIPM.EM-K13.a and b, concerning the calibration of two 1 Ω and two 10 kΩ BIPM travelling standards was carried out from October 2022 to February 2023 between INRIM and the BIPM itself according to the measurement protocol [1].

2. Travelling resistors

The four K13 traveling standards were two resistors with 1 Ω and two 10 kΩ resistors identified as (Table 1):

Table 1. Travelling resistors to of the comparison.

Standard	*Manufacturer*	*Mod.*	*Serial no.*	*Label*
1 Ω	CSIRO	*NML*	64200	BIV200
1 Ω	CSIRO	*NML*	64203	BIV203
10 kΩ	TEGAM	SR104	K203039730104	B10K09
10 kΩ	TEGAM	SR104	K201089830104	B10K12

Table 2 reports the temperature, pressure and power coefficients of the travelling resistors.

Table 2. Temperature, pressure and power coefficients of the travelling resistors

	Relative temperature oefficients			**Relative pressure coefficient**		**Relative power coefficient**	
Item	α_{23} (10^{-6}/K)	β/ (10^{-6}/K^2)	u_T (10^{-6}/ K)	γ (10^{-9}/hPa)	u_P (10^{-9}/hPa)	P (10^{-9}/mW)	u_W (10^{-9}/mW)
BIV200	− 0.0074	− 0.0004	0.001	− 0.130	0.200	− 2.0	2.0
BIV203	− 0.0096	− 0.0016	0.001	− 0.200	0.200	− 2.0	2.0
B10K09	− 0.0400	− 0.0220	0.010	− 0.164	0.100	1.0	3.0
B10K12	+ 0.0100	− 0.0230	0.010	− 0.226	0.100	1.0	3.0

3. Measurements at the BIPM

3.1. 1 Ω measurements

The 1 Ω measurements were carried out by comparison with a 100 Ω reference resistor referred every 6 months to the BIPM QHR. The comparison 100 Ω -1 Ω was performed by means of a cryogenic current comparator (CCC) with 50 mA in the 1 Ω resistors. The travelling 1 Ω resistors were measured 11 times in October 2022 (before in Tab. 3) and 10 times in January 2022 – February 2023 (after in Tab. 3). The 1 Ω resistors were placed in a thermo-controlled oil bath at a 23 °C (within a few mK). The oil temperature close to each standard was determined by means of a calibrated Standard Platinum Resistance Thermometer (SPRT) and thermocouples near the resistors [1]. Table 3 reports the 1 Ω values determined by the BIPM, expressed as relative deviation from the nominal value and after application of the temperature and pressure corrections. The measurements were made respectively before and after the INRIM measurements. The same Table reports also the standard deviation of the measurements and the same measurements interpolated at the reference date of the INRIM measurements.

Table 3. BIPM results for the 1 travelling Ω standards.

BIPM	**Relative deviation from nominal 1 Ω value** (μΩ/Ω)					
Item	Before	Std dev. u_{1B}	After	Std dev. u_{1A}	Interpolated at 24-11-2022	Std dev. u_1
BIV200	− 0.717	0.011	− 0.763	0.010	− 0.733	0.007
BIV203	+ 0.596	0.007	+ 0.521	0.008	+ 0.572	0.005

3.2. 10 kΩ measurements

The 10 kΩ measurements were carried out by comparison with a set of two 10 kΩ reference resistors in turn calibrated every 6 months vs. the BIPM QHR standard. The comparison was performed with a Warshawsky bridge [2] at 0.1 mA (1 V). The 10 kΩ travelling resistors were placed in a temperature-controlled air bath at (23 °C ± 0.05) °C. The travelling 10k Ω resistors were measured 13 times in October 2022 (before in Tab. 4) and 10 times in January 2022 – February 2023 (after in Tab. 4). Table 4 reports the 10 kΩ values determined by the BIPM, expressed as relative deviation from the nominal value and after application of the temperature and pressure corrections. The measurements were made respectively before and after the INRIM measurements. The same Table reports also the standard deviation of the measurements and the same measurements interpolated at the reference date of the INRIM measurements.

Table 4. BIPM results for the 10 kΩ travelling standards.

BIPM	**Relative deviation from nominal 10 kΩ value** (μΩ/Ω)					
Item	Before	Std dev. u_{1B}	After	Std dev. u_{1A}	Interpolated at 24-11-2022	Std dev. u_1
B10K09	+ 0.027	0.002	+ 0.097	0.001	+ 0.055	0.001
B10K12	+ 1.155	0.002	+ 1.093	0.005	+ 1.130	0.003

4. Measurements at INRIM

The travelling standards were carefully packed by the BIPM, but when the container arrived at INRIM one of side had been deformed during the travel (Fig. 1 a, and b). The condition of the container was promptly communicated to the BIPM.

Fig. 1a, b – Unboxing and check

All the resistors were connected to the measuring instrumentation by means of Teflon-insulated twisted AXON-type cables equipped with copper termination forks. The temperatures of the two baths were measured using platinum thermometers, carrying out two series of resistance measurements consecutively at two different current values (1 mA and 1.41 mA), and applying the correction for self-heating. In correspondence with the resistance measurements of the traveling samples, atmospheric pressure was also acquired using a Barocap-Technology instrument.

4.1. 1 Ω measurements

The 1 Ω resistors were placed in a Guildline 9732 VT no. 58.805 oil bath with a set point temperature of 23.000°C (Fig. 2a). The oil temperature was measured with a calibrated Leeds & Northrup 25 Ω thermometer while the depth of the resistors in the oil, measured between the surface and the top of the standards was 32 mm (Fig. 2b). The oil used is Marcol 52 type with RD the relative density of the oil = 0.83

Fig. 2a. Guildline 9732 VT oil bath. The short-term stability of the bath is approximately 0.001°C.

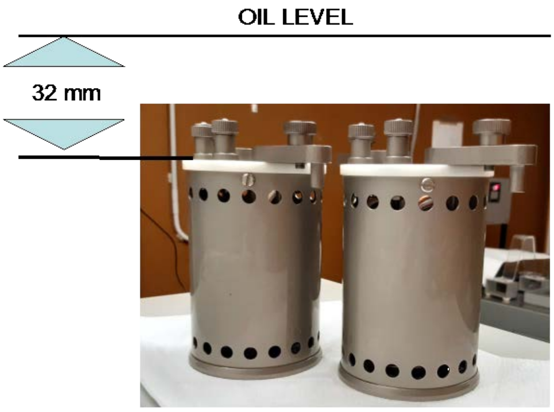

Fig. 2b. Oil immersion depth of the 1 Ω standards.

The 1 Ω resistors were measured 30 times between November 2022 and December 2022. Table 5 reports the mean values at the mean date (24th of November 2022) as communicated by INRIM for the two 1 Ω resistors, hence without temperature and pressure corrections. The repeatability is estimated by the standard deviation of the measurements.

Table 5. INRIM results for the 1 Ω travelling resistors before corrections.

INRIM before corrections	Relative difference from nominal 1 Ω value (μΩ/Ω)	Std dev. (μΩ/Ω)	Mean temperature /°C	Mean pressure at the reference plane /hPa
BIV200	− 0.780	0.026	22.999	986.80
BIV203	+ 0.480	0.026	22.999	986.80

The INRIM measurements were then corrected by the BIPM and reported to the reference temperature and pressure of 23,000 °C and 1,013.25 hPa, respectively, using the coefficients of the resistors listed in Table 1. Additional elements and information can be found in [3, 4]. The correction result is visible in Table 6, along with the repeatability and systematic uncertainties of the INRIM measurements, as well as a cumulative uncertainty due to the temperature and pressure corrections of the same measurements. For the analysis of Type B uncertainties, refer to Section 6, Table 11.

Table 6. INRIM results for the 1 Ω travelling resistors after corrections.

INRIM after corrections	Relative difference from nominal 1 Ω value (μΩ/Ω)	Type A u_1 (μΩ/Ω)	Type B u_2 (μΩ/Ω)	Corrections u_3 (μΩ/Ω)
BIV200	− 0.783	0.026	0.062	0.005
BIV203	+ 0.475	0.026	0.062	0.005

4.2. 10 kΩ measurements

The 10 kΩ resistors were housed inside a Kambic air bath mod. TK-105 US no. 15115002 set at the temperature of 23°C. The air temperature near the two resistors was measured with a calibrated 100 Ω no. 4446 FLUKE thermometer.

Fig. 3 - 10 kΩ standards air-bath. The resistors were placed inside a Kambic air-bath mod. 105 with a set-point of 23.000°C and a short-term stability of approximately 0.005°C. The temperature was monitored with a PT100 platinum thermometer.

The 10 kΩ resistors were measured 30 times between November 2022 and December 2022. The measurements were made at 100 μA with a current reversal time of 12 seconds (filter 0.3 s). Table 7 reports the mean values at the mean date (24th November 2022) without temperature and pressure corrections. The repeatability is estimated by the standard deviation of the measurements.

Table 7. INRIM results for the 10 kΩ travelling resistors before corrections.

INRIM before corrections	Relative difference from nominal 10 kΩ value (μΩ/Ω)	Std dev. (μΩ/Ω)	Mean temperature /°C	Mean pressure at the reference plane /hPa
B10K09	+ 0.012	0.021	22.978	988.35
B10K12	+ 1.095	0.017	22.978	988.35

As for the 1 Ω measurements, these readings were subsequently adjusted by BIPM to the reference temperature and pressure of 23.000 °C and 1013.25 hPa, respectively. This was achieved by employing the established coefficients of the resistors (refer to Table 1). Subsequently, Table 8 presents the values post-correction, inclusive of both the random and systematic uncertainties of the INRIM measurements, as well as an aggregate uncertainty stemming from the temperature and pressure adjustments applied to these same measurements.

Table 8. INRIM results for the 10 kΩ travelling resistors after corrections.

INRIM after corrections	Relative difference from nominal 10 kΩ value (μΩ/Ω)	Type A u_1 (μΩ/Ω)	Type B u_2(μΩ/Ω)	Corrections u_3 (μΩ/Ω)
B10K09	+ 0.007	0.021	0.085	0.003
B10K12	+ 1.089	0.017	0.085	0.003

5. INRIM instrumentation and measurement systems

The measurement system used by INRIM for this international comparison is described in detail in the INRIM technical procedure [5]. Nevertheless, the same system is still based on the one described in [6] with only little difference as the use of a new high-performance automated DCC bridge in place of a previous one. The group of INRIM working standards consists in the resistors in fig. 4. The arrows indicated in the diagram show the comparisons and the direction of the traceability in the measurements for the calibration of all the INRIM standards and for the periodic checks. The source of traceability stems from the Quantum Hall Effect (QHE), which is utilized to calibrate the series side of a 12960 Ω/10 Ω Hamon network. Subsequently, by means of ratios 1:10 directly with a Measurements International MI 6010Q (DCC) bridge, or in ratio 1:100 with the parallel side of the Hamon network and DCC networks, all the elements of the scale are calibrated. The first step of the K13 precision measurements was carried out during week No. 46 with a calibration of the QHE-10 Ω transfer Hamon network with an associated uncertainty equal to U= 7×10^{-7} at 2σ confidence level. The transfer of traceability from the 10 Ω Hamon network to the first element of the decade scale must take place within a short time, usually a few hours. The first group of resistors to receive traceability consists of the 10 Ω– 1 Ω – 100 Ω standards, which are calibrated and compared with each other both in a 1:10 ratio and with the 10 Ω Hamon network. Due to the impossibility of exchanging the compared resistors when they are in a ratio of 1:10, to reduce the bridge interchange error, the Hamon network is used by comparing the resistors following the diagram in Fig. 5, which allows reducing this type of systematic error. The second group of resistors consists of 100 Ω– 1kΩ – 10kΩ. Traceability is transferred to the 10 kΩ resistor via a 1kΩ step Hamon network, while the 1 kΩ resistor is calibrated against both 100 Ω and 10 kΩ, again in a 1:10 ratio. The precision measurements on the K13 traveling standards are carried out in a 1:1 ratio with 1 Ω and 10 kΩ standards of the INRIM working standard scale.

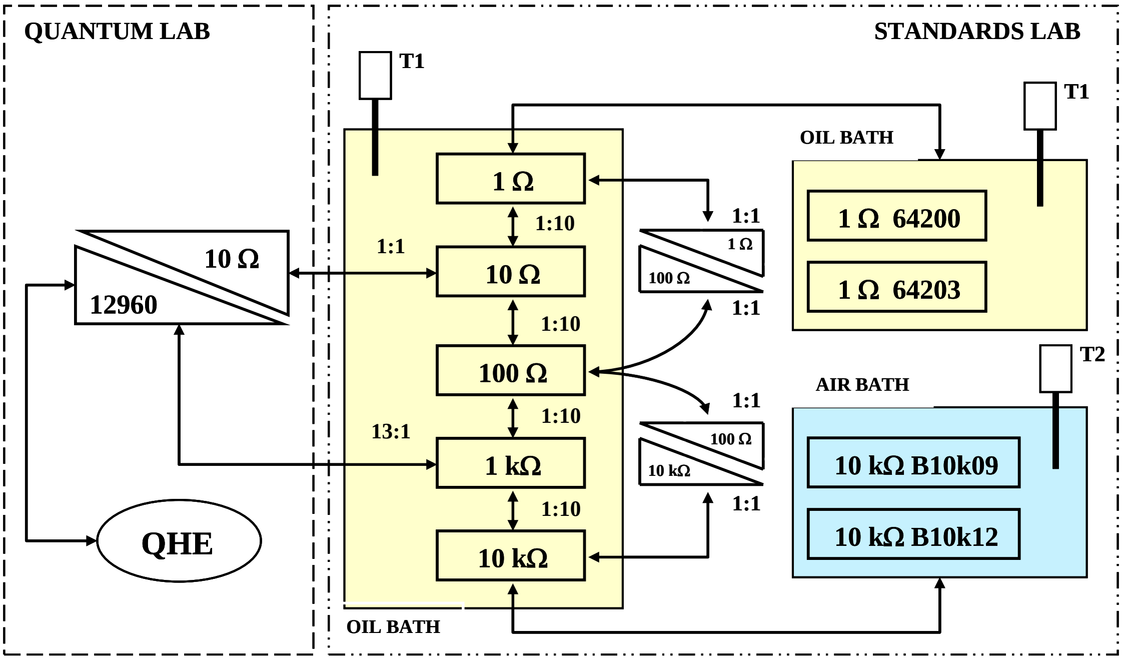

Fig. 4 - Scheme of the measurement system for the K13 a & b key comparison. The traceability comes from the QHE realized in the INRIM Quantum laboratory and transferred to the decade scale in two ways: through a specifically designed Hamon network and by 1:13 ratio direct comparison, with a MI 6010Q DCC bridge. The working references and the two K13 1 Ω were kept together in a Guildline oil bath while the reference ESI and the K13 10 kΩ resistors were kept at 23.000 ± 0.005°C in a Kambic air-bath.

6. Uncertainties analysis

6.1 BIPM uncertainties

Tables 9 and 10 report, for the1 Ω and 10 kΩ respectively, the BIPM combined standard uncertainties budget for the 1 Ω and 10 kΩ K13 resistors [7].

Table 9 – BIPM combined standard uncertainty budget for the K13 1 Ω resistors.

Source of uncertainty	**Relative standard uncertainty (nΩ/Ω)**
Imperfect realisation of R_K	2
Calibration of the BIPM 100 Ω reference (BI100-3) against R_K	3
Interpolation / extrapolation of the value of BI100-3	13
Measurement of the (1 Ω / BI100-3) ratio	8
Temperature correction for the 1 Ω standard	2
Pressure correction for the 1 Ω standard	3
Combined standard uncertainty u_2	**16**

Table 10 – BIPM combined standard uncertainty budget for the K13 10 kΩ resistors.

Source of uncertainty	**Relative standard uncertainty(nΩ/Ω)**
Imperfect realization of R_K	2
Calibration of the BIPM 100 Ω reference (BI100-3) against R_K	3
Link 100 Ω / 10 kΩ	5
Link 10 000 Ω / (mean reference B10K1-B10K2)	7
Extrapolation of mean value of 10 kΩ reference	8
Measurement of the voltage applied to the bridge	5
Measurements of the bridge unbalance voltage	5
Leakage resistances	1
Temperature correction for travelling standard	3
Pressure correction for travelling standard	2
Combined standard uncertainty u_2	**15**

6.2 INRIM uncertainties

Tables 11 and 12 report, for the1 Ω and 10 kΩ respectively, the INRIM type B uncertainties budget for the 1 Ω and 10 kΩ K13 resistors.

Table 11 – Type B uncertainty budget for the K13 1 Ω resistors.

	Source of uncertainties	*Type*	u_r (nΩ/Ω)	ν
	Link from R_K to Hamon 36×360 Ω (H_{SS} = 12960 Ω)	B	35.2	∞
	H_{SS}/H_{sp} (10 Ω) ratio correction	B	17.1	∞
	H_{sp} temperature coefficient	B	0.03	∞
	temperature difference H_{SS} and H_{sp}	B	0.58	∞
	H_{sp} drift (1 day)	B	0.69	∞
	$u_c(R_{HSP})$		**39.1**	
Transfer $H_{ss} \rightarrow H_{sp}$	Bridge ratio error correction	B	46.2	∞
	Effect due to H_{sp} temperature coefficient	B	5.8	∞
	H_{sp} drift (1 day)	B	0.3	∞
	Emf	B	9.99	∞
	Temperature instability on R_x	B	7.1	∞
	Resistors Johnson noise	B	0.74	∞
	$u(R_x)$ type B		**62.1**	

Table. 12 - Type B uncertainty budget for the K13 10 kΩ resistors.

Source of uncertainties	*Type*	u_r (nΩ/Ω)	ν
Link from RK to standard 100 Ω ESI	B	61.7	50
Bridge ratio error correction	B	8.0	∞
Hamon ratio transfer error	B	57.7	∞
Temperature coefficient	B	0.23	∞
Hamon drift	B	negl.	∞
Emf	B	0.02	∞
Temperature instability on R_x		3.12	∞
Resistors Johnson noise	B	0.74	∞
$u(R_x)$ type B		**84.9**	

7. Comparison INRIM – BIPM

The measurements of the four standards are shown in Figs 5 to 8. The plots also show the mean value of the INRIM measurements with the uncertainty bar corresponding to the expanded uncertainty of the comparison U_c and a BIPM linear fit before and after measurements.

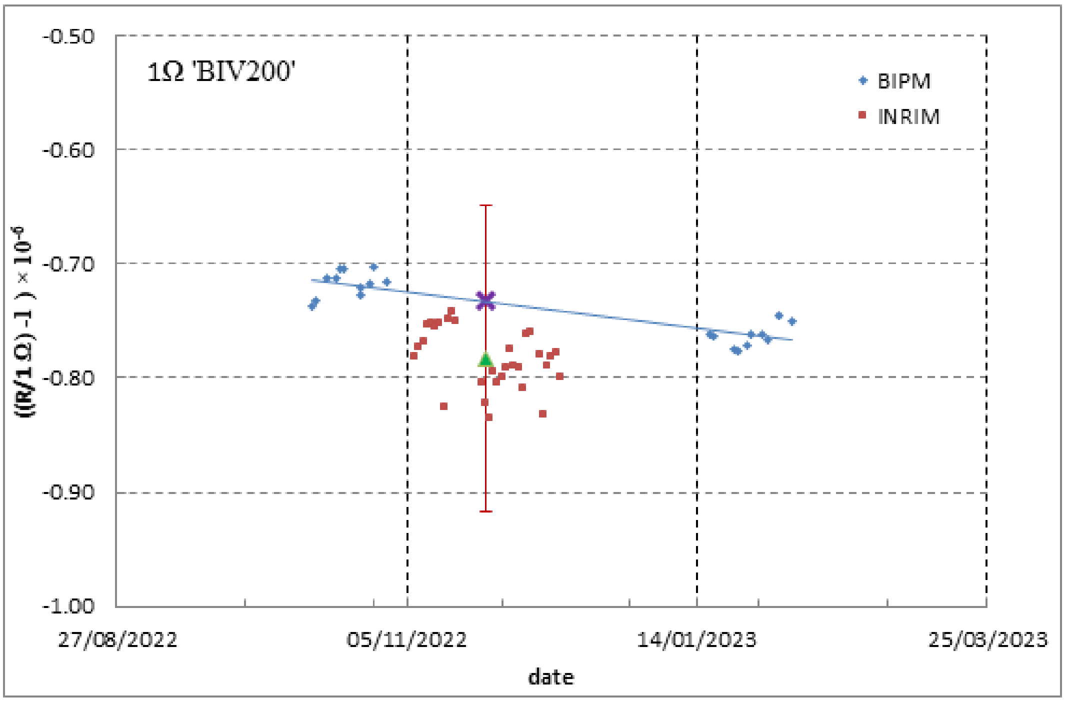

Fig. 5

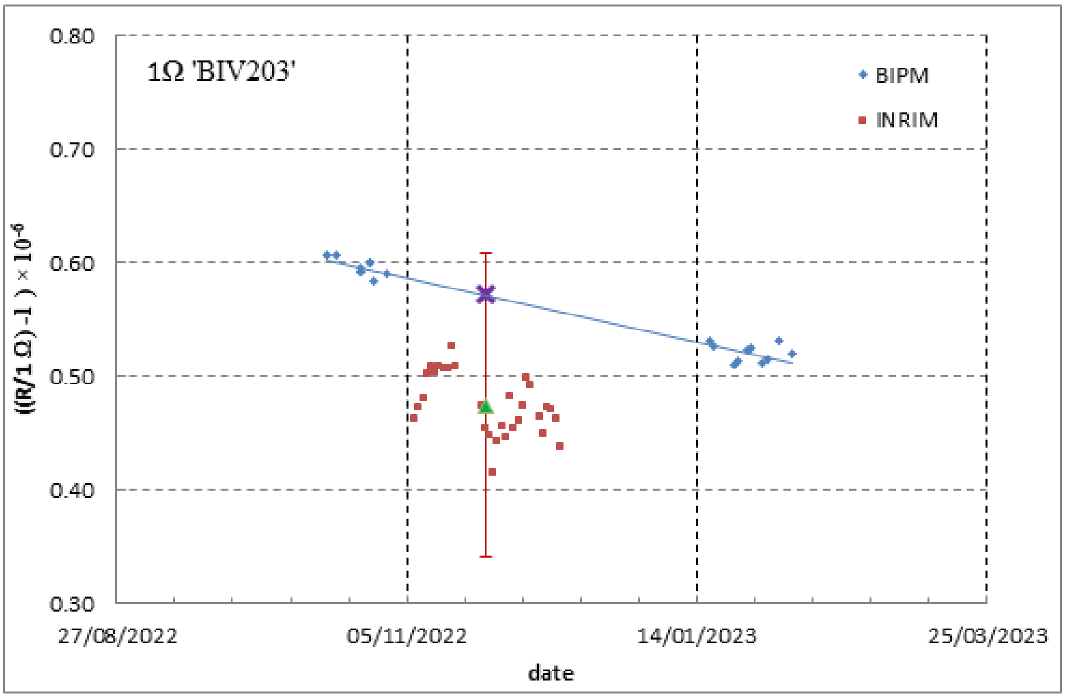

Fig. 6

Fig. 5-6. Results for the 1 Ω BIV200 and BIV203. BIPM (blue diamonds) and INRIM (red squares). The cross is the extrapolated BIPM measurement at the mean date of measurement at INRIM like the green triangle is the mean value of INRIM measurements. The uncertainty bar corresponds to the expanded uncertainty of the comparison of the mean INRIM results.

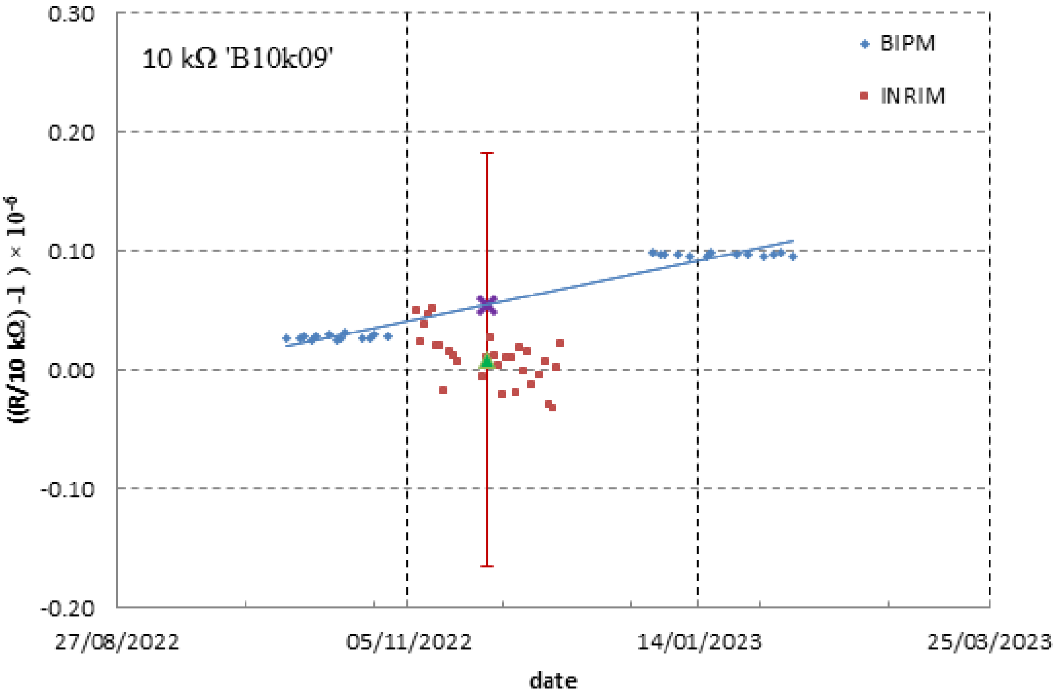

Fig. 7

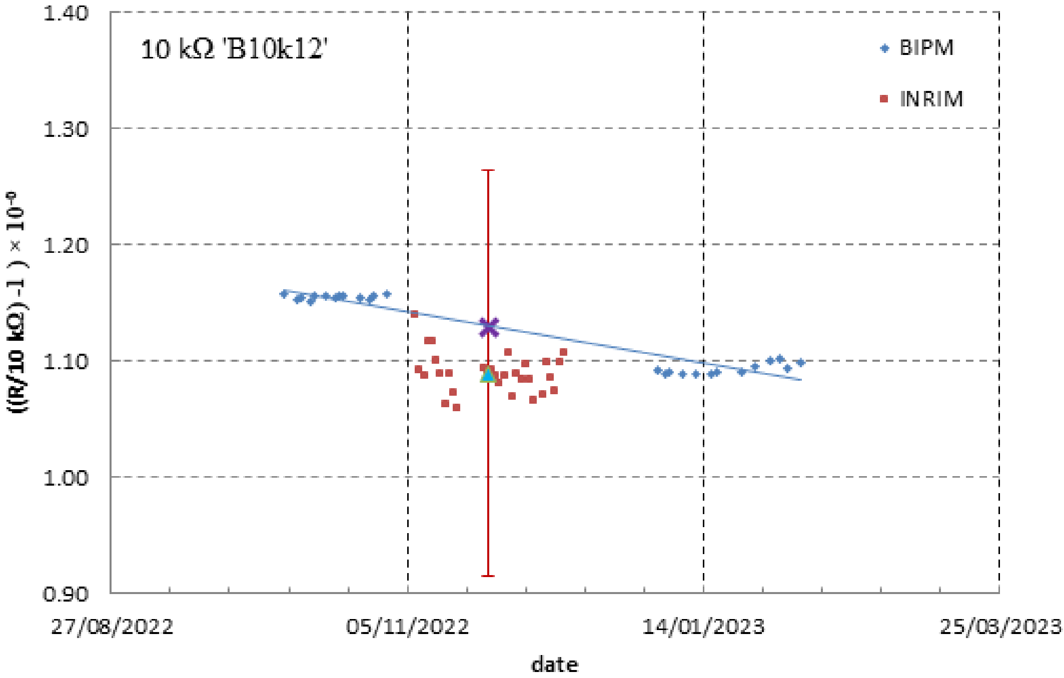

Fig. 8

Fig. 7-8. Results for the 10 kΩ B10K12. Symbols and measurement parameters are the same of Fig. 5-6

The result of the comparison for a single resistor is the difference between the mean of the INRIM measurements and the interpolated value of the linear fit of the BIPM measurements on the mean date of the INRIM measurements. The difference between the INRIM and the BIPM calibrations on a single R_i is:

$$\Delta_i = R_{\mathrm{INRIM},i} - R_{\mathrm{BIPM},i} \tag{1}$$

With two standards, the mean differences is:

$$\Delta_{\mathrm{INRIM-BIPM}} = \frac{1}{2}\sum_{i=1}^{2}\left(R_{\mathrm{INRIM}\,,i} - R_{\mathrm{BIPM},i}\right) \tag{2}$$

For each standard, the uncertainty u_1 of the interpolated BIPM value comes from the linear fit while a component u_2 comes from the BIPM measurement system and its traceability. u_2 is strongly correlated between calibrations in the same period. Therefore, for a single standard *Ri*, the BIPM uncertainty $u_{\mathrm{BIPM},i}$ is:

$$u^2_{\mathrm{BIPM},i} = u^2_{1,i} + u^2_{2,i}. \tag{3}$$

For two standards the BIPM uncertainty is:

$$u^2_{\mathrm{BIPM}} = \sum_{i=1}^{2}\frac{u^2_{1,i}}{2^2} + u^2_2 \tag{4}$$

For the INRIM measurements, the uncertainty components u_2 and u_3 of Tables 6 and 8 are correlated between standards, and u_1 is uncorrelated. Therefore, the total INRIM uncertainty is:

$$u^2_{\mathrm{INRIM}} = \sum_{i=1}^{2}\frac{u^2_{1,i}}{2^2} + u^2_2 + u^2_3 \tag{5}$$

An additional uncertainty component due to the transfer was not added as the before' and ‘after' measurements differ negligibly compared to the total uncertainty of the comparison.

7.1 Results at 1 Ω

Table 13 reports the differences between the values assigned by INRIM, and those assigned by the BIPM, to the two travelling standards on the mean date of the INRIM measurements.

Table 13: INRIM – BIPM differences for the two 1 Ω travelling standards.

INRIM − BIPM

Item #	$10^6\times(R_{\mathrm{INRIM}} - R_{\mathrm{BIPM}})$ /(1 Ω)
BIV200	− 0.050
BIV203	− 0.097
Mean	**− 0.074**

The relative combined standard uncertainty of the comparison, u_c is:

$u_c^2 = u_{\mathrm{BIPM}}^2 + u_{\mathrm{INRIM}}^2 = 0.067 \times 10^{-6}$ (6)

Where $u_{\mathrm{BIPM}} = 0.017 \times 10^{-6}$ and $u_{\mathrm{INRIM}} = 0.065 \times 10^{-6}$

The degree of equivalence as deviation D and expended uncertainty U_C at a confidence level of 95 % between INRIM and BIPM at 1 Ω is:

$$D = (R_{\mathrm{INRIM}} - R_{\mathrm{BIPM}}) / 1\ \Omega = -0.074 \times 10^{-6} \quad (7)$$

$$U_C = 0.134 \times 10^{-6} \quad (8)$$

7.2 Results at 10 kΩ

Table 14 reports the differences between the values assigned by INRIM, and those assigned by the BIPM, to the two travelling standards on the mean date of the INRIM measurements.

Table 14: INRIM – BIPM differences for the two 10 kΩ travelling standards.

	INRIM − BIPM
Item #	$10^6 \times (R_{\mathrm{INRIM}} - R_{\mathrm{BIPM}}) / (10\ \mathrm{k\Omega})$
B10K09	− 0.048
B10K12	− 0.041
Mean	**− 0.045**

The relative combined standard uncertainty of the comparison, u_c is:

$u_c^2 = u_{\mathrm{BIPM}}^2 + u_{\mathrm{INRIM}}^2 = 0.087 \times 10^{-6}$ (6)

Where $u_{\mathrm{BIPM}} = 0.015\ 10^{-6}$ and $u_{\mathrm{INRIM}} = 0.086 \times 10^{-6}$

The degree of equivalence as deviation D and expended uncertainty U_C at a confidence level of 95 % between INRIM and BIPM at 10 kΩ is:

$$D = (R_{\mathrm{INRIM}} - R_{\mathrm{BIPM}}) / 10\ \mathrm{k\Omega} = -0.045 \times 10^{-6} \quad (7)$$

$$U_C = 0.174 \times 10^{-6} \quad (8)$$

7.3 Results of the comparison

The differences between the INRIM and the BIPM calibration results at 1 Ω and 10 kΩ are within the expanded uncertainty.

Acknowledgment

The authors would like to extend their heartfelt thanks to Dr. Benjamin ROLLAND, Dr. Pierre Gournay, and the entire BIPM team for all the effort that enabled INRIM's participation in the EM-K13 key comparison cycle. Special gratitude is extended to B. Rolland for the courtesy and exceptional availability demonstrated throughout all the comparison.

References

[1] R. Goebel, Technical protocol for BIPM.EM-K13a&b comparisons, BIPM/ELEC-T-17 for Electrical Resistance Standards using 1 Ω and 10 kΩ Resistors as Travelling Standards, rev. 1.1, December 2015.

[2] R. F. Dziuba and L. L. Kile, "*An Automated Guarded Bridge System for the Comparison of 10 kΩ Standard Resistors*", IEEE Instrumentation and Measurement Technology Conference,Ottawa, Canada, May 19-21,(1997).

[3] B. Rolland, P. Gournay, M. Stock, P. P. Capra, F. Galliana, Bilateral comparison of 1 Ω and 10 kΩ standards (ongoing BIPM key comparisons BIPM.EM-K13) between the INRIM (Italy) and the BIPM, April 2023, Draft B.

[4] B Rolland et al 2023 Metrologia 60 01010 DOI 10.1088/0026-1394/60/1A/01010.

[5] PT-EM.2.1-07, Calibration of 1 Ω ÷ 10 kΩ INRIM standard resistors rev.03, 2021.

[6] G. Boella and G. Marullo Reedtz, "A Room Temperature Setup to Compare the Quantized Hall Resistance with 1 Ω Standards, IEEE Trans Instrum Meas, 41. I, February 1992.

[7] JCGM 100:2008 Evaluation of measurement data - Guide to the expression of uncertainty in measurement First edition.