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ANALYSIS OF A NATIONAL INTER-LABORATORY COMPARISON OF HIGH DC RESISTANCE AT 10 MΩ AND 1 GΩ LEVEL

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Abstract

A national comparison of dc resistance at 10 MΩ and 1GΩ level was organized by the Electromagnetic Division of National Institute of Metrological Research (INRIM, Italy) and piloted by the same Division. This comparison took place between January and April of 2008 with the participation of 8 secondary Laboratories accredited by the Italian Accreditation of Calibration Laboratories Service (SIT). The travelling package included a wire-wound 10 MΩ standard and a thick film-type 1 GΩ standard in a wooden anti-shock container designed by INRIM. The obtained results indicate that the differences at 10 MΩ and 1 GΩ between each laboratory's value and its reference value are all within the expanded relative uncertainties of these differences.

1. Introduction

The interest in calibration of high value resistors in research and industrial fields recently increased due to the needs of traceable measurements in particular in companies with certified quality systems. On the other hand, an inter-laboratory comparison aims to verify the compatibility of the measures of two or more Laboratories performed on the same measurand. The measurement compatibility is a concept strictly connected with measurement uncertainty and plays a central role in the comparison of the measurement results of the same measurand, in well-defined conditions, obtained through measurement processes based on different methods and instruments or performed by different Laboratories. There are two typical situations in which a comparison can be performed: the first is among National Primary Metrological Laboratories belonging to different countries, the second among a National Primary Laboratory and some accredited Secondary Laboratories belonging to the same country. In this paper attention is paid to this second situation. In past years the Italian Accreditation of Calibration Laboratories Service (SIT) used to perform bilateral comparisons between the Italian Metrology Institutes and the Secondary calibration Laboratories to evaluate their technical competence in the framework of their first accreditation or in the periodical renewal of their accreditation as SIT Centres elaborating the results according to the ISO Guide 43. Nowadays, as the number of accredited Laboratories grew significantly, the possibility to perform bilateral comparisons became quite difficult. A possible solution for this problem could be the possibility that National Institute of Metrological Research (INRIM) operates as Inter-laboratory Comparisons (ILCs) provider [1, 2].

In this paper an example of the function that INRIM could assume is presented. The Electromagnetic Division of INRIM organized and piloted a national comparison of high dc resistance at 10 MΩ and 1GΩ level at which participated 8 SIT Centres. These Centres measured the two standard resistors of the comparison following their accredited measurement procedures or following new calibration procedures to be evaluated by SIT. The results of this comparison will be also utilized by SIT for the evaluation of the measurement competence of the participating SIT Centres in the field of high dc resistance.

2. Participating Laboratories

The participating Laboratories (SIT Centres) were the following (in alphabetical order):

- AGILENT TECHNOLOGIES ITALIA SIT Center no. 05;
- ARO s SIT Center no. 46;
- AVIATRONIK SIT Centre no. 19;
- FIRENZE TECNOLOGIA SIT Center no. 56;
- NEMKO S.p.A. SIT Center no. 42;
- SIMAV S.p.A. (Naples) SIT Center no. 14;
- SIMAV (Milan) SIT Center no. 04;
- SIMAV (Turin) SIT Center no. 64.

Table 1 lists the participant Laboratories in chronological order (not corresponding to alphabetical order) and the mean dates of their measurements on the standards with also the dates of the measurements at INRIM.

Table 1. List of participants and measurement dates.

Laboratory	**Mean date of measure**
INRIM	15/01/2008
LAB 1	22/01/2008
LAB 2	30/01/2008
LAB 3	07/02/2008
LAB 4	13/02/2008
LAB 5	21/02/2008
LAB 6	27/02/2008
INRIM	07/03/2008
LAB 7	14/03/2008
LAB 8	31/03/2008
INRIM	11/04/2008

3. Travelling standard resistors

3.1. Vishay-Mann – 10 MΩ Resistor

This standard (Fig. 1) is a Vishay component with a metallic body fixed to an aluminium support inside an aluminium cylinder, in which were made some holes to facilitate the thermal exchange, while in the central body a groove allows to insert a thermometer. In the original realization, the resistor is defined for two terminal resistance measure, but at INRIM a third terminal was added to allow also its use also as a three terminal standard. The resistor was calibrated by all Laboratories, including INRIM at a voltage of 10 V, according to their accredited (or submitted to accreditation) by SIT procedures. Some Laboratories measured the resistor also at different voltages but for the elaboration of the results were taken into account only the measurements at 10 V.

The 10 MΩ resistor showed, in the measurements performed by INRIM, a satisfactory stability(on the order of 5×10^{-7} in the time period of the comparison). So it can be consider irrelevant the uncertainty due to its drift and/or transport for the evaluation of the inter–laboratory comparison.

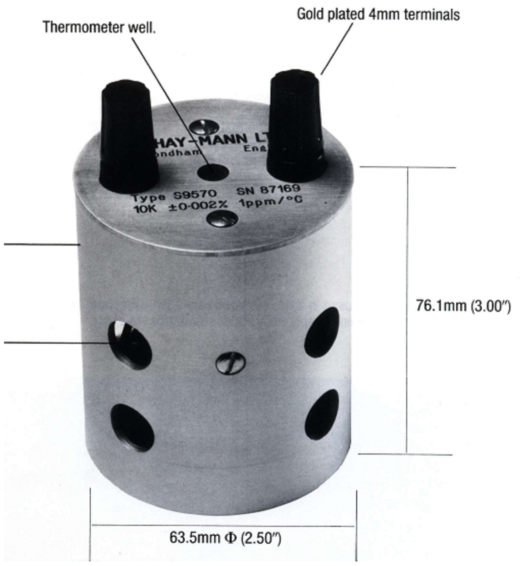

Fig. 1. The Vishay-Mann 10 MΩ.

3.2. INRIM HR1G – thermo-regulated resistor 1 GΩ

The 1 GΩ resistor was assembled at INRIM. The adopted resistive element was a commercially available thick film component with the following characteristics, as declared by the manufacturer: nominal value of 1 GΩ ± 0.25%, temperature coefficient $<5\times10^{-5}$/°C, voltage coefficient $<0.3\times10^{-6}$/V.

The resistor was projected to minimize the effects due to the variations of the environment parameters in order to improve its stability. Block diagram of the resistor is reported in Fig. 2. The standard is a three–terminal resistor. The resistive component was mounted inside a metal cylinder (Fig. 3) that represents both the thermal sensor and the heating system. The cylinder, electrically insulated from its electronic control, represents also the shield (third) terminal, and its potential could be also be controlled externally. These elements were placed in a hermetic container in an environment with dry nitrogen at environment pressure.

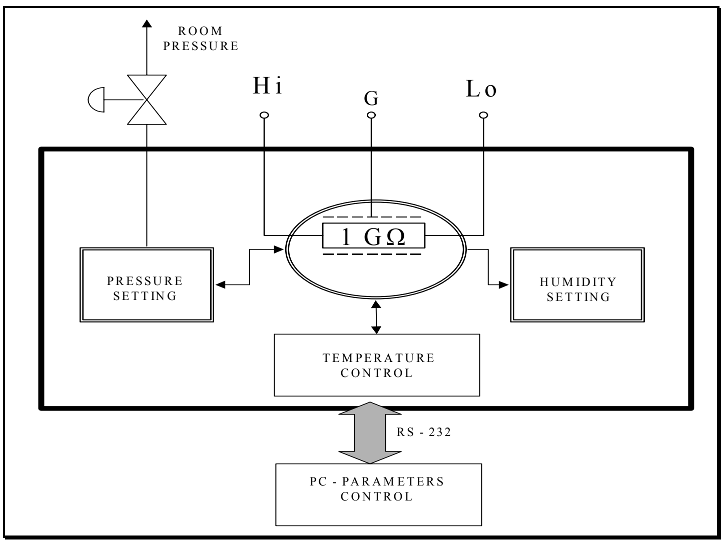

Fig. 2 . Block diagram of the 1 GΩ resistor.

The electronic circuit of the temperature control is external from the resistor container. The long term stability of the temperature inside the container can be maintained (25 ± 0.01)°C.

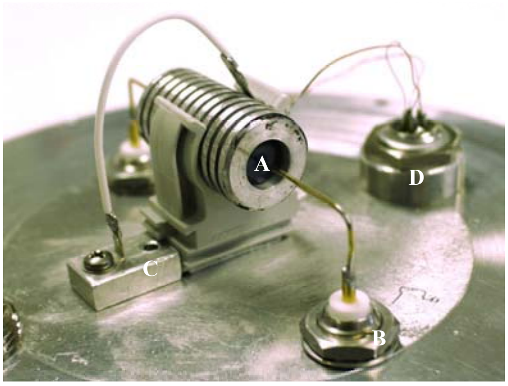

Fig. 3. View of the standard: the resistive element is placed inside the thermal–regulation cylinder (A) and soldered to the measurement terminals (B). In the picture are also visible: the third terminal (C) and the connector (D) used for temperature control.

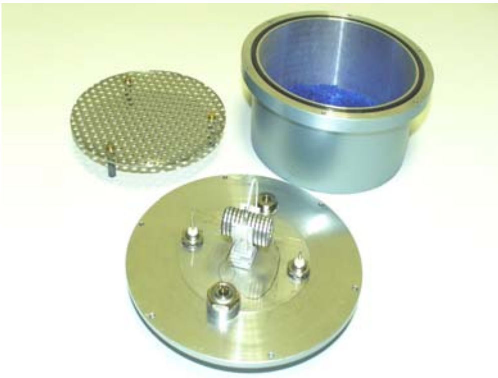

Fig. 4. The hermetic container of the resistor: is also visible the grid under which there is the silica gel for humidity control.

The silica gel that, as we can see in Fig. 4, is placed in the container separated from the resistor by means a very thin grid and allows to maintain the relative humidity to a level less than 10 %. Fig. 5 shows the whole system with the resistor and its external temperature control. For further details see [3].

Fig. 5. The 1 GΩ standard developed at INRIM with its external temperature control.

The resistor was calibrated by all the Laboratories at (100, 500, 1000) V and at two and three terminal configuration. In the time period of the comparison in the measurements performed by INRIM the 1 GΩ resistor showed a linear growing in time (on the (about 8×10^{-7}/day and of 6×10^{-7}/day in two and three terminal configuration respectively) behaviour. With the interpolation of the measurements of INRIM at the date of the measurements of the Laboratories it can be assumed as irrelevant the uncertainty due to the drift and/or transport of the resistor for the evaluation of the inter-laboratory comparison. The reference value for the 1 GΩ resistor at the day of the measurement of a specific Laboratory, is calculated as follows:

$$R = R_{nom} + \Delta R_{nom} + \frac{\Delta R_{drif}}{tot_{days}} \times no_{day} \,, \tag{1}$$

where R_{nom} = 1 GΩ is the nominal value of the resistor, ΔR_{nom} is the difference from nominal value measured by INRIM at the beginning of the comparison, ΔR_{drift} is the difference between the values of the resistor measured by INRIM respectively at the end and at the beginning of the comparison, tot_{days} is the total number of days of the inter-laboratory comparison (approximately 87) and no_{day} the number of the day in which a specific Laboratory made its measurement.

4. Measurement procedures

The Laboratories adopted their accredited (or submitted to accreditation) by SIT procedures and in particular for:

The 10 MΩ standard:

- reading directly to DMM: Centre 04;
- substitution with 10 MΩ standard resistor and auxiliary DMM: Centres 42, 46, 64;
- reading directly to Multifunction Transfer Standard: Centres 19, 56;
- DMM, dc voltage calibrator method [4]: Centres 05, 14, 19, 42, 64.

The 1 GΩ standard:

- DMM, dc voltage calibrator method for all Centres [4].

5. Statistical analysis of the results

To evaluate the measurement comparison the results of each measurement point were elaborated in the following way [5]:

The measurement values obtained by the Laboratories were defined as:

$$m_{Lab} \pm u_{Lab} \,. \tag{2}$$

While the results of INRIM were defined as:

$$m_I \pm u_I \,, \tag{3}$$

where u_{Lab} is the relative standard uncertainty declared by a Laboratory and u_I is the relative standard uncertainty associated to the value measured by INRIM.

A new measurand $y_{Labr}=(m_{Lab}-m_I)/m_I$ was defined for each measurement point, where m_I, for the 10 MΩ resistor, is the mean of the measurements of INRIM, while for the 1 GΩ is the interpolation of the measurement values of INRIM at the date of the measurement of the Labs. The relative standard uncertainty of this measurand is:

$$u^2_{y_{Lab,r}} = \frac{1}{m_I^2} \times [u^2_{Lab} + u^2_I - 2u_{Lab}u_I \times r(m_{Lab}, m_I)] \,, \tag{4}$$

where $r(m_{Lab},m_I)$ is the correlation factor between the measurements of the Laboratories and those of INRIM, evaluated as the ratio between the uncertainty with which INRIM calibrated the standards used by the Laboratories for the comparison, and the uncertainty declared by the same Laboratories for the comparison. Finally, for each measurement point and for each Laboratory, the degree of equivalence *DE* vs. INRIM was evaluated as:

$$DE = \frac{y_{Lab,r}}{U_{y_{Lab,r}}}, \quad (5)$$

where $U_{yLab,r} = 2u_{yLab,r}$ at 2σ level.

6. Measurement results

In Tables 2-9 the results of the inter-laboratory comparison are reported. In column 1 are reported the Laboratories, in column 2 the date of the measurements at the Laboratories, in column 3 the means or the interpolated values of the measurements m_I of INRIM, in column 4 the relative standard uncertainties u_I/m_I associated to these values, in column 5 the results m_{Lab} of the Laboratories, in column 6 the relative standard uncertainties u_{Lab}/m_{Lab} declared by the Laboratories, in column 7 the relative differences $y_{Lab,r}$ between the measurement, of the Laboratories and of INRIM, in column 8 the relative standard uncertainties $u_{yLab,r}$ of these differences and in column 9 the degree of equivalences *DE* according to (5).

Table 2. Results for the 10 MΩ resistor at 2 terminals.

Laboratory	**date**	m_I (MΩ)	u_I/m_I ($\times10^{-6}$)	m_{Lab} (MΩ)	u_{Lab}/m_{Lab} ($\times10^{-5}$)	y_{Lab},r ($\times10^{-6}$)	u_{yLab},r ($\times10^{-5}$)	***DE***
LAB 1	23/01/08	10.0010605	2.1	10.001056	0.4	–0.5	0.29	–0.08
LAB 2	29/01/08	10.0010605	2.1	10.00101	1.1	–5.1	1.1	–0.23
LAB 3	07/02/08	10.0010605	2.1	10.00111	3.5	4.9	3.4	0.07
LAB 4	13/02/08	10.0010605	2.1	10.00102	1.8	–4.1	1.6	–0.13
LAB 5	21/02/08	10.0010605	2.1	10.00109	1.3	2.9	1.1	0.13
LAB 6	27/02/08	10.0010605	2.1	*	*	*	*	*
LAB 7	14/03/08	10.0010605	2.1	10.00109	2.0	2.9	1.9	0.08
LAB 8	31/03/08	10.0010605	2.1	10.00104	1.0	–2.1	0.90	–0.11

Table 3. Results for the 10 MΩ resistor at 3 terminals.

Laboratory	**Date**	m_I (MΩ)	u_I/m_I ($\times10^{-6}$)	m_{Lab} (MΩ)	u_{Lab}/m_{Lab} ($\times10^{-5}$)	$y_{Lab}.r$ ($\times10^{-6}$)	u_{yLab},r ($\times10^{-5}$)	***DE***
LAB 1	23/01/08	10.0010756	2.1	*	*	*	*	*
LAB 2	29/01/08	10.0010756	2.1	*	*	*	*	*
LAB 3	07/02/08	10.0010756	2.1	*	*	*	*	*
LAB 4	13/02/08	10.0010756	2.1	10.00102	1.8	–5.6	1.6	–0.17
LAB 5	21/02/08	10.0010756	2.1	10.0011	5.0	2.4	1.1	0.03
LAB 6	27/02/08	10.0010756	2.1	10.00105	0.5	–2.6	0.4	0.37
LAB 7	14/03/08	10.0010756	2.1	10.00109	2.0	1.4	1.2	0.06
LAB 8	31/03/08	10.0010756	2.1	10.00107	1.0	–0.6	0.9	0.03

The boxes with * are relative to measurements not performed by the Laboratories.

Table 4. Results for the 1 GΩ resistor at 2 terminals at 100 V.

Laboratory	**Date**	m_I (GΩ)	u_I/m_I ($\times 10^{-6}$)	m_{Lab} (GΩ)	u_{Lab}/m_{Lab} ($\times 10^{-4}$)	$y_{Lab}.r$ ($\times 10^{-6}$)	$u_{yLab}.r$ ($\times 10^{-4}$)	***DE***
LAB 1	23/01/08	1.0027114	8.0	1.00272	2.5	8.6	2.5	0.02
LAB 2	29/01/08	1.0027177	8.0	1.00275	0.7	32	6.5	0.02
LAB 3	07/02/08	1.002724	8.0	1.0027	5.0	–24	5.0	0.02
LAB 4	13/02/08	1.0027285	8.0	1.002675[1]	2.0	–54	1.9	–0.14
LAB 5	21/02/08	1.0027357	8.0	1.002720	2.5	–16	2.5	–0.03
LAB 6	27/02/08	1.0027402	8.0	1.00274	1.1	–0.2	1.0	0.00
LAB 7	14/03/08	1.0027528	8.0	1.0028	1.8	47	1.8	0.13
LAB 8	31/03/08	1.0027672	8.0	1.00276	0.5	–7.2	0.4	–0.08

Table 5. Results for the 1 GΩ resistor at 3 terminals at 100 V.

Laboratory	**Date**	m_I (GΩ)	u_I/m_I ($\times 10^{-6}$)	m_{Lab} (GΩ)	u_{Lab}/m_{Lab} ($\times 10^{-4}$)	$y_{Lab}.r$ ($\times 10^{-6}$)	$u_{yLab}.r$ ($\times 10^{-4}$)	***DE***
LAB 1	23/01/08	1.0027231	8.0	1.00273	2.5	6.9	2.5	0.01
LAB 2	29/01/08	1.0027281	8.0	1.00273	0.9	1.9	8.5	0.01
LAB 3	07/02/08	1.0027332	8.0	1.0027	5.0	–33	5.0	0.03
LAB 4	13/02/08	1.0027368	8.0	1.0027089[1]	2.0	–28	1.9	–0.07
LAB 5	21/02/08	1.0027426	8.0	1.002740	2.5	–2.6	2.5	–0.01
LAB 6	27/02/08	1.0027463	8.0	1.00273	1.1	–16	1.0	–0.08
LAB 7	14/03/08	1.0027564	8.0	1.00275	1.0	–6.4	1.8	–0.03
LAB 8	31/03/08	1.002769	8.0	1.00274	0.5	–29	0.4	–0.33

Table 6. Results for the 1 GΩ resistor at 2 terminals at 500 V.

Laboratory	**Date**	m_I (GΩ)	u_I/m_I ($\times 10^{-6}$)	m_{Lab} (GΩ)	u_{Lab}/m_{Lab} ($\times 10^{-4}$)	$y_{Lab}.r$ ($\times 10^{-6}$)	$u_{yLab}.r$ ($\times 10^{-4}$)	***DE***
LAB 1	23/01/08	1.0026591	7.0	1.00269	9.0	31	9.0	0.17
LAB 2	29/01/08	1.0026667	7.0	1.00268	6.0	13	6.0	0.11
LAB 3	07/02/08	1.0026723	7.0	1.0027	50	28	50	0.03
LAB 4	13/02/08	1.002677	7.0	1.002650[1]	10	–27	9.4	–0.14
LAB 5	21/02/08	1.0026845	7.0	1.002680	25	–4.5	25	–0.01
LAB 6	27/02/08	1.0026892	7.0	1.002675	3.9	–14	3.5	–0.20
LAB 7	14/03/08	1.0027023	7.0	1.00271	6.0	7.7	5.8	0.07
LAB 8	31/03/08	1.0027173	7.0	1.00271	5.0	–7.3	4.5	–0.08

Table 7. Results for the 1 GΩ resistor at 3 terminals at 500 V.

Laboratory	Date	m_I (GΩ)	u_I/m_I (×10^{-6})	m_{Lab} (GΩ)	u_{Lab}/m_{Lab} (×10^{-4})	y_{Lab},r (×10^{-6})	u_{yLab},r (×10^{-4})	***DE***
LAB 1	23/01/08	1.0026752	7.0	1.00267	9.0	–5.2	9.0	–0.03
LAB 2	29/01/08	1.0026801	7.0	1.00267	6.0	–10	6.0	–0.08
LAB 3	07/02/08	1.002685	7.0	1.0027	50	15	50	0.02
LAB 4	13/02/08	1.0026885	7.0	1.0026821[1]	10	–6.4	9.4	–0.03
LAB 5	21/02/08	1.0026941	7.0	1.002690	25	–4.1	25	–0.01
LAB 6	27/02/08	1.0026976	7.0	1.002673	3.9	–25	3.5	–0.35
LAB 7	14/03/08	1.0027068	7.0	1.002708	2.3	1.2	2.2	0.03
LAB 8	31/03/08	1.0027194	7.0	1.00269	5.0	–29	4.5	–0.33

Table 8. Results for the 1 GΩ resistor at 2 terminals at 1000 V.

Laboratory	Date	m_I (GΩ)	u_I/m_I (×10^{-6})	m_{Lab} (GΩ)	u_{Lab}/m_{Lab} (×10^{-4})	y_{Lab},r (×10^{-6})	u_{yLab},r (×10^{-4})	***DE***
LAB 1	23/01/08	1.0025421	6.5	1.00256	7.0	18	7.0	0.13
LAB 2	29/01/08	1.0025512	6.5	1.00255	6.0	–1.2	6.0	–0.01
LAB 3	07/02/08	1.0025566	6.5	1.0026	50	4.3	50	0.04
LAB 4	13/02/08	1.0025611	6.5	1.002535[1]	10	–26	9.5	–0.14
LAB 5	21/02/08	1.0025683	6.5	1.002560	25	–8.3	25	–0.02
LAB 6	27/02/08	1.0025729	6.5	1.002558	2.8	4.5	5.3	0.04
LAB 7	14/03/08	1.0025855	6.5	1.00259	5.5	0.5	1.4	0.02
LAB 8	31/03/08	1.0026000	6.5	1.00256	5.0	–1.0	4.5	–0.11

Table 9. Results for the 1 GΩ resistor at 3 terminals at 1000 V.

Laboratory	Date	m_I (GΩ)	u_I/m_I (×10^{-6})	m_{Lab} (GΩ)	u_{Lab}/m_{Lab} (×10^{-4})	y_{Lab},r (×10^{-6})	u_{yLab},r (×10^{-4})	***DE***
LAB 1	23/01/08	1.0025589	6.5	1.00257	7.0	11	7.0	0.08
LAB 2	29/01/08	1.0025635	6.5	1.00255	6.0	–13	6.0	–0.11
LAB 3	07/02/08	1.0025682	6.5	1.0026	50	32	50	0.03
LAB 4	13/02/08	1.00257161	6.5	1.002565[1]	10	–6.2	9.5	–0.03
LAB 5	21/02/08	1.0025769	6.5	1.002556	25	–17	25	–0.03
LAB 6	27/02/08	1.0025803	6.5	1.002586	2.8	–24	2.5	–0.48
LAB 7	14/03/08	1.0025903	6.5	1.00257	5.5	–4.3	1.4	–0.15
LAB 8	31/03/08	1.0026010	6.5	1.00257	5.0	–31	4.5	–0.34

6.1. Graphical results

In Figs from 6 to 13 the measurements obtained by the 8 Laboratories and by INRIM are reported associated with their 2σ uncertainties.

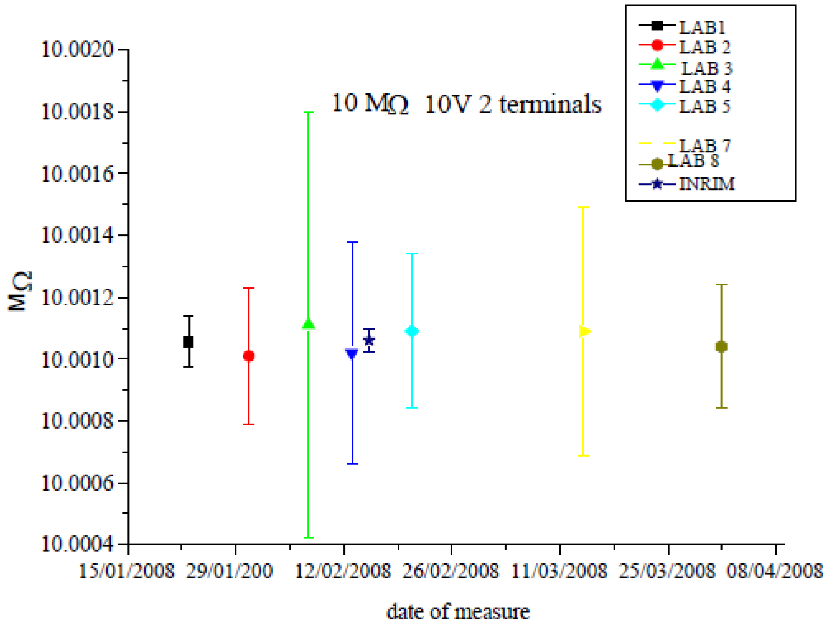

Fig. 6. Results for the 10 MΩ at 2 terminals at 10 V.

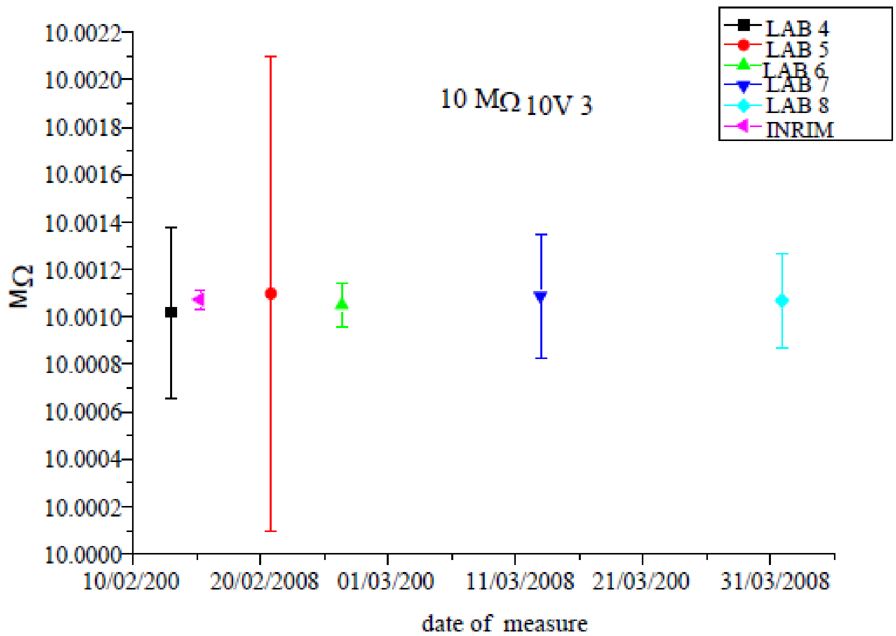

Fig. 7. Results for the 10 MΩ at 3 terminals at 10 V.

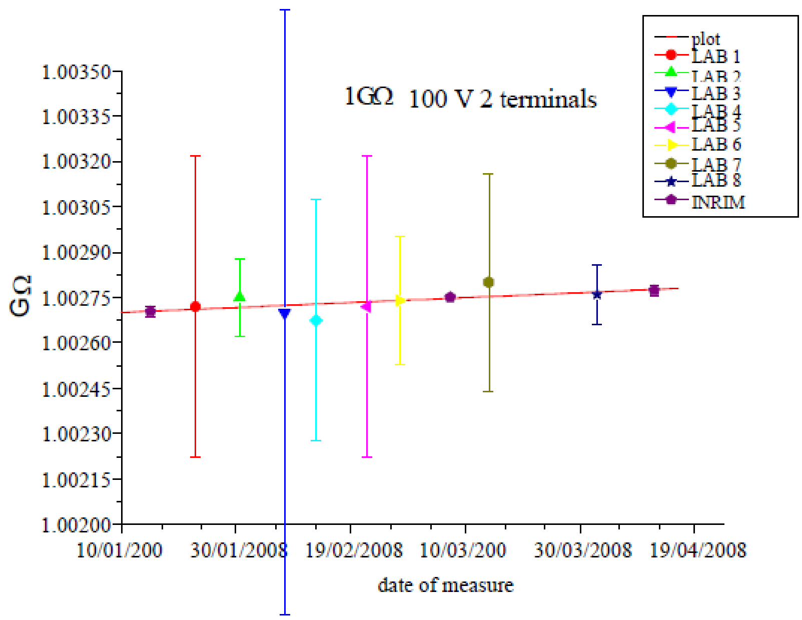

Fig. 8. Results for the 1 GΩ at 2 terminals at 100 V.

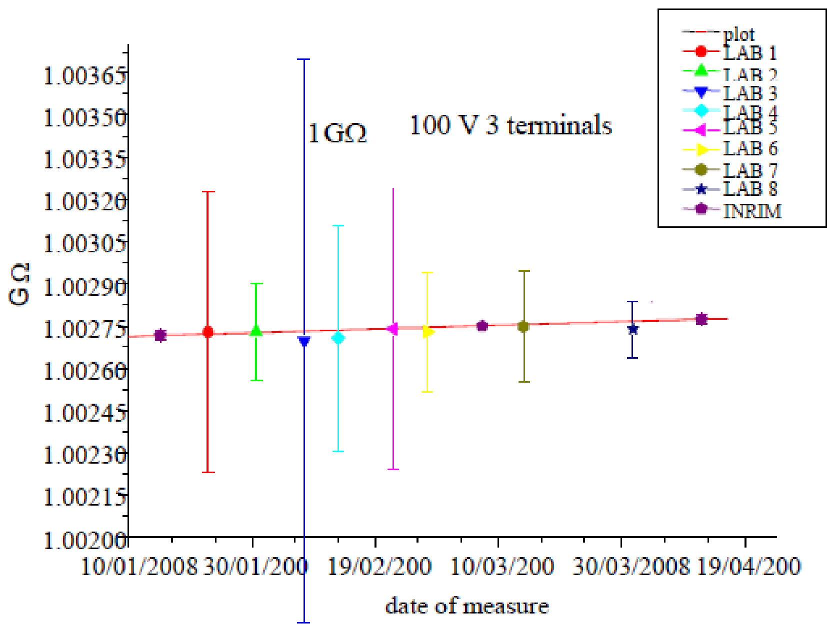

Fig. 9. Results for the 1 GΩ at 3 terminals at 100 V.

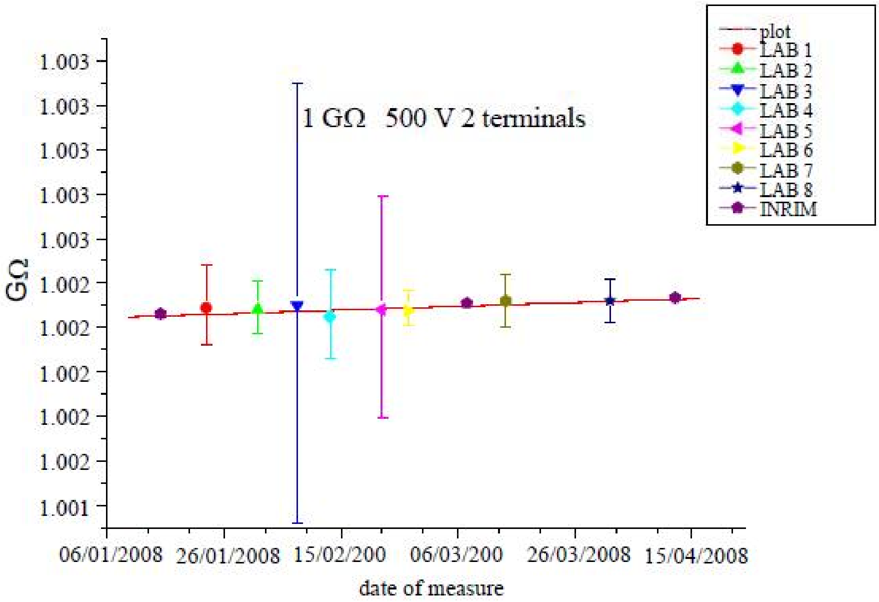

Fig. 10. Results for the 1 GΩ at 2 terminals at 500 V.

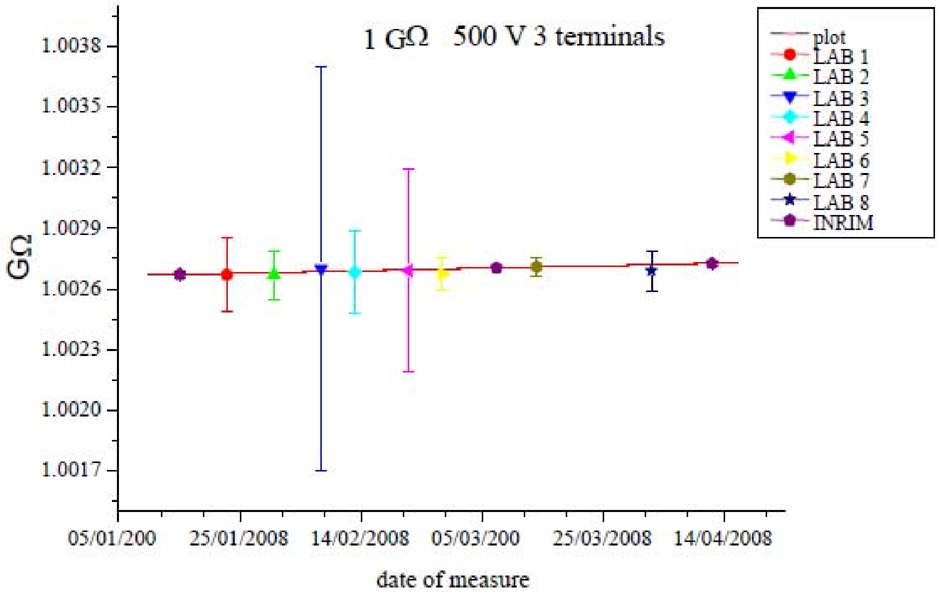

Fig. 11. Results for the 1 GΩ at 3 terminals at 500 V.

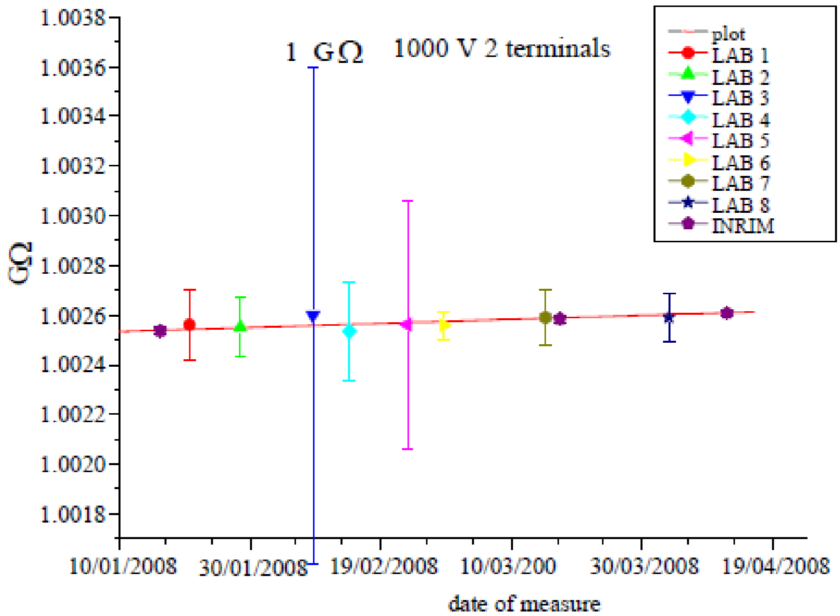

Fig. 12. Results for the 1 GΩ at 2 terminals at 1000 V.

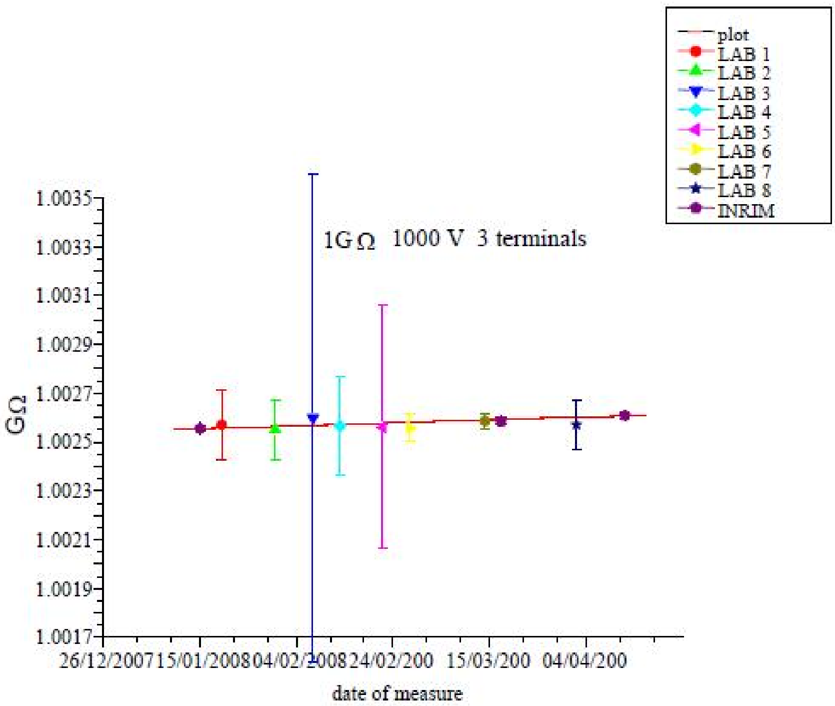

Fig. 13. Results for the 1 GΩ at 3 terminals at 1000 V.

7. Discussion

From Tables 2 to 9 the and from Figs 6 to 13 the degrees of equivalence ***DE*** resulted less than 1 for all the examined measurement. This result shows that the metrological dissemination process or traceability transfer from INRIM to the SIT Centres, and the accreditation criteria of the Italian Accreditation Service (SIT) in the field of high dc resistance are correct. On the other hand, during the comparison the travelling 10 MΩ resistor showed a very satisfactory stability while the 1 GΩ resistor showed a linear increasing behaviour but with a unsatisfactory drift, presumably due to a stabilization process after its opening before the comparison to clean its resistive component and to substitute its silica gel. Nevertheless it will be revised or replaced to increase its reliability. transportable standard.

8. Conclusions

This comparison can be considered a typical example of an inter-laboratory comparison among a National Metrological Laboratory and Secondary Laboratories of the same country. As a matter of fact in this comparison to determine the reference values were taken into account only the measurement values of INRIM, considered at a upper metrological level with respect the other Laboratories accredited by the Italian Accreditation Service. Another situation is instead described for example in [6] in which all the participating Laboratories in the comparison are Primary National Laboratories, so formerly treated at the same technical level. In the case the so called “consensus” values for each measurement point originate from the declared values and uncertainties of all the participating Laboratories. The plotted values of the pilot Laboratory are only utilized to analyze the behaviour of the involved travelling standards.

Acknowledgments

The authors wish to thank all the personnel of the 8 SIT Centres that participated at this inter-laboratory comparison for their courtesy and technical competence.

References

[1] ISO/IEC CD 17043 “Conformity *assessment* – General requirements for proficiency testing” *CASCO 05/2008, draft edition, 2008–03–14 previously ISO/IEC Guide 43–2:1997.*

[2] EA–2/10. EA Policy for Participation in National and International Proficiency Testing Activities, *rev.0, August 2001.*

[3] F. Galliana, P.P. Capra: “A temperature, humidity and pressure independent 1 GΩ standard resistor: realization and preliminary results”. *Proc. of the 12th IMEKO TC–4 Int. Symp. on Electrical Meas. Instrum.,* 25–27 September 2002, pp. 25-28.

[4] F. Galliana, P.P. Capra, E. Gasparotto: “Metrological management of the high dc resistance scale at INRIM”. *Measurement*, no. 42, 2009, pp. 314-321.

[5] BIPM, IEC, IFCC, ISO, IUPAC, IUPAP, OIML: Guide to the expression of uncertainty in measurement, *First edition, 1993, ISBN 92–67–10188–9.*

[6] R.F. Dziuba, D.G. Jarrett: “Final report on key comparison CCEM-K2 of resistance standards at 10 MΩ and 1 GΩ”. *Metrologia,* 39, 01001, doi:10.1088/0026–1394/39/1A/1.