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Metrological management of the high dc resistance scale at INRIM

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ABSTRACT

In the framework of the revision of the high dc resistance scale from 10 kΩ to 1 TΩ at National Institute of Metrological Research (INRIM) a measurement method for calibration of high value resistors based on the use of a digital multimeter (DMM) and of a dc voltage calibrator (DCVC) was projected, developed and characterized. A method based on the Hamon scaling technique was revised and extended to 1 GΩ. Two other methods, based respectively on a modified Wheatstone bridge and on a current comparator bridge, were implemented. A new 100 MΩ step Hamon standard, a humidity generator to characterize high value resistors vs. relative humidity and a scanner for high resistances were developed and characterized. The relative 2σ best measurement capabilities, obtained in the range 10 kΩ ÷ 1 TΩ at INRIM with all these methods, span from 2.0×10^{-7} of the 10 kΩ standard resistor at the measurement voltage of 10 V to 1.0×10^{-3} of the 1 TΩ standard resistor at a voltage of 100 V. The relative standard uncertainties of the utilized methods at INRIM are summarized.

1. Introduction

The interest in calibration of high value resistors in research and industrial fields has recently increased due to the needs of traceable measurements in particular in companies with certified quality systems. The National Institute of Metrological Research (INRIM), formerly Istituto Elettrotecnico Nazionale "G. Ferraris" (IEN), developed and characterized a measurement method for calibration of high value resistors mainly in the range 10 MΩ ÷ 1 TΩ based on the use of a digital multimeter (DMM) and of a dc voltage calibrator (DCVC) [1,2]. This method is also suitable for the determination of the voltage coefficients of high value resistors [3]. Using this method and a Hamon scaling method [4] IEN participated a Comité Consultatif d'Electricité (CCE) inter-comparison 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), were $(0.9 \pm 5.5) \times 10^{-6}$ and $(2.5 \pm 19.3) \times 10^{-6}$, respectively, for the 10 MΩ and 1 GΩ [5]. The results of this comparison show that the utilized DMM–DCVC and the Hamon scaling methods at INRIM (described, respectively, in Sections 3.1 and 3.2) agree satisfactorily at international level with the evaluated uncertainties. Moreover, this result means that the process of the transfer of the dc resistance unit from the national standard (1 Ω primary group of standard resistors referred to the value $R_{\mathrm{K\text{-}90}}$ of the von Klitzing constant) to high resistance at INRIM can be consider correct.

The modified Wheatstone bridge based measurement system, developed at National Physical Laboratory (NPL UK) [6], successively at National Institute of Standards and Technology (NIST USA) [7] and at Physikalisch-Technische Bundesanstalt (PTB Germany) [8], has also been implemented at INRIM. Another measurement method, based on the Hamon scaling technique, was already implemented at INRIM to transfer the resistance unit from a high-precision 10 kΩ resistor up to 100 MΩ [1,2]. Recently an extension of the range of this method to 1 GΩ was performed. To reach this aim a 100 MΩ step Hamon network

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with guarding system was developed [9]. With this transfer standard it is also possible to decrease the uncertainty of the maintained 1 GΩ standard at INRIM.

2. Standard resistors employed

An evaluation of the characteristics of the resistors employed in the resistance scale from 100 kΩ to 1 TΩ was already made [1]. As the characteristics of the resistors from 10 GΩ to 1 TΩ were not completely satisfactory (in particular of the 1 TΩ resistor), three new resistors with better characteristics were employed. Their temperature coefficients were determined by placing them in an air temperature controlled bath whose temperature can be varied from 16 to 30 °C. The resistance values at (19, 21, 23, 25, 27) °C and at a measurement voltage of 1000 V were determined. The voltage coefficients were determined at 23 °C by placing the resistors in an air temperature and humidity controlled bath (described in Section 4.2). The resistance values of the three resistors were determined at (250, 500, 750, and 1000) V. All these measurements were performed by means of the DMM–DCVC based method. The measurement results were fitted with the least squares method. Table 1 reports the evaluated temperature and voltage coefficients with their 2σ uncertainties due to the fit.

The standard resistors of the resistance scale at INRIM from 100 kΩ to 1 GΩ are kept permanently in an air temperature-controlled bath with long-term temperature of (23 ± 0.02) °C [10], while higher value resistors up to 1 TΩ are placed in another air temperature-controlled bath with mid-term temperature of (23 ± 0.01) °C.

3. Measurement methods for calibration of high value resistors at INRIM

3.1. The DMM–DCVC based measurement system

This measurement method can be involved for calibration of high value resistors from 100 kΩ to 1 TΩ, but at INRIM is utilized mainly in the range 10 MΩ ÷ 1 TΩ. A scheme of the measurement system is shown in Fig. 1. R_x is the resistor under calibration and R_s is the reference standard resistor. A calibrated DCVC supplies a voltage V_{out} to the series of R_x and R_s while a DMM measures the voltage V_s on R_s. In our practice, typical values for R_s are 1 MΩ and 10 MΩ, respectively, for the calibration of resistors from 10 MΩ to 10 GΩ and from 100 GΩ to 1 TΩ. Consequently the possible ratios between R_x and R_s. are comprised between 10:1 and 10^5:1. The measure of V_s is affected by systematic errors due to:

- the input impedance of the DMM that shunts R_s [11,12];
- the voltage due to the input bias current of the DMM in the resistance made by R_s in parallel with the input impedance of the DMM;
- the thermal voltages.

These two kinds of voltage are often unstable and algebraically added to V_s. The polarity of V_{out} is reversed in order to minimize their effects. An auxiliary resistive divider, indicated in Fig. 1 with two resistors R_A and R_B, provides a guard voltage. R_A and R_B are chosen so that $R_A/R_B = R_x/R_s$ and with nominal values at least three orders lower than R_x and R_s. This guard voltage minimizes the leakage currents in the insulation resistances $R_{L1,2}$ (drawn with dashed lines in Fig. 1). To provide this guard voltage a Kelvin–Varley voltage divider can also be utilized. The adopted remote controlled measurement sequence inserts, after applying V_{out}, a waiting time before measuring V_s. This time reduces the transients effects due to the shunt capacitance of the DMM and to the dielectric absorption in the insulator material of R_x. 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:

$$R_x = \frac{V_x}{I_x} = V_x\frac{R_s}{V_s} = (V_{out} - V_s)\cdot\frac{R_s}{V_s} = \left(\frac{V_{out}}{V_s} - 1\right)\cdot R_s \cong \left[\left(\frac{V_{out} + v_1 + v_2}{V_s + v_3 + v_4 + v_5 + v_6} - 1\right)\cdot R_s\right] + v_7 \quad (1)$$

where:

$v_1 \cong 0$ is a corrective term to take into account the error in the calibration of the DCVC;

$v_2 \cong 0$ is a corrective term to take into account the deviation of the supplied voltage by the DCVC from the set voltage;

$v_3 \cong 0$ is a corrective term to take into account the error in the calibration of the DMM;

$v_4 \cong 0$ is a corrective term to take into account the deviation of the voltage measured by the DMM with respect to the applied voltage;

$v_5 \cong 0$ is a corrective term to take into account the error of the DMM due to its input impedance in parallel with R_s;

$v_6 \cong 0$ is a corrective term to take into account the error of the DMM due to the thermal voltages and DMM bias current residual effects;

$v_7 \cong 0$ is a corrective term to take into account the error due to the leakages.

A detailed budget of the relative type A and type B standard uncertainties in the range 10 MΩ ÷ 1 TΩ at a mea-

Table 1
Temperature and voltage coefficients for the standard resistors 10 GΩ, 100 GΩ and 1 TΩ with their 2σ uncertainties due to the fit of the measurements results

Resistor	Temperature coefficient				Voltage coefficient at 23 °C	
	α	Relative uncertainty	β	Relative uncertainty	Value	Relative uncertainty
10 GΩ	-2.0×10^{-5}/°C	3.9×10^{-7}	1.0×10^{-6}/°C^2	1.6×10^{-7}	Negl.	
100 GΩ	-1.0×10^{-5}/°C	8.7×10^{-8}	1.6×10^{-7}/°C^2	3.7×10^{-8}	-4.8×10^{-8}/V	1.8×10^{-8}
1 TΩ	-2.0×10^{-5}/°C	4.9×10^{-6}	5.7×10^{-6}/°C^2	1.9×10^{-6}	-9.0×10^{-7}/V	8.4×10^{-8}

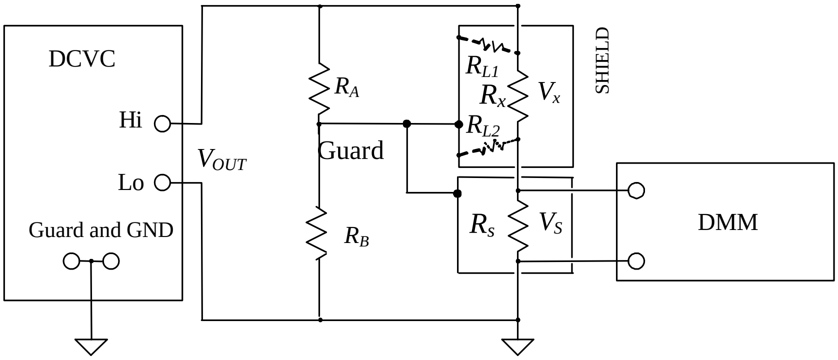

Fig. 1. Scheme of the DMM–DCVC measurement method.

surement voltage of 100 V is reported in Table 2. In this Table the uncertainty contributions $u_{xi}(R_x)$ to the standard uncertainty of R_x, due to each input quantity in Eq. (1) are reported. $u_{xi}(R_x)$ are obtained multiplying the standard uncertainties $u(x_i)$ of each input quantity in (1) for their sensitivity coefficients c_i [13]. The uncertainties of the corrective terms $v_{1,..,7}$ are taken into account. Further details are reported in [1,2].

3.2. Hamon scaling method

The traceability chain of this method is shown in Fig. 2. It starts from a high performance 10 kΩ standard resistor with temperature coefficients $\alpha_{23} = -3.4 \times 10^{-8}$/°C, $\beta = -2.9 \times 10^{-8}$/°C^2 and drift on the order of 7.5×10^{-8}/year [14]. This resistor is calibrated at a 2σ relative uncertainty of 2.0×10^{-7}in terms of the INRIM 1 Ω primary group of standard resistors referred to the recommended value $R_{K\text{-}90}$ of the von Klitzing constant. With two 10 × 10 kΩ and 10 × 100 kΩ transfer boxes, both configured to measure their resistors individually or in series[3], two high performance 100 kΩ and 1 MΩ standard resistors are calibrated. In the same way the parallel outputs of a 1 MΩ step and of a 10 MΩ step Hamon boxes are calibrated. The series outputs of these two boxes are compared with two high performance 10 MΩ and 100 MΩ standard resistors respectively. Moreover, with the series output of the 1 MΩ step box, the parallel output of a developed 100 MΩ step Hamon network (described in Section 4.1) is calibrated [9]. All the comparisons among each pair of resistors are made in four terminal configuration, in 1:1 ratio to the DMM and following the indications of the user manual of the DMM for the guard connection. the series output 100 MΩ step Hamon network is compared with a high performance 1 GΩ standard resistor at 100 V by substitution through the DMM–DCVC method. In Table 3 the relative standard uncertainties budget for the Hamon scaling from 10 kΩ to 1 GΩ is reported in detail. In Table 4 the type B and type A relative standard uncertainties for each decade value from 100 kΩ to 1 GΩ are listed. In Fig. 2 the relative total 2σ uncertainties for the same steps are reported. Further details are given in [1,2,9].

3.3. Current comparator bridge and modified Wheatstone bridge based methods

The current comparator method involves a commercial automatic dc current comparator bridge which has a voltage generator for the comparisons of high value resistors in 1:10 ratio up to 1 GΩ [1].

The reliable and accurate method based on a modified Wheatstone bridge, developed at NPL [6], at NIST [7] and at PTB [8], was also implemented at INRIM in some measurement points to check the measurement results obtained with the DMM–DCVC method.

3.4. Measurement uncertainties

Table 4 reports the relative standard uncertainties at different measurement voltages of each of the methods for calibration of high value resistors developed at INRIM. The type B uncertainties were estimated considering the utilization of a DMM whose input bias current is <1 pA for the 100 mV and 1 V ranges, <10 pA for the 10 V range while input impedance is about 10^{12} Ω for the three considered ranges. The type A uncertainties were evaluated from repeated measurements performed on the resistors of the INRIM high resistance scale.

4. Developed standard and devices

4.1. Guarded 100 MΩ step Hamon network

In Fig. 3 a scheme of the Hamon network developed at INRIM is shown. It is formed by 10 main 100 MΩ resistors and by a guard system consisting of a chain of ten 10 MΩ resistors mounted on the body of the main resistors by means of two copper rings (Fig. 4a). This guard system is referred to an effective guard system for high value Hamon transfer standards described in [15]. Moreover, in our network a guard driver circuit sets the voltage of the low side of the guard chain at the same value of the low side of the

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[3] Each resistor of the box is firstly compared with the equal nominal value standard, then the 10 resistors are connected in series. The series is compared in 1:1 ratio with another resistor performing the 1:10 unit transfer.

Table 2
Relative type A and type B[a] standard uncertainties in the range 10 MΩ ÷ 1 TΩ at a measurement voltage of 100 V for the DMM–DCVC based method

Source of uncertainty		Nominal resistance					
		10 MΩ	100 MΩ	1 GΩ	10 GΩ	100 GΩ	1 TΩ
Type A relative standard uncertainties ($\times 10^{-6}$)							
Std dev. of $R_x \cong u_{\text{Vout/Vs}}(R_x)$		0.7	1.1	0.7	1.2	118	17
Type B relative standard uncertainties ($\times 10^{-6}$)							
$u_{Rs}(R_x)$	Cal, temp. inst. drift of R_s	0.9	1.0	1.0	1.0	2.0	2.0
$u_{v1}(R_x)$	Cal of DCVC	3.0	3.0	3.0	3.0	3.0	3.0
$u_{v2}(R_x)$	Accuracy of DCVC	3.5	3.5	3.5	3.5	3.5	3.5
$u_{v3}(R_x)$	Cal of DMM	3.3	3.1	5.0	10	10	10
$u_{v4}(R_x)$	Accuracy of DMM	3.0	3.1	3.8	6.0	6.0	29
$u_{v5}(R_x)$	DMM input impedance	0.9	1.0	1.0	1.0	10	10
$u_{v6}(R_x)$	Thermal voltages and bias curr. residual effects	0.01	0.1	1.0	10	50	500
$u_{v7}(R_x)$	Leakages	0.05	0.1	0.1	1.0	1.0	10
RSS Total of type B relative standard uncertainties		6.5	6.5	8.0	16	53	500

[a] $u_{xi}(R_x)$ are the uncertainty contributions to the standard uncertainty of R_x due to the input quantities in Eq. (1) obtained multiplying the standard uncertainties $u(x_i)$ of each input quantity for the sensitivity coefficients c_i [13].

chain of the main resistors. The network of resistors is kept in a aluminium cylindrical case. The BNC connectors for the series and the binding post terminals for the parallel connections are, respectively, placed on the top and on the bottom of the case. The switch between series and parallel configurations is done by means of a mobile element with telescopic electrical contacts. All utilized connectors and cables are coaxial ones. A 10 kΩ thermistor is placed inside the case to verify the stability of the temperature during the transfer. The 10 main resistors are commercially available thick film-type resistance elements with average temperature coefficients $\alpha_{23} = 3.3 \times 10^{-6}$/°C and $\beta = 5.4 \times 10^{-7}$/°C^2. The voltage coefficient of the series of the main resistors is negligible (on the order of $-1{,}2 \times 10^{-8}$/V). Fig. 4a and b show internal and external views of the Hamon network. Further details are reported in [9].

4.2. Humidity generator to determine humidity dependence of high value resistors

A system, constituted by an enclosure in which the temperature and the relative humidity are externally and remotely controlled, was developed at INRIM. In this enclosure the temperature is maintained, with instability of ±0.01 °C, by means of a electronic controller and a Thermo-Electric Cooling device (TEC). The relative humidity can be varied in the range (10 ÷ 90)% with instability of about ± 3%. This device (block diagram in Fig. 5) is suitable to characterize high value resistors versus relative humidity [16]. The enclosure was developed by means of two aluminium coaxial cylinders insulated with polystyrene and with a polystyrene block surrounding them. All these elements are enclosed in a wooden case. An aluminium thermal conducting bar connects the internal cylinder and the TEC. The resistors are connected to the measurement system by means of shielded cables covered by teflon sheets. These cables go out from the internal cylinder forming a winding around the cylinders to minimize the thermal power that the enclosure exchanges with the environment. This arrangement minimizes also the effect of thermal voltages. Inside the enclosure there are a platinum thermo-resistance and a hygrometer. The humidity is remotely controlled by means of a humidity generator that consists

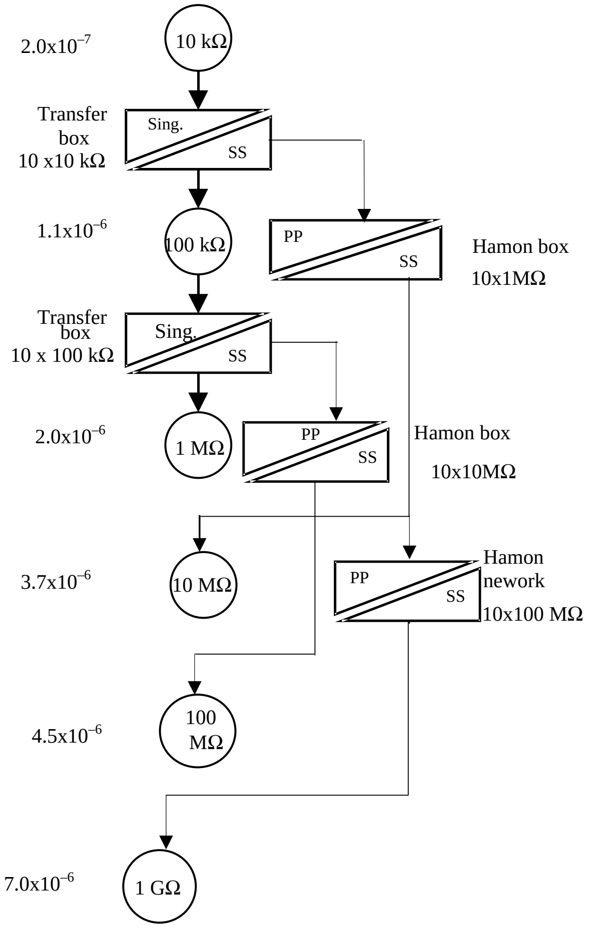

Fig. 2. Metrological chain of the Hamon scaling method from 10 kΩ to 1 GΩ. The reported relative uncertainties are at 2σ level.

Table 3
Relative standard uncertainties according to the metrological chain of the Hamon scaling method from 10 kΩ to 1 GΩ of Fig. 2

Step	Source of uncertainty	Type	1σ (×10^{-6})
10 kΩ	Cal, drift, temper. instab. of the 10 kΩ	B	0.1
↓	Thermal voltages instability	B	0.2
	DMM non-linearity and instability	B	0.2
	10 kΩ → 10 kΩ RSS of standard deviations	A	0.2
	1:10 transfer error	B	0.5
10 × 10 kΩ Transfer box	Temperature instability and drift	B	0.3
	Thermal voltages instability	B	0.1
↓	DMM non-linearity and instability	B	0.3
	Standard deviation of the comparison	A	1
10 × 1 MΩ Hamon box	Temperature instability and drift	B	0.2
	Thermal voltages instability	B	0.1
↓	1:100 transfer error	B	1
	DMM non-linearity and instability of input bias current	B	1
	Standard deviation of the comparison	A	1
10 × 100 MΩ Hamon box	Temperature instability and drift	B	0.3
	Thermal voltages instability	B	0.1
↓	1:100 transfer error	B	1.2
	Substitution with DMM–DCVC method	B	2
	Standard deviation of the comparison	A	1.2
Standard 1 GΩ		Total RSS	3.5

of two micro-pumps, of two conditioning containers, respectively, filled with silica gel and with pure water (Fig. 6) and of an electronic controller connected to the hygrometer. Both pumps take air inside the enclosure and send it in one of two conditioning containers according to the need to increase or to decrease the humidity. A view of the whole system is reported in Fig. 7. Further details are reported in [16].

Table 4
Measurement relative standard uncertainties of the methods at INRIM

Resistor	Voltage (V)	DMM–DCVC method		Hamon scaling method		Current comp. bridge	
		Type B (×10^{-6})	Type A (×10^{-6})	Type B (×10^{-6})	Type A (×10^{-6})	Type B (×10^{-6})	Type A (×10^{-6})
100 kΩ	10	9.7	0.5	0.5	0.2	0.3	0.5
1 MΩ	10	9.7	0.8	0.9	0.4	0.4	0.2
	50	7.7	0.5			0.5	0.5
10 MΩ	10	9.7	0.7	1.8	0.4	1.4	1.8
	50	6.8	0.7				
	100	6.5	0.7				
100 MΩ	10	15	1.7	2.1	0.8		
	100	6.5	1.1				
	250	7.4	1.0			4.1	1.0
	500	7.4	1.2				
1 GΩ	100	8.0	0.7	3.3	1.2		
	250	6.6	0.8			30	12
	500	6.6	0.8	Modif. Wheast. bridge method		15	7.0
	750	6.5	0.8			11	6.0
	1000	6.4	0.7	3.9	0.7		
10 GΩ	100	16	1.2				
	250	13	1.0				
	500	12	0.7				
	750	12	1.0				
	1000	12	1.0	7.5	1.6		
100 GΩ	100	53	118				
	250	25	43				
	500	18	27				
	750	17	24				
	1000	16	23	50	20		
1 TΩ	100	500	17				
	250	200	6.4				
	500	100	3.4				
	750	69	1.9				
	1000	53	1.6				

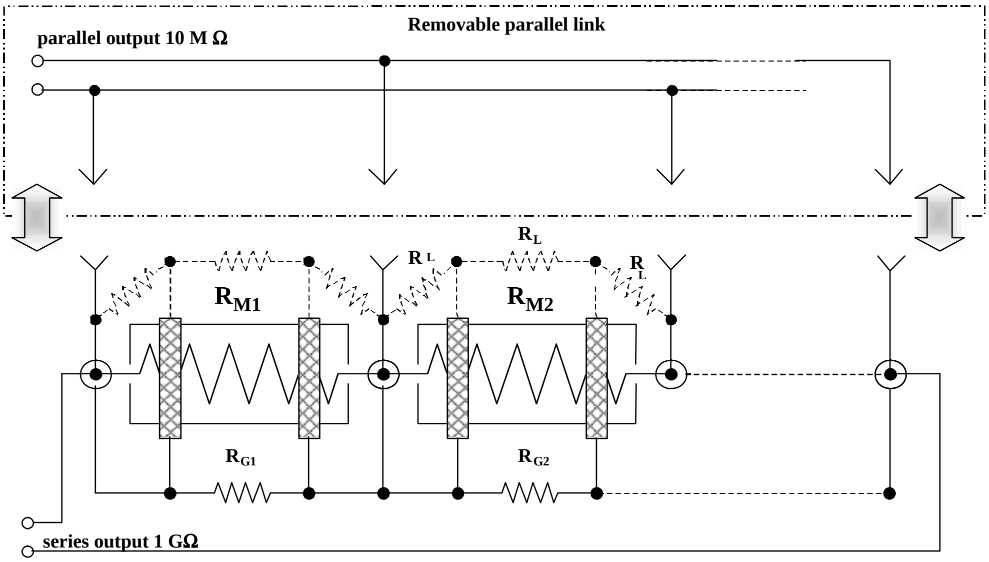

Fig. 3. Scheme of the 100 MΩ step Hamon network. The R_{Mi} are the 100 MΩ main resistors while R_{Gi} are the 10 MΩ guard resistors. In the figure are reported also, in dashed lines, some insulation resistances indicated with R_L. The guard system limits the leakage currents in these resistances.

4.3. Automated guarded scanner for high value resistors

An effective low thermal guarded scanner for high resistance systems was developed by NIST in collaboration with Data Proof [17]. A guarded scanner for automatic selection of high value resistors to be utilized with the DMM–DCVC based method was developed at INRIM. Fig. 8 shows a block diagram of the DMM–DCVC system with the scanner. The diagram shows, besides to the scanner, a DMM, a DCVC and the personal computer that manages both the selection of the resistors and the measurement procedure. The scanner is suitable to operate at high voltages and allows to connect up to six resistors. Each section is a single unity independent from the others and consists of a pair of separated relays for the high and low connection of each resistor, respectively. Inside the scanner a resistive divider, formed by the auxiliary resistors R_A and R_{Bi}, supplies the guard voltage. R_{Bi} is set so that $R_A/R_{Bi} = R_{xi}/R_s$ for each measurement situation. The guard system reduces the leakage currents due to the insulation resistances:

- between the resistors under calibration and their shields;
- among connections of the relays;
- among connections and relays spools;
- of cables and connectors.

All the mentioned insulation resistances can be represented with R_{eqHi} and R_{eqLo} for the high side and low side respectively connections of the scanner (Fig. 9). With this configuration R_{eqHi} is in parallel with R_A (Fig. 8). The voltage on R_{eqLo} is approximately null (as in R_{L2} in Fig. 1) limiting the leakage current in R_{eqLo}. Every resistor connected to the scanner and not under measurement is shorted. The external case of the scanner is connected to a ground point. Further details of the development and characterization of the scanner are given in [18].

5. Discussion

From Table 4 it is possible to see that in the range 100 kΩ ÷ 1 GΩ the Hamon scaling and the current comparator bridge methods (except for the comparator method at 1 GΩ) offer at INRIM better accuracy with respect the DMM–DCVC method. Other Institutes reach even better accuracies in this range of resistance directly comparing the Quantized Hall Resistance to 1 MΩ [19] or 100 MΩ [20]. For values up to 1 TΩ the DMM–DCVC method shows satisfactory reliability and accuracy, in particular for calibration of high value resistors at high measurement voltages (up to 1000 V). The development of the DMM–DCVC method represented an improvement at INRIM with respect the older measurement system that consisted in the utilization of a commercial Teraohmmeter that could operate only in manual way and with considerably higher uncertainties with respect to the DMM–DCVC method. The development of the 100 MΩ step Hamon network allowed to extend the range of the Hamon scaling method up to 1 GΩ, and to decrease the uncertainty of the maintained 1 GΩ standard at INRIM. Moreover the results of the CCE international comparison showed that these measurement methods agreed in satisfactory way at international level [5]. Unfortunately the DMM–DCVC method was not yet verified for calibration of resistors of value higher than 1 TΩ while other Institutes developed methods suitable to calibrate resistors up to 100 TΩ [8,21] and capable to reach high accuracies in particular for calibration of sealed resistors [22]. The development of the humidity generator allows to characterize in a better way the high value resistors of INRIM and of those Laboratories that send their resistors for

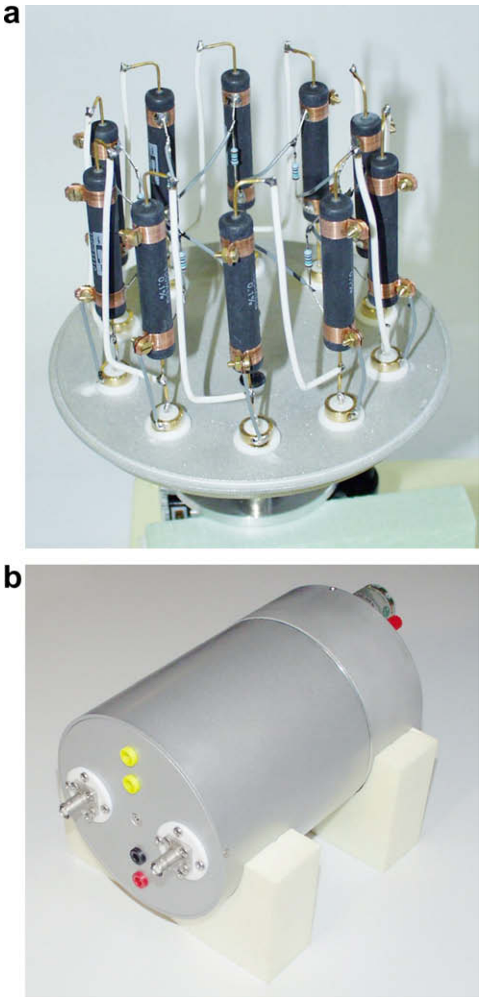

Fig. 4. (a) Internal view of the Hamon network. (b) External view of the Hamon network (output 1 GΩ).

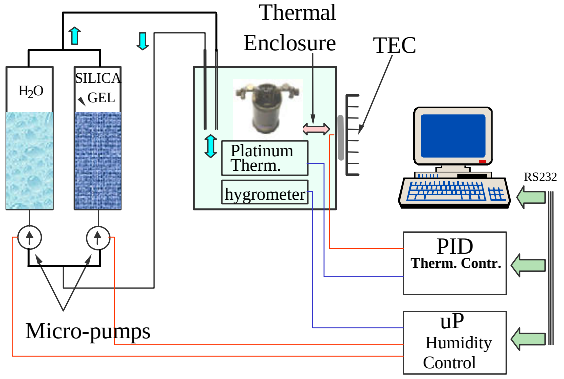

Fig. 5. Scheme of the temperature-humidity controlled system. The units for the thermostatic control (PID) and for the relative humidity control (uP) are external from the enclosure in which the resistors are placed.

periodical calibration at INRIM. The development of the scanner for high resistances allows to perform measurements in automatic way avoiding possible measurement

Fig. 6. Containers of the humidity conditioning of the air inside the enclosure.

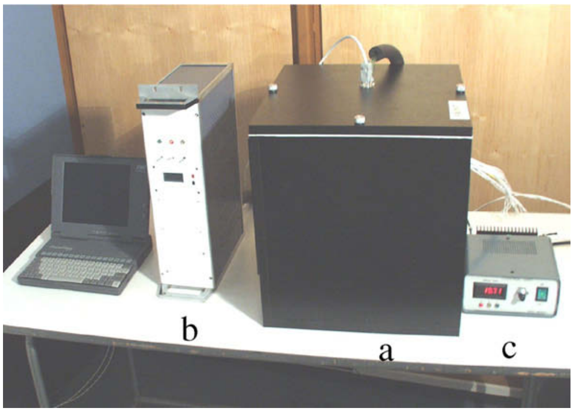

Fig. 7. View of the thermostatic enclosure (a) with the humidity conditioning (b) and the temperature conditioning units (c).

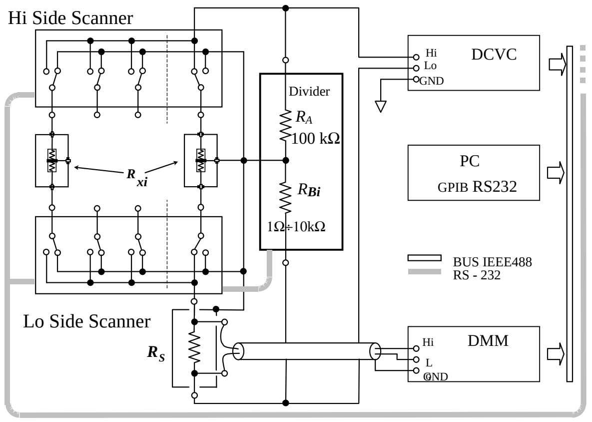

Fig. 8. Block diagram of the scanner inserted in the DMM–DCVC system. The sets of relays for the connections of the high and low terminals of the resistors under calibration are respectively denoted as “Hi Side Scanner” and “Low Side Scanner”.

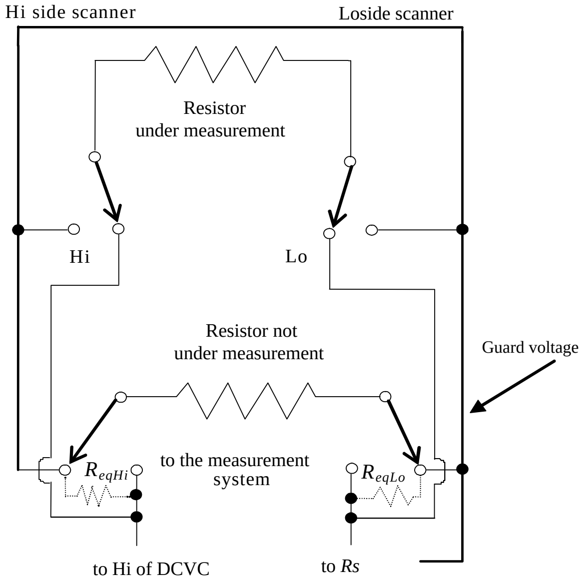

Fig. 9. Connection system adopted in the scanner: the insulation resistances and the resistors not inserted for measure are connected to the guard circuit.

errors due to the presence of the operators in the laboratory. Future work aims to verify the possibility to use the DMM–DCVC method at higher values than 1 TΩ and to complete the implementation of the method based on the modified Wheatstone bridge up to 100 TΩ, with the characterization of new high performance 10 TΩ and 100 TΩ standard resistors and of a new high stability DCVC. With this system INRIM could participate in a key comparison for these ranges of values.

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