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Expansion of traceability chain of PEL to high value resistance standards

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Abstract-In the Primary Electromagnetic Laboratory (PEL), which is a part of the Faculty of Electrical Engineering and Computing of the University of Zagreb and a part of the Croatian metrology system, the unit of resistance (ohm) is maintained by the group of reference and working standards of nominal decade values ranging from 1 mΩ to 100 GΩ. The group of high value resistance standards from 100 MΩ to 100 GΩ is the hindmost incorporated in the resistance traceability chain, and relies on laboratory developed measurement system for high resistance comparison, which is presented in this paper. Realization of traceability of high value resistance standard group toward both of 10 kΩ and 10 MΩ reference standards is described. Based on 10 MΩ as starting point, the high resistance scaling was performed and temperature coefficients of resistors were determined. The achieved results of high value resistance comparison were analyzed and compared with the calibration values obtained at Physikalisch-Technische Bundesanstalt (PTB).

I. Resistance traceability in PEL

One of main tasks of PEL, as a holder of Croatian national standards of dc voltage, resistance and capacitance, is to ensure a traceability chain for group of resistance standard in the range from 1 mΩ to 100 GΩ. The reference standards of PEL are 1 Ω (L&N 4210), 10 kΩ (L&N 4040-B) and 10 MΩ (Fluke 742A-10M), which are regularly calibrated at PTB in Germany. Reference standards 1 Ω and 10 kΩ have been maintaining into the oil-thermostat at a temperature of 23 °C, and their characteristics are presented in [1]. Some other resistance standards of PEL, as well as their maintenance, methods of comparison and achieved uncertainties, were presented in [2, 3]. Recently, a new 10 kΩ standard resistor of ESI/Tegam SR104 type has been added to the group, and was firstly calibrated in May 2007 at PTB. Since it is constructed as a reliable transfer standard, our intention is to (possibly) use it as the only traveling resistor for future calibrations. For transferring traceability of reference standards to other working standard resistances in the range 1 mΩ − 10 MΩ several methods had been developed in the laboratory, presented elsewhere in details [4, 5]. Until last year, the highest working resistance standard in PEL was laboratory-built 100 MΩ resistor of Hamon type, marked as VOO-100, that consists of 100 equally balanced 1 MΩ resistors connected in series, and placed in a sealed and very well temperature controlled casing, filled with dry air. The construction of this standard is depicted in [6], and the determinations of it long-time drift in [7, 8]. Using a special switching device the resistors can be circuited in parallel, and 100^2 times smaller resistance (i.e. 10 kΩ) can be obtained, allowing direct comparison with one of 10 kΩ reference standards. Together with reference standard of 10 MΩ, this Hamon type resistor represents reliable origin for internal laboratory traceability transfer toward series of high resistance standard.

Table1. Manufacturer's and PTB calibrated values of the high value resistance standard group

Resistor	IET certificates			PTB calibrations, May 2007	
	Value	Uncertainty	Drift	Value	Uncertainty (95 %)
SRC-100M	100,0028 MΩ	30 ppm	200 ppm/year	99,9911 MΩ	8 ppm
SRC-1G	1,00306 GΩ	100 ppm	500 ppm/year	0,998983 GΩ	12 ppm
SRC-10G	9,99153 GΩ	200 ppm	500 ppm/year	9,9965 GΩ	50 ppm
SRC-100G	99,737 GΩ	2000 ppm	500 ppm/year	99,766 GΩ	120 ppm

Last year the laboratory resistance standards decade was extended with the group of high value resistance standards ranging from 100 MΩ to 100 GΩ (Table 1). All four resistors are of SRC type made by IET (IET Labs, Inc., Westbury, USA), self-contained in air-tight aluminum case. The resistors were accompanied by manufacturer's calibration certificates, where calibrated values and long-term drift are clearly declared. A first calibration of this series of standards has been performed in May 2007 at PTB, and given results are also presented in Table 1 (ppm means "parts per million").

II. Method for comparison of high value resistance standards

The extension of internal laboratory resistance traceability chain to values higher than 100 MΩ requires a measurement method which can accomplish specific requirements regarding suppression of parasitic quantities (parasitic capacitances of measured resistors, insulation leakage currents) and somewhat greater measuring voltages, needed to attain smaller measuring uncertainty. The method that is currently being implemented is based on modified Wheatstone bridge (or "active-arm" bridge), illustrated in Fig.1 [9].

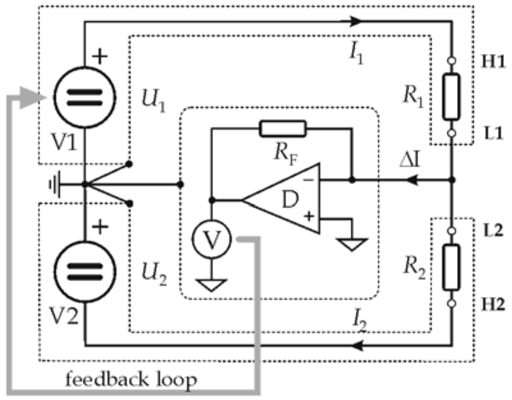

Figure 1. Modified Wheatstone bridge ("active-arm" bridge) for high value resistance comparison

The active-arm bridge is formed by substituting two of the resistive arms of a Wheatstone bridge circuit with the low-impedance voltage calibrator sources V1 and V2, having only the source V1 programmable and a part of bridge balancing feedback loop. The detector D is low-impedance current-sensing transimpedance amplifier with non-inverting input connected to grounded guard. In this way, the connection point of resistors R_1 and R_2 ("low" terminals L1 and L2, respectively) is constantly held at potential of "virtual null" during bridge balancing, thus eliminating flow of leakage currents from detector inverting input towards guard. The bridge guard circuit is connected to earth ground at a single point between the two voltage calibrators V1 and V2. The grounded guard includes the shield of the detector D, the guard circuit at the high and low sides of the detector, and the grounded cases of resistors R_1 and R_2. In this way a leakage resistance paths between "high" terminals H1 and H2 and cases are connected in parallel with the voltage sources V1 and V2, respectively, and does not produce a systematic error. The detector multiplies current difference $\Delta I = I_1 - I_2$ by transimpedance factor equal to R_F, generating the output voltage measured by digital voltmeter V, which then acts as a part of balancing feedback. The detector feedback resistor R_F defines sensitivity of the detector, and based on values of compared resistors, applied voltages and Johnson noise of R_F itself, it was chosen to be $R_F = 10^9$ Ω.
The measuring equipment used in this method are: calibrator Fluke 5700A as adjustable voltage source V1, calibrator Fluke 5400B as fixed voltage source V2, and Keithley 2000 as digital voltmeter V. Procedure of bridge balancing has two steps. At the first step, the "H1" and "H2" terminals of resistors R_1 and R_2 are disconnected from voltage sources and shorted to the ground, and resulting output voltage U_0 of the detector is measured. This voltage is a consequence of input offset voltage of detector's amplifier (U_{of}) and its input offset current (I_{of}) flowing through the resistor R_F:

$$U_0 = U_{of} \cdot \left(1 + \frac{R_F}{R_1 \| R_2}\right) - R_F \cdot I_{of} \,. \qquad (1)$$

Assuming steady values of offset parameters U_{of} and I_{of}, U_0 stands for "null" that has to be established by bridge balancing. In the second step the voltage sources V1 and V2 are enabled at initial values for 10:1 resistance comparison, which are chosen to be 100 V and 10 V, respectively. The detector senses the currents difference ΔI flowing through R_1 and R_2 and gives a voltage proportional to the bridge misbalance. A new estimated output of source V1 (i.e. voltage U_1'), which should drive ΔI close to a null, is calculated using the equation:

$$U_1' = U_1 + U_D \frac{R_1}{R_F}\,, \tag{2}$$

where U_1 is the starting voltage of source V1, and U_D is the output voltage of detector. The PC-controlled feedback loop changes the output voltage of V1 to a new value U_1' and the voltage U_D is measured again. It must be emphasized that the voltmeter V is taking readings in the programmed sequence, using algorithm that calculates moving average value U_D and standard deviation s_{UD} of the group of significant readings until satisfactory stationary state is achieved. Experiments have shown that four to five cycles should be taken until the difference $|U_D| - |U_0|$ falls within margins

$$|U_D| - |U_0| < 1{,}5 \cdot s_{UD}, \tag{3}$$

which is compromise between the measurement accuracy and duration of balancing process. Once a bridge balance was established, the ratio r_{12} of compared resistances R_1 and R_2 follows from the ratio of reached voltages U_1' and U_2:

$$r_{12} = \frac{R_1}{R_2} = \frac{U_1'}{U_2}\,. \tag{4}$$

The repeatability of resistance ratio comes from series of 17 subsequent balancing procedures described above, with total duration of approximately 15 minutes. The final result of measurement is expressed as arithmetic mean of given ratios and associated measurement uncertainty.

III. Defining starting value(s) for high value resistance comparison chain

Calibration of high value resistor standard group (100 MΩ to 100 GΩ) begins with comparison of 100 MΩ standard with 10 MΩ reference standard Fluke 742A, for which the most recent calibrated value (PTB, April 2007) is $R_{10M\text{-}PTB} = 10{,}000490 \cdot (1 \pm 2 \cdot 10^{-6})$ MΩ, stated for temperature range (23 ± 0,2) °C. The Hamon divider VOO-100 (section I) is used to perform internal laboratory comparison of 10 MΩ reference standard with 10 kΩ transfer standard Tegam SR104 (R_{TE}). Initially, 10 kΩ transfer standard had been compared with Hamon divider in parallel connection ($r_{HP,TE}$) using "two digital voltmeters method" for resistance comparison [2]. By switching to serial connection, Hamon divider is multiplied with exact Hamon ratio of 10^4 (R_{HS}/R_{HP}) resulting in traceable 100 MΩ value (R_{HS}). Comparison of high value Hamon resistance and reference standard of 10 MΩ ($r_{HS,10M}$) by the method of measurement described in section II yielded $R_{10M\text{-}PEL} = 10{,}000502 \cdot (1 \pm 0{,}76 \cdot 10^{-6})$ MΩ.

Table 2. Components of uncertainty budget of laboratory calibration of 10 MΩ reference standard

	Date	Value	Uncertainty (95 %)
$R_{10M\text{-}PTB}$ (Fluke 742A-10M)	PTB, April 2007	10,000490 MΩ	2 ppm in temp. range (23 ± 0,2) °C
R_{TE} (Tegam SR104)	PTB, May 2007	9,99999926 kΩ	0,1 ppm
$r_{HP,TE} = R_{HP}/R_{TE}$	PEL October 31, 2007	1,0108455	0,11 ppm
R_{HP} (Hamon divider 10 kΩ)		10,108454 kΩ	0,15 ppm
R_{HS}/R_{HP} (Hamon ratio)		10^4	0,25 ppm
R_{HS} (Hamon divider 100 MΩ)		101,08454 MΩ	0,29 ppm
$r_{HS,10M} = R_{HS}/R_{10M}$		10,1079463	0,69 ppm
Temp. coefficient of 10 MΩ reference standard: $\alpha_{10M} = -0{,}15$ ppm/K Uncertainty of 10 MΩ reference standard due to change of laboratory temperature within range (23 ± 0,5) °C:			0,15 ppm
$R_{10M\text{-}PEL}$ (Fluke 742A-10M)		10,000502 MΩ	0,76 ppm in temp. range (23 ± 0,5) °C

The components of uncertainty budget, including uncertainty of 10 MΩ reference resistance due to the change of laboratory temperature within range (23 ± 0,5) °C, are listed in Table 2. Evaluating the presented results of calibration of 10 MΩ reference standard, it is obvious that the difference between the values $R_{10M\text{-}PTB}$ and $R_{10M\text{-}PEL}$ is only −1,2 ppm, which is within the uncertainty interval pointed out for $R_{10M\text{-}PTB}$ (2 ppm). Therefore, the value $R_{10M\text{-}PEL}$ can be used as a starting value for high value resistance comparison chain.

IV. High resistance comparison chain and determination of temperature coefficients

Altogether, comparison chain determines following ratios: 100 MΩ/10 MΩ, 1 GΩ/100 MΩ, 10 GΩ/1 GΩ and 100 GΩ/10 GΩ, where nominators are measured (R_1) and denominators are reference resistances (R_2) used in particular measurement of resistance ratio. Measured resistance R_1 of preceding comparison becomes reference resistance in the next comparison in sequence.
In order to found linear temperature coefficient of resistance R_1, ratio r_{T12} has been repeatedly measured by stabilizing R_1 on several temperatures T_1 in the range [T_2, T_2 + 5 °C], where T_2 is temperature of reference resistance R_2. Generally, each ratio r_{T12}, which stands for temperature pair (T_1, T_2), follows the relation:

$$r_{T12} = \frac{R_{1,T1}}{R_{2,T2}} = \frac{R_{1,T1}}{R_{2,23}\left[1+\alpha_2\left(T_2 - 23\,°\mathrm{C}\right)\right]}, \tag{5}$$

and from the known $R_{2,T2}$ and measured ratio r_{T12}, a value of resistance R_1 at temperature T_1 ($R_{1,T1}$) can easily be found. Since the value $R_{2,T2}$ corresponds to actual laboratory temperature T_2 which could slightly differ from 23 °C, the reference value $R_{2,23}$ at a nominal temperature of 23 °C must be corrected by introducing its temperature coefficient α_2.

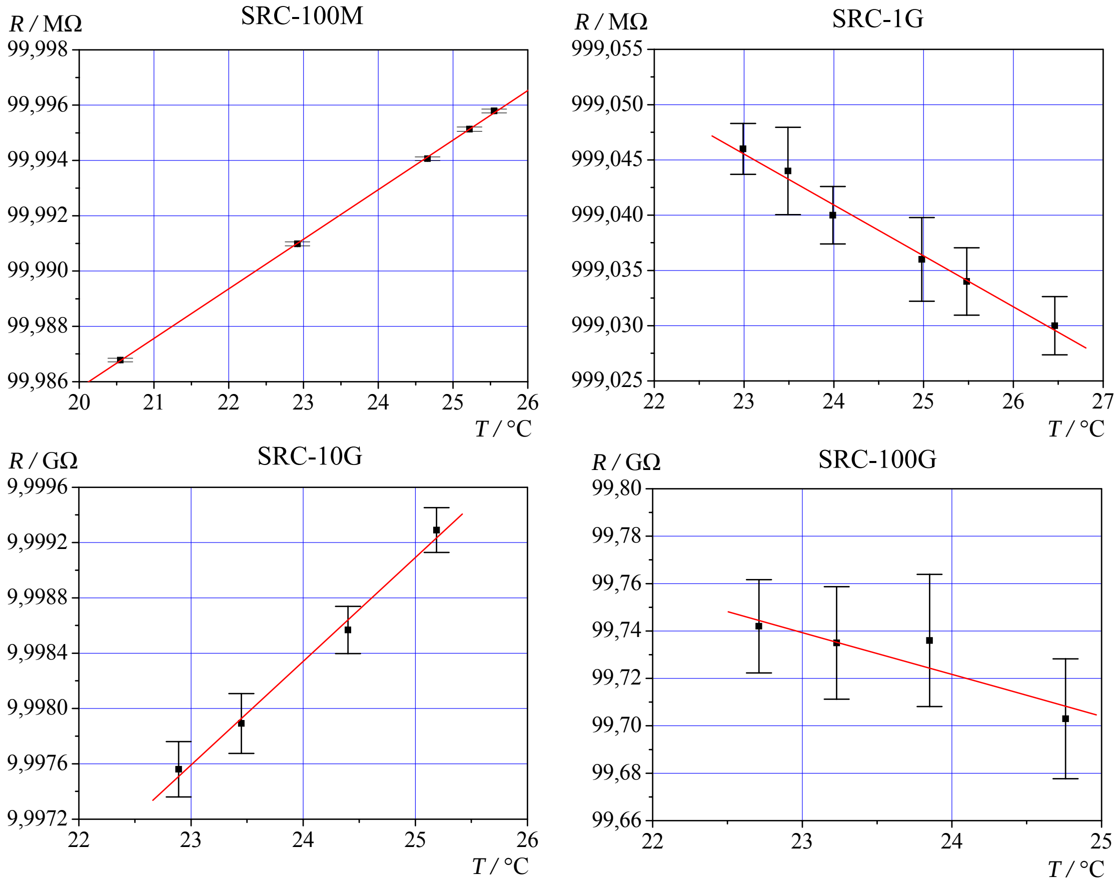

Figure 2. Results of measurements of high-value resistances temperature characteristics with expressed regression lines used for determination of temperature coefficients

Starting with comparison 100 MΩ/10 MΩ, the parameters of reference resistance are well-defined ($R_{2,23}$=$R_{10M\text{-}PEL}$ and α_2=α_{10M}), making possible to calculate accurate values $R_{1,T1}$ of 100 MΩ standard at several different temperatures T_1. The given values $R_{1,T1}$ are then subjected to the weighted regression line analysis, by which the parameters $R_{1,23}$ and α_1 for 100 MΩ resistance standard are calculated. The obtained parameters will be used as reference values ($R_{2,23}$ and α_2 respectively) in the following comparison in sequence, i.e. 1 GΩ/100 MΩ. In this way the measurements and analysis is performed for the whole high value resistance chain up to 100 GΩ. The measured temperature characteristics of high-value resistances are given in Fig. 2, together with associated regression lines. Finally, the resistance at 23 °C, R_{23}, and temperature coefficient α for each standard are listed in Table 3, where temperature coefficients are constant in range (22 – 25) °C.

Table 3. Results of PEL high value resistance comparison chain (2007-09-27 – 2007-10-10)

Resistance standard	Serial number	Value, R_{23}	Temperature coefficient, α	Uncertainty (95 %)	Date
SRC-100M	J1-0610169	99,99114 MΩ	+17,9 ppm/K	1,6 ppm	2007-27-09
SRC-1G	J1-0610170	0,999046 GΩ	+ 4,6 ppm/K	3,9 ppm	2007-10-01
SRC-10G	J1-0610171	9,99532 GΩ	+75,5 ppm/K	44 ppm	2007-10-04
SRC-100G	J1-0610172	99,739 GΩ	−177 ppm/K	534 ppm	2007-10-10

V. Conclusions

Comparing two groups of resistance values, those given by PTB calibration reports and those acquired with the presented laboratory method, we can conclude that: (i) the 10 MΩ and 100 MΩ PEL values and uncertainties ranges are completely enclosed within corresponding uncertainty ranges stated from PTB report, verifying the accuracy of developed method; (ii) the PEL values of 1 GΩ and 10 GΩ are showing deflection from PTB values of 63 ppm and −118 ppm respectively, although the uncertainties of PEL values are smaller than PTB ones; (iii) the 100 GΩ PEL value was determined with four times greater uncertainty than PTB value, but completely comprises PTB uncertainty range; (iv) the differences between PTB and PEL values for 1 GΩ and 10 GΩ resistance standards are possibly caused by time-drift of resistance material, taking into account manufacturer's time-drift values and time interval between PTB and PEL calibrations. Therefore, the determination of high value resistance standards time-drifts is a matter of further investigations and measurements.

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