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NRIM-BIPM.EM-K13.a and b Key Comparisons of Electrical Resistance Standards using 1 Ω and 10 k Ω Resistors as Travelling Standards: INRIM results

Original

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**INRIM-BIPM.EM-K13.a and b Key Comparisons of Electrical Resistance
Standards using 1 Ω and 10 k Ω Resistors as Travelling Standards:
INRIM results**

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Abstract

The technical report summarizes the results provided by the Resistance laboratory of the National Research Institute (INRIM) concerning the calibration of two 1 Ω and two 10 k Ω standard travelling resistors related to the comparison BIPM.EM-K13.a (1 Ω resistance standards) and BIPM.EM-K13.b (10000 Ω resistance standards) which constitute the BIPM programme of on-going bilateral comparisons of resistance standards. The chosen resistors normally assure a transfer uncertainty less than a few parts in 10^8 . The comparison was carried out following an initial/return measurement pattern: the BIPM travelling standards were first calibrated at the BIPM then they were sent to INRIM where they were calibrated, and finally re-calibrated after their return at the BIPM. All measurement results will be corrected to the reference temperature (23.000 $^{\circ}\text{C}$) and the reference pressure (1013.25 hPa). The result of the comparison will be expressed as the difference between the calibrations from the two laboratories, together with its associated uncertainty. The pilot laboratory was the BIPM.

1. Introduction

The National Institute of Metrological Research (INRIM), acting as National Measurement Institute (NMI), it is signatory of the CIPM Mutual Recognition Arrangement (CIPM MRA). The CIPM Mutual Recognition Arrangement is the framework through which National Metrology Institutes demonstrate the international equivalence of their measurement standards and the calibration and measurement certificates they issue. The outcomes of the Arrangement are the internationally recognized (peer-reviewed and approved) Calibration and Measurement Capabilities (CMCs) of the participating institutes. Approved CMCs and supporting technical data are publicly available from the CIPM MRA database (the KCDB) and the results of scientific comparisons underpinning these CMCs. In this last framework, INRIM participated to the bilateral comparison BIPM.EM-K13a&b comparison with the Bureau international des poids et mesures, BIPM), acting as pilot laboratory. The INRIM measurements on the four travelling standards (two 1 Ω and two 10 k Ω took part between November and December 2022. The present document reports the details on:

- The travelling resistors to be calibrated;
- The receiving of the same standards;
- The climatic condition in which they were maintained at INRIM;
- The instrumentation and measurement system to calibrate them at INRIM;
- The INRIM results reports as measurements, tables and graphs;
- An analysis of the calibration uncertainties.

The measurements, the standards conditioning and the expression of the results with their associated uncertainties were made according to the technical protocol of the comparison [1].

2. Standards package

The four K13 traveling standards arrived at INRIM on 04/11/2022 inside an aluminum box. The group of standards, two resistors with nominal value 1 Ω and two 10 k Ω were identified as follows:

Table 1. Travelling resistors to be calibrated in the comparison.

<i>Standard</i>	<i>Manufacturer</i>	<i>Mod.</i>	<i>no.</i>
STD 1 Ω	CSIRO	NML	64200
STD 1 Ω	CSIRO	NML	64203
STD 10 k Ω	TEGAM	SR104 - B10K09	K205039730104
STD 10 k Ω	TEGAM	SR104 - B10K12	K201089830104

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



Fig. 1a, b – Unboxing and check

3. Climatic conditioning of the standards – oil and air baths

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. 2). The oil temperature is measured with a calibrated Leeds & Northrup 25 Ω s/ 177 3055 thermometer. The depth of the resistors in the oil, measured between the surface and the top of the standards was 32 mm as indicated in Fig. 3.



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

The 10 k Ω 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.

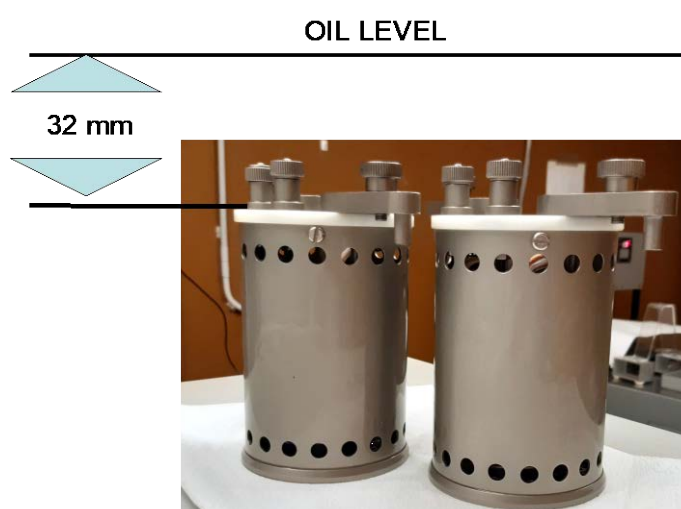


Fig. 3 - Oil immersion depth of the 1 Ω standards.

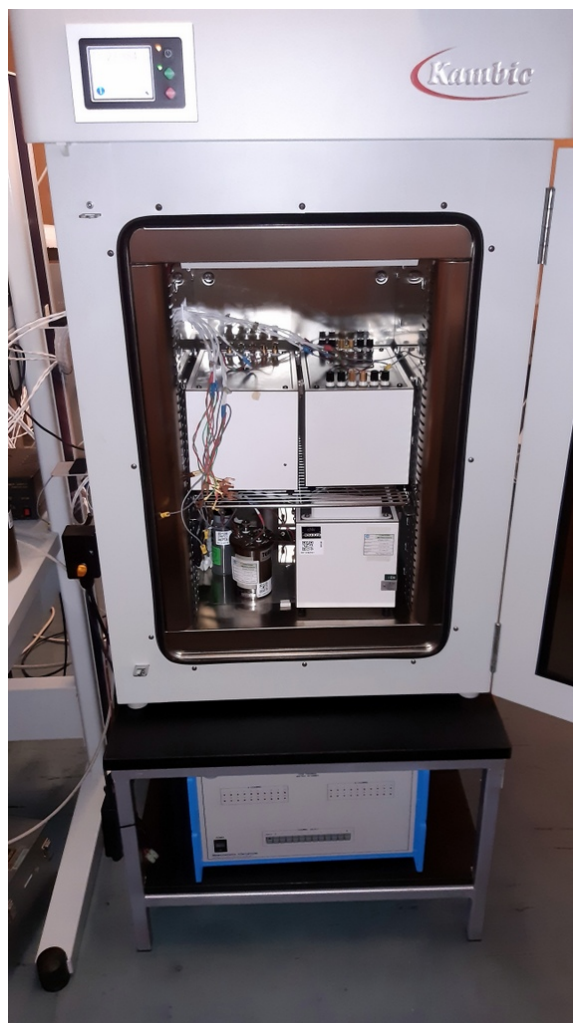


Fig. 4 - 10 k Ω standards air-bath. The resistors were placed inside a Kambic air-bath mod. 105 with a set-point of 23.000°C and a short-term stability of approximately 0.005°C. The temperature was monitored with a PT100 platinum thermometer.

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 by always carrying out two series of resistance measurements in succession and at two different current values (1 mA and 1.41 mA) and making the correction due to self-heating. In correspondence with the resistance measurements of the traveling standards, the atmospheric pressure was also acquired using a Barocap-Technology instrument.

4. Main instrumentation – Measurements system

The measurement system used by INRIM for this international comparison is described in detail in the INRIM technical procedure [2]. Nevertheless, the same system is still based on the one described in [3] with only little difference as the use of a new high-performance

automated CC bridge in place of the previous manually operated bridge. The group of INRIM working standards consists of the resistors in fig. 5. 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 origin of traceability is the QHE that is used for the calibration of a material transfer resistor, used for the calibration 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 Hamon and DCC networks, all the elements of the scale are calibrated. The first step of the 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 $U = 7 \times 10^{-7}$ 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 – 1k Ω – 10k Ω . Traceability is transferred to the 10 k Ω resistor via a 1k Ω step Hamon network, while the 1 k Ω resistor is calibrated against both 100 Ω and 10 k Ω , 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 k Ω standards of the INRIM working standard scale.

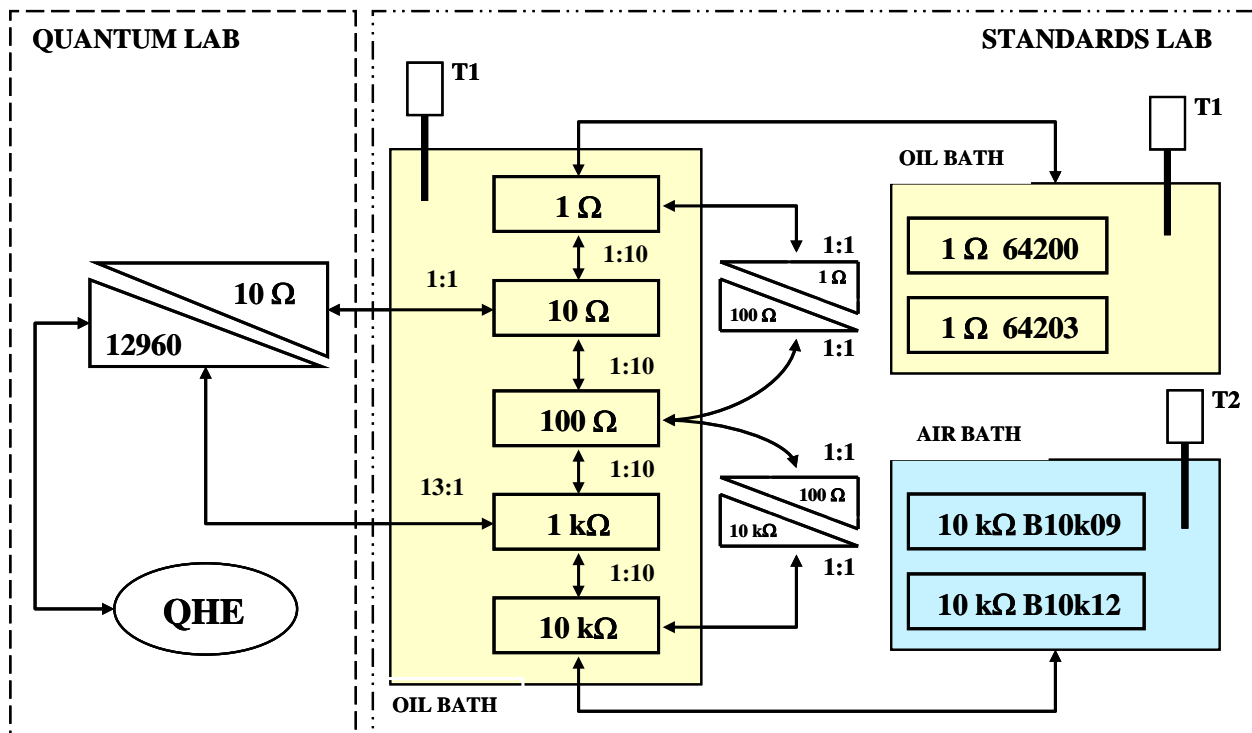
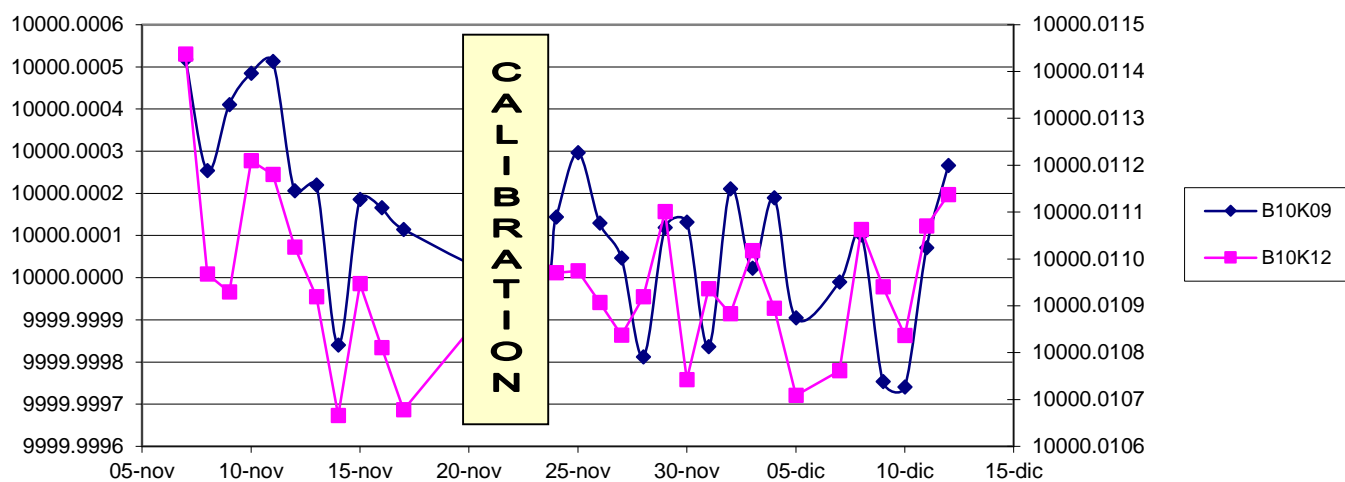
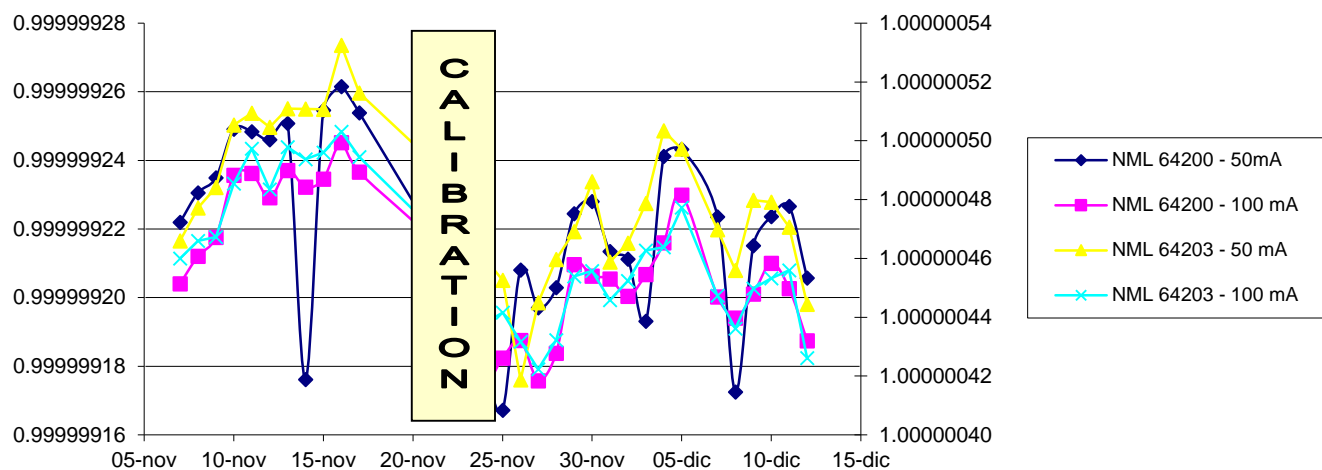


Fig. 5 - Complete diagram of the measurement system used for the K13 a&b key comparison. The traceability derives from the QHE realized in the INRIM Quantum laboratory and transferred to the decade scale in two ways: through a specifically designed Hamon network and by 1:13 ratio direct comparison, with a MI 6010Q DCC bridge. The working references and the two 1 Ω traveling resistors (k13) are kept at $T = 23.000 \pm 0.001^\circ\text{C}$ in a Guildline oil bath. All ESI resistors and 10 k Ω (K13) resistors were kept at $23.000 \pm 0.005^\circ\text{C}$ in a Kambic US-105 air bath.

5. Measurements, tables and graphs

The measurements were carried out every day, starting at 5:00 am when low level electrical and thermal noise and drift usually occur. The data of the measurements are contained in table 2. The data making up the average are those included between 10 November and 12 December. The measurements of the first few days were removed from the average because they were considered unstable, furthermore in the period between 18 and 23 November no measurements were made on the K13 standards due to the calibration of the INRIM working standards. The reference date of the k13 INRIM measurements is Monday November 21.



The final resistance values of the 1 and 10 k Ω standards are:

Table 2. Resistance values for the 1 Ω and 10 k Ω travelling resistors.

1 Ω

Standard	no.	Value	U (n Ω / Ω)
STD 1 Ω	64200 – 50 mA	0.99999922	139.0
STD 1 Ω	64200 – 100 mA	0.99999921	139.0
STD 1 Ω	64203 – 50 mA	1.00000048	139.0
STD 1 Ω	64203 – 100 mA	1.00000046	139.0

10 kΩ

<i>Standard</i>	No.	Value	U (nΩ/Ω)
STD 10 k Ω	K205039730104	10000.0001	170.4
STD 10 k Ω	K201089830104	10000.0109	170.4

6. Uncertainties analysis

Tables 3 and 4 report, for the 1 Ω and 10 kΩ respectively, the uncertainties budget taking into account the main components and following [4]. The highest type A uncertainty values, evaluated for the two resistors at each resistance level, were assigned to both of them.

Table 3 – Uncertainty budget for the 1 Ω resistors

	Source of uncertainties	Type	$u_r(\times 10^{-9})$	ν
Transfer $H_{SS} \rightarrow H_{SP}$	Link from R_{K-90} to Hamon 36×360 Ω ($H_{SS} = 12960 \Omega$)	B	35	∞
	H_{SS}/H_{SP} (10 Ω) ratio correction	B	17	∞
	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 $H_{SP} \rightarrow R_x 1 \Omega$	Ratio noise $H_{SP} - 1 \Omega$	A	3.14	50
	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	∞
	$u_c(R_x)$		69.5	

Table. 4 - Uncertainty budget for the 10 k Ω resistors

Source of uncertainties	Type	$u_r (\times 10^{-9})$	ν
Link from R_{K-90} to standard 100 Ω ESI	B	61.7	50
Ratio noise 100 Ω ESI/Hamon LN ₁₀₀ Ω	A	1.9	50
Ratio noise Hamon LN ₁₀ k Ω /10k Ω	A	5.9	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	B	3.12	∞
Resistors Johnson noise	B	0.74	∞
$u_c(R_x)$		85.2	

Acknowledgment

We would like to thank the BIPM team for the technical, scientific and logistical organization of the K13 comparison.

References

- [1] R. Goebel, Technical protocol for BIPM.EM-K13a&b comparisons, BIPM/ELEC-T-17, rev. 1.1, December 2015.
- [2] PT-EM.2.1-07, Calibration of 1 Ω \div 10 k Ω INRIM standard resistors rev.03, 2021.
- [3] 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.
- [4] JCGM 100:2008 Evaluation of measurement data - Guide to the expression of uncertainty in measurement First edition.

Annex

In table 5 the measurement results provided by INRIM to the BIPM for the comparison are listed along with the corresponding dates, temperature and pressure values.

Table. 5 –INRIM measurement results in Excel format along with the corresponding dates, temperature and pressure values.

INRUM EM_K13 a & b 2022																	
		07-nov	08-nov	09-nov	10-nov	11-nov	12-nov	13-nov	14-nov	15-nov	16-nov	17-nov	23-nov	24-nov	25-nov	26-nov	27-nov
NML 64200 - 50mA	0.999999222	0.999999231	0.999999235	0.999999249	0.999999248	0.999999246	0.999999251	0.999999176	0.999999255	0.999999262	0.999999254	0.999999201	0.999999181	0.999999167	0.999999208	0.999999197	
NML 64200 - 100 mA	0.999999204	0.999999212	0.999999218	0.999999236	0.999999236	0.999999229	0.999999237	0.999999232	0.999999235	0.999999245	0.999999237	0.999999206	0.999999177	0.999999182	0.999999187	0.999999176	
NML 64203 - 50 mA	1.000000466	1.000000477	1.000000484	1.000000505	1.000000509	1.000000505	1.000000511	1.000000511	1.000000511	1.000000532	1.000000516	1.000000482	1.00000046	1.000000453	1.000000419	1.000000445	
NML 64203 - 100 mA	1.00000046	1.000000466	1.000000467	1.000000485	1.000000497	1.000000483	1.000000498	1.000000494	1.000000496	1.000000503	1.000000494	1.000000459	1.000000438	1.000000442	1.000000432	1.000000422	
T oil bath (°C)	23.001	23.001	23.001	23.000	23.001	23.001	23.002	23.001	23.002	23.001	23.001	23.001	22.999	22.998	22.999	22.999	
P (hPa)	991.4	988.0	990.0	992.0	1005.0	1003.5	996.5	993.7	989.9	978.1	974.0	972.8	983.7	989.5	996.4	1000.0	
		07-nov	08-nov	09-nov	10-nov	11-nov	12-nov	13-nov	14-nov	15-nov	16-nov	17-nov	23-nov	24-nov	25-nov	26-nov	27-nov
B10K09	10000.00052	10000.00025	10000.00041	10000.00048	10000.00051	10000.00021	10000.00022	9999.99984	10000.00019	10000.00017	10000.00011	9999.999979	10000.00014	10000.0003	10000.00013	10000.00005	
B10K12	10000.01144	10000.01097	10000.01093	10000.01121	10000.01118	10000.01103	10000.01092	10000.01067	10000.01095	10000.01081	10000.01068	10000.01102	10000.01097	10000.01097	10000.01091	10000.01084	
T air bath (°C)	22.959	22.960	22.980	22.980	22.981	22.980	22.981	22.980	22.980	22.979	22.979	22.976	22.980	22.979	22.979	22.980	
P (hPa)	991.91	990.60	990.50	994.00	1005.81	1003.42	998.37	995.31	989.52	977.52	974.93	975.06	985.56	991.23	997.57	1000.00	

[illegible]