Evaluation of Two Alternative Methods to Calibrate Ultrahigh Value Resistors at INRIM

Flavio Galliana, Pier Paolo Capra, and Enrico Gasparotto

***Abstract*—In the framework of the preparation of the National Institute of Metrological Research (INRIM) to its participation at the "Supplementary Comparison EURAMET.EM-S32 Comparison of Resistance Standards at 1 TΩ and 100 TΩ," an evaluation of two measurement techniques for high resistance measurements at INRIM has been made. The first method, working between 10 MΩ and 1 TΩ, is based on a digital multimeter and on a dc voltage (dcv) calibrator, the second, working up to 100 TΩ, is based on a modified Wheatstone bridge with two programmable dcv calibrators in two arms of the bridge and a programmable electrometer as null detector. A description of both methods, a detailed uncertainties budget at 1 TΩ at 1000 V level according to the ISO Guide to the Expression of Uncertainty in Measurements, and the comparison of the results at 100 GΩ and at 1 TΩ are reported.**

***Index Terms*—Compatibility of the measurements, guarding system, measurement model, measurement uncertainties, resistance measurement method, ultrahigh value resistor.**

I. Introduction

In the past years, in the field of high dc resistance, the National Institute of Metrological Research (INRIM), formerly the "Istituto Elettrotecnico Nazionale Galileo Ferraris" (IEN), developed and characterized a measurement method based on a digital multimeter (DMM) dc voltage (dcv) calibrator to be used for the calibration of standard resistors mainly in the range from 10 MΩ to1 TΩ[1]. This method is also suitable for determining the voltage coefficients of high-value resistors [2]. Using this method and a Hamon scaling method, [3] the IEN participated at a Consultative Committee for Electricity and Magnetism (CCEM) intercomparison on 10 MΩ and 1 GΩ values. The degrees of equivalence of IEN, expressed as differences from the reference values X_{KCRV} (Key Comparison Reference Value), resulted $0.9 \pm 5.5 \times 10^{-6}$ and $2.5 \pm 19.3 \times 10^{-6}$, respectively, at 10 MΩ and 1 GΩ[4].

Another well-known and reliable measurement method for the calibration of high-value resistors is the modified Wheatstone-bridge-based measurement system, developed at the National Physical Laboratory (NPL UK) [5] at the National Institute of Standards and Technology (NIST USA) in guarded [6] and with dual balance [7] versions and at the Physikalisch-Technische Bundesanstalt (PTB DE) [8]. In the following, the implementation of both methods at the INRIM in the framework of the Comparison EURAMET.EM-S32, a detailed uncertainties budget of the two methods according to the ISO Guide to the Expression of Uncertainty in Measurement at 1 TΩ at 1000 V level, a compatibility test between them and some adjunctive considerations are reported.

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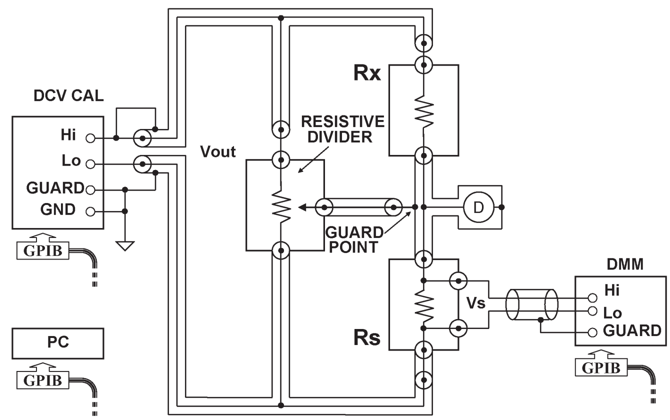

Fig. 1. Detailed scheme of the DMM-dcv calibrator measurement method.

II. DMM-DCV Calibrator Method

The DMM-dcv calibrator measurement method is usually utilized for the calibration of high-value resistors in the range 10 MΩ to 1 TΩ. A scheme of this measurement system is shown in Fig. 1. R_x is the resistor under calibration and R_s is the reference resistor. An auxiliary resistive divider provides a guard voltage. For this guard voltage, a Kelvin-Varley voltage resistive divider was actually utilized. Its ratio Z is always chosen so that $Z \cong R_x/R_s$. More details of the method are reported in [1].

The dcv calibrator supplies a positive voltage V_{out} to the series of R_x and R_s, while the DMM measures the voltage V_s on R_s. After a set number of measurements, the polarity of V_{out} is reversed and R_x is the mean value of R_{x+} and R_x. From Fig. 1, being V_x, the voltage on R_x and I_x the current through R_x and R_s, the value of R_x is given by

$$\begin{aligned} R_x &= \frac{V_x}{I_x} \\ &= (V_{out} - V_s)\frac{R_S}{V_s} \\ &= R_s\left(\frac{V_{out}}{V_S} - 1\right) \\ &\cong \left[\left(\frac{V_{out} + k_{cal-cal} + k_{Cal-acc}}{V_s + k_{DMM-cal} + k_{load} + k_{DMM-acc} + k_{term-bias}} - 1\right)\right. \\ &\quad \left.\times (R_S k_{drift} k_T)\right] k_{leak} k_{repeat} k_{Ax} \qquad (1) \end{aligned}$$

where

R_s is the reference resistor and

V_{out} is the voltage supplied by the dcv calibrator. As uncertainty contribution, its midterm instability, as declared by the manufacturer, was taken into account.

V_s is voltage on R_s read by the DMM. For this quantity, its type A standard uncertainty was taken into account.

$k_{\rm drift} \cong 1$ is the corrective term to take into account the drift of R_s;

$k_T \cong 1$ is corrective term to take into account the instability of R_s due to temperature instability;

$k_{load} \cong 0$ V is corrective term to take into account the error due to the DMM input impedance;

$k_{DMM-cal} \cong 0$ V is the corrective term to take into account the error in the calibration of the DMM;

$k_{DMM-acc} \cong 0$ V is corrective term to take into account the accuracy of the DMM;

$k_{cal-cal} \cong 0$ V is the corrective term to take into account the error in the calibration of the dcv calibrator;

$k_{cal-acc} \cong 0$ V is corrective term to take into account the accuracy of the calibrator;

$k_{term-bias} \cong 0$ V is corrective term to take into account the error of the DMM due to effects of bias current and of thermal and offset voltages residual to polarity inversion;

$k_{\rm leak} \cong 1$ is corrective term to take into account the error due to the leakages; and

$k_{\rm repeat} \cong 1$ is corrective term to take into account the repeatability of R_x. For this quantity its type A standard uncertainty was taken into account;

$k_{Ax} \cong 1$ corrective term to take into account the instability of R_x due to instability of environment conditions during the measurements.

A corrective term to take into account the voltage dependence of R_S was not inserted because in its characterization, $R_{\rm s}$ did not show any dependence from the applied voltage.

III. Modified Wheatstone Bridge Method

As shown in Fig. 2, in this modified Wheatstone bridge, two resistive arms are replaced by two programmable low-impedance voltage calibrators. The null detector is a picoammeter ideally considered with internal resistance $\cong 0$. The traceability of this method at INRIM starts from a 10 GΩ standard resistor calibrated with the DMM-dcv calibrator method and follows to higher decade values in the 1:10 step-up ratio.

The adopted automatic measurement procedure consists of setting the nominal direct voltages V_{xd} supplied by the Hi calibrator and V_{sd} supplied by the Lo calibrator so that $V_{xd}/(-V_{sd}) \cong R_x/R_s$. Then, V_{sd} is automatically adjusted to obtain close to 0 successively positive and negative values

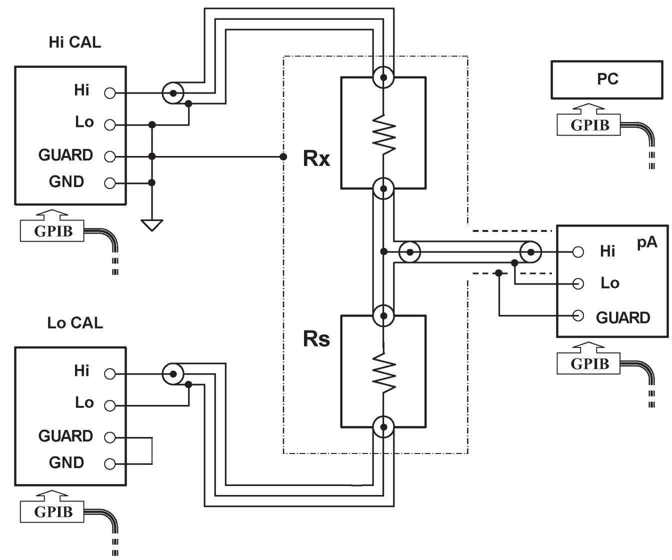

Fig. 2. Detailed coaxial setup of the modified Wheatstone bridge method.

$I(0+)$ and $I(0-)$ at the picoammeter at the voltages V_{sd1} and V_{sd2}, respectively. Then, with a linear fit, V_{sd} at $I = 0$ is evaluated. The procedure is then repeated for the reverse values V_{xr} and V_{sr} to evaluate V_{sr} at $I = 0$. Finally, V_x and V_{seq} at the bridge balance are evaluated as

$$V_{seq} = \frac{|V_{sd}| + |V_{sr}|}{2} \tag{2}$$

$$V_x = \frac{|V_{xd}| + |V_{xr}|}{2}. \tag{3}$$

The value of R_x is given by

$$R_x \cong R_s \left[(k_{drift} k_T k_V) \left(\frac{V_x + k_{calhi-acc} + k_{calhi-cal} - V_b}{(V_s + k_{callo-acc} + k_{callo-cal} - V_b} \right) \right] \times k_{\rm det} k_{\rm int} k_{repeat} k_{conn} k_{leak} k_{Ax} \tag{4}$$

where

R_s is the reference resistor and

V_x is the voltage supplied by the Hi calibrator. As uncertainty contribution, its midterm instability, as declared by the manufacturer, was taken into account.

V_s is the voltage supplied by the Lo calibrator at the balance of the bridge. For this quantity, its type A standard uncertainty was taken into account.

$k_{\rm drift} \cong 1$ is the corrective term to take into account the drift of R_s;

$k_T \cong 1$ is the corrective term to take into account the instability of R_s due to instability of temperature;

$k_V \cong 1$ is the corrective term to take into account the voltage dependence of $R_{\rm s}$;

$k_{\rm det} \cong 1$ is the corrective term to take into account the resolution of the detector;

TABLE I
UNCERTAINTIES BUDGET AT 1 TΩ AT 1000 V OF THE DMM–dcv CALIBRATOR METHOD

Quantity X_i	Estimate x_i	Standard unc. $u(x_i)$	Probab. distrib.	Sensitivity coeff. c_i	Uncertainty contrib. $u_i(R_x)$	Degrees freed. ν_i
R_s	≅ 10 MΩ	19.5 Ω	normal B	1.0×10^5	1.9×10^6 Ω	∞
$k_{\rm drift}$	≅ 1	5.8×10^{-8}	Rect B	1.0×10^{12} Ω	5.8×10^4 Ω	50
k_T	≅ 1	5.8×10^{-8}	Rect B	1.0×10^{12} Ω	5.8×10^4 Ω	50
k_{load}	≅ 0 V	5.8×10^{-8} V	Rect B	1.0×10^{14} Ω/V	5.8×10^6 Ω	50
$k_{DMM-cal}$	≅ 0 V	2.4×10^{-7} V	Rect B	1.0×10^{14} Ω/V	2.4×10^7 Ω	∞
$k_{DMM-acc}$	≅ 0 V	4.9×10^{-8} V	Rect B	1.0×10^{14} Ω/V	4.9×10^6 Ω	∞
$k_{cal-cal}$	≅ 0 V	5.2×10^{-3} V	Rect B	1.0×10^9 Ω/V	5.2×10^6 Ω	> 100
V_s	≅ 10 mV	1.2×10^{-8} V	Normal A	1.0×10^{14} Ω/V	1.2×10^6 Ω	∞
V_{out}	≅ 1000 V	3.8×10^{-4} V	Rect B	1.0×10^9 Ω/V	3.8×10^5 Ω	∞
$k_{cal-acc}$	≅ 0 V	1.8×10^{-3} V	Rect B	1.0×10^9 Ω/V	1.8×10^6 Ω	∞
$k_{term-bias}$	≅ 0 V	4.6×10^{-7} V	Rect B	1.0×10^{14} Ω/V	4.6×10^7 Ω	50
$k_{\rm leak}$	≅ 1	5.8×10^{-6}	Rect B	1.0×10^{12} Ω	5.8×10^6 Ω	∞
$k_{\rm repeat}$	≅ 1	1.2×10^{-6}	Normal A	1.0×10^{12} Ω	1.2×10^6 Ω	> 100
k_{Ax}	≅ 1	2.9×10^{-5}	Rect B	1.0×10^{12} Ω	2.9×10^7 Ω	50
R_x	≅ 1 TΩ					
$\boldsymbol{u_c(R_x)}$	Combined standard uncertainty				**6.1×10^7 Ω**	k = 2
	Effective degrees of freedom				> 100	
$\boldsymbol{U(R_x)}$	Expanded uncertainty 2 δ				**1.2×10^8 Ω**	

$k_{\rm int} \cong 1$ is the corrective term to take into account the interpolation of the readings of the detector; and

$k_{repeat} \cong 1$ is the corrective term to take into account the repeatability of R_x. For this quantity, its type A standard uncertainty was taken into account.

$k_{Ax} \cong 1$ is the corrective term to take into account the instability of R_x due to instability of environment conditions;

$k_{conn} \cong 1$ is the corrective term to take into account the error due to the connections;

$k_{\rm leak} \cong 1$ is the corrective term to take into account the error due to the leakages;

$k_{calhi-cal} \cong 0$ V is the corrective term to take into account the error in the calibration of the Hi dcv calibrator;

$k_{calhi-acc} \cong 0$ V is the corrective term to take into account the accuracy of the Hi dcv calibrator;

$k_{callo-cal} \cong 0$ V is the corrective term to take into account the error of the calibration of the Lo dcv calibrator;

$k_{callo-acc} \cong 0$ V is the corrective term to take into account the accuracy of the Lo dcv calibrator; and

V_b is the estimate of the voltage burden of the detector [9] and of the residual effects to the polarity inversion of offset and thermal voltages.

IV. DETAILED UNCERTAINTIES BUDGETS AT 1 TΩ LEVEL

The participation at the Supplementary Comparison EURAMET.EM-S32 was also the occasion to review the uncertainties of the methods with respect to those in [1]. As an example, in Tables I and II, detailed budgets according to the ISO Guide to the Expression of Uncertainty in Measurements [10] at 1 TΩ level at 1000 V, respectively, with the DMM-dcv calibrator method and with the modified Wheatstone bridge method, are reported. All input quantities are considered to be independent.

V. COMPARISON OF THE METHODS

The DMM and the three calibrators utilized in the two measurement setups were previously calibrated by the INRIM-Lab for calibration of high-precision multifunction instruments [11]. The calibration system of the INRIM-Lab consists of a group of reference standards such as a 10-V Zener voltage reference standard, two resistive voltage dividers, a set of standard resistors and shunts, and a programmable ac/dc voltage transfer standard, which are periodically calibrated versus the national standards. The INRIM-Lab typically calibrates high-stability DMMs, multifunction calibrators, and dc voltage calibrators, and them compares them with reference standards.

To check the compatibility between the two methods, one 100 GΩ and three 1 TΩ resistors were measured with both systems. The scheme of these resistors is shown in Fig. 3. In these resistors, the required resistance value is achieved by a

TABLE II
UNCERTAINTIES BUDGET AT 1 TΩ AT 1000 V OF THE MODIFIED WHEATSTONE BRIDGE METHOD

Quantity X_i	Estimate x_i	Standard unc. $u(x_i)$	Probability distribution	Sensitivity coeff. c_i	Uncertainty contribution $u_i(R_x)$	Degrees freed. ν_i
R_s	≅100 GΩ	3.7×10^{6} Ω	Normal B	10	3.7×10^{7} Ω	∞
k_{drift}	≅ 1	2.9×10^{-6}	Rect B	1.0×10^{12} Ω	$2.9.0\times10^{6}$ Ω	50
k_T	1	5.8×10^{-6}	Rect B	1.0×10^{12} Ω	5.8×10^{6} Ω	50
k_{det}	≅ 1	1.4×10^{-5}	Rect B	1.0×10^{12} Ω	1.4×10^{7} Ω	∞
k_{int}	≅ 1	2.9×10^{-6}	Rect B	1.0×10^{12} Ω	2.9×10^{6} Ω	5
k_V	≅ 1	1.1×10^{-5}	Rect B	1.0×10^{12} Ω	1.1×10^{7} Ω	5
k_{repeat}	≅ 1	5.0×10^{-5}	Normal A	1.0×10^{12} Ω	5.0×10^{7} Ω	> 100
k_{conn}	≅ 1	5.8×10^{-6}	Rect B	1.0×10^{12} Ω	5.8×10^{6} Ω	∞
k_{leak}	≅ 1	5.8×10^{-6}	Rect B	1.0×10^{12} Ω	5.8×10^{6} Ω	∞
k_{Ax}	≅ 1	2.9×10^{-5}	Rect B	1.0×10^{12} Ω	2.9×10^{7} Ω	50
$k_{calhi-cal}$	≅ 0 V	1.7×10^{-3} V	Rect B	1.0×10^{9} Ω/V	1.7×10^{6} Ω	∞
$k_{calhi-acc}$	≅ 0 V	1.3×10^{-3} V	Rect B	1.0×10^{9} Ω/V	1.3×10^{6} Ω	∞
V_x	≅ 1000 V	3.8×10^{-4} V	Rect B	1.0×10^{9} Ω/V	3.8×10^{5} Ω	∞
V_s	≅ −100 V	1.0×10^{-6} V	Normal A	1.0×10^{10} Ω/V	1.0×10^{4} Ω	> 100
$k_{callo-cal}$	≅ 0 V	1.7×10^{-6} V	Rect B	1.0×10^{10} Ω/V	1.7×10^{4} Ω	∞
$k_{callo-acc}$	≅ 0 V	1.0×10^{-3} V	Rect B	1.0×10^{10} Ω/V	1.0×10^{7} Ω	∞
V_b	≅ 20 µV	2.0×10^{-6} V	Rect B	9.0×10^{9} Ω/V	1.8×10^{4} Ω	∞
R_x	≅ 1 TΩ					
$u_c(R_x)$				combined standard uncertainty	**7.2×10^{7} Ω**	
		Effective degrees of freedom			> 100	
$U(R_x)$		Expanded uncertainty 2 δ			**1.4×10^{8} Ω**	**k = 2**

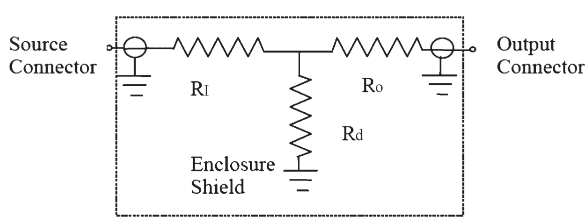

Fig. 3. Measured standards, one 100 GΩ and three 1 TΩ.

resistance network. Assuming that the output terminal and the shield are at the same potential, the resistance R_x between the source and output terminal is given by

$$R_x = R_i + R_0\left(1 + \frac{R_i}{R_d}\right). \quad (5)$$

With the DMM-dcv calibrator method, the shield of one terminal of the resistors under calibration and the shield of the reference resistor were connected to the guard point (Fig. 1), while with the modified Wheatstone bridge method, the shield of one terminal of both the reference and the under calibration resistors were connected to the ground (Fig. 2). To perform the measurements with the DMM-dcv calibrator method, an accurate balancing of the guard voltage annulling the residual voltage between the guard point and the low terminal of R_x by means of a null detector D and acting on the resistive divider (Fig. 1) was done. A 10 MΩ standard resistor was involved as R_s. Moreover, an accurate determination of the input impedance of the involved DMM, which resulted better than 1×10^{12} Ω, was done.

In the modified Wheatstone bridge method, the reference and under calibration resistors were both placed in a metallic shield box (dashed lines in Fig. 2). The measurements with both systems were carried out in the INRIM resistance laboratory, normally regulated at (23 ± 0.3) °C and with relative humidity at $(40 \pm 10)\%$. The reference and under calibration resistors of both systems were placed in air thermostatic baths at a temperature of (23 ± 0.1) °C. Both measurement setups were managed by battery-supplied portable computers to minimize noise. The mean values of the measurements are shown in Figs. 4–7.

The results shown in Figs. 4–7 are in good agreement within the respective uncertainties. The agreement must be taken with some care since there is some degree of correlation between the two methods (essentially due to the calibration of the 10 GΩ resistor). A full evaluation of the uncertainty of such comparison would require one to take into account such correlation, which is beyond the aim of this paper. A full validation of the methods will come from the results of the Comparison EURAMET.EM-S32 Comparison of Resistance Standards at 1 TΩ and 100 TΩ.

With the DMM-dcv calibrator method, the spread of the measurements significantly increased lowering the applied measurement voltage with respect to the other method, as the voltage that the DMM reads on the reference resistor decreases.

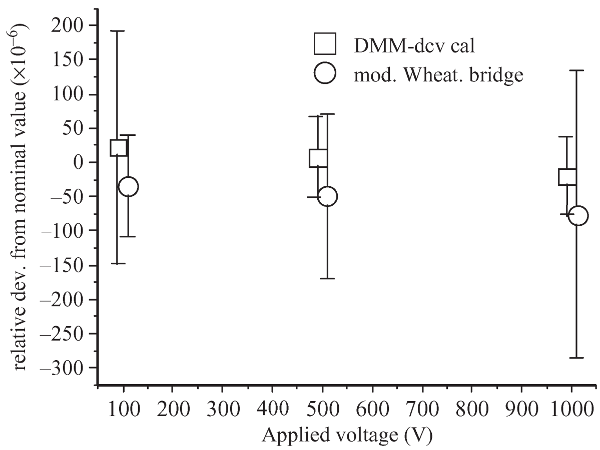

Fig. 4. Results on the 100 GΩ resistor with both methods. The measurements were carried out at 100 V, 500 V, and 1000 V, but in the graph are reported at little different voltages between the two methods to better distinguish their 2σ uncertainty bars.

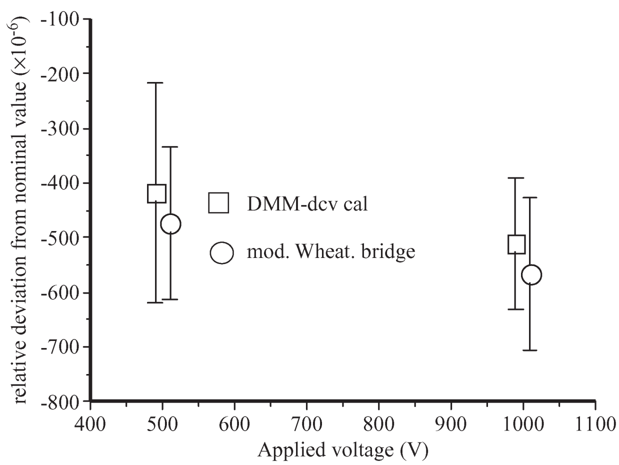

Fig. 5. Results on the first 1 TΩ resistor with both methods. The measurements were carried out at 500 V and 1000 V, but in the graph are reported at little different voltages between the two methods to better distinguish their 2σ uncertainty bars.

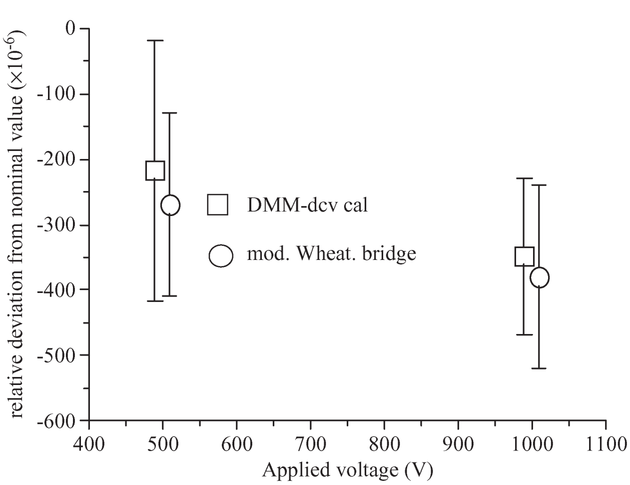

Fig. 6. Results on the second 1 TΩ resistor with both methods. The measurements were carried out at 500 V and 1000 V, but in the graph are reported at little different voltages between the two methods to better distinguish their 2σ uncertainty bars.

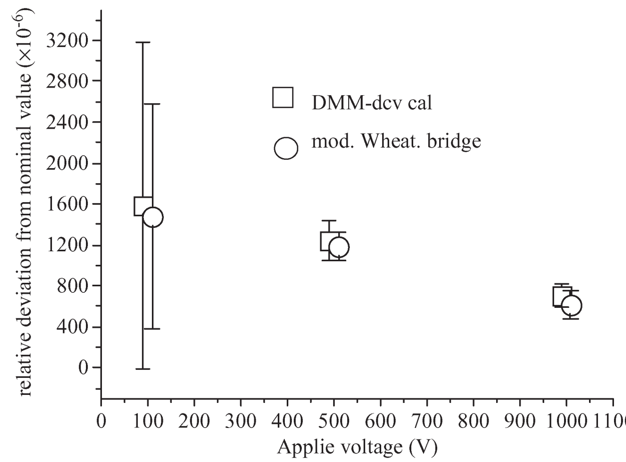

Fig. 7. Results on the third 1 TΩ resistor with both methods. The measurements were carried out at 100 V, 500 V, and 1000 V, but in the graph are reported at little different voltages between the two methods to better distinguish their 2σ uncertainty bars.

Another interesting fact is that in all the measurements, the results with the modified Wheatstone bridge method have lower values with respect to those with the other method. Presumably, the reason is that the DMM-dcv calibrator method is a fully guarded method, while in the other method, the high side of the detector is not guarded as in [6], [7]. As a matter of fact, guarding the high side of the detector reduces leakages at that point, which could cause a little shift of the balance of the bridge, thereby causing lower measured resistance values.

VI. Conclusion

The evaluation of two alternative measurement methods for the calibration of ultrahigh value resistors involved at the INRIM for the measurement of the Supplementary EURAMET.EM-S32 comparison was reported. The comparison between the two methods showed satisfactory agreement between them. Future aim will be implementation of a full guarding system for the modified Wheatstone bridge method to extend the INRIM dc resistance capabilities up to 1–10 PΩ.

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