17th Symposium IMEKO TC 4, 3rd Symposium IMEKO TC 19 and 15th IWADC Workshop
Instrumentation for the ICT Era
Sept. 8-10, 2010, Kosice, Slovakia

Inter-laboratory Comparison of the Electrical Reference Standards of FEIT-Skopje and FER-Zagreb

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Abstract- In the paper two procedures of inter-laboratory comparisons of the electrical reference standards (resistance and DC voltage) of the Metrological Calibration Laboratory of the Faculty of Electrical Engineering and Information Technologies in Skopje (FEIT), R. Macedonia and the Primary Electromagnetic Laboratory at Faculty of Electrical Engineering and Computing in Zagreb (FER), R. Croatia will be described. An original improved resistance comparison method, developed by R. Malaric is used for the resistance comparison, as well as an expanded model for uncertainty estimation. The improvement of the model and uncertainty estimation is considered referring to the Example S3, shown in EA-4/02 publication. By comparison it is shown how the used method and model improve the quality of the resistance calibration. The paper presents also the inter-comparison of reference DC voltage standards of the both laboratories. The inter-comparison was done by a new automatic system, which contains self-developed 16 channel low-thermal switch and digital nano-voltmeter, controlled by the computer, using an optimized procedure, and control software in LabVIEW. The results of the inter-comparison are discussed.

I. Introduction

In the frame of a joint research project, an inter-laboratory comparison of resistance standards and DC voltage reference standards of the Metrological Calibration Laboratory of FEIT in Skopje, R. Macedonia and the Primary Electromagnetic Laboratory at FER in Zagreb, R. Croatia has been realised. The maintenance of the unit of Volt is one of the important tasks for every electromagnetic calibration laboratory. FEIT Laboratory uses the electronic (Zener) voltage standard, Fluke 732A, as reference of DC voltage. This standard has its last calibration in 1999 at Physikalisch-Technische Bundesanstalt (PTB)–Germany calibration laboratory, by means of Josephson voltage standard. Besides the very rare use of the standard and the good laboratory condition of keeping it, an urgent need for calibration or inter-comparison existed. At the FER-Zagreb, the Primary Electromagnetic Laboratory (PEL) is a holder of national standards of voltage. Although the best possible accuracy (or maintenance of the highest level) of PEL is attained by means of Josephson voltage standard (JVS) [1-3], due to its costs and limited availability, it is not the most practical. So the value of 1 V and 10 V annually is transferred to, and further maintained by electronic (Zener) voltage standards. For the inter-comparison it was chosen the time right after the transfer of the value of 1 V and 10 V from the JVS to the electronic (Zener) voltage standards of PEL. To provide the inter-comparison of DC reference standards (DCRS), a simple, reliable, ready for everyday use, low cost procedure developed by PEL is used [1]. The procedure is based on the comparison of *n* number of DCRS with unknown voltage on their outputs (usually 1 V, 1,018 V and 10 V) to the reference one, of the same type (Fluke 732A and 732B, Datron 4910, or similar).

The inter-comparison of resistance standards has been accomplished for the resistance values of 0,1 Ω, 1 Ω, 10 Ω, 100 Ω, 1 kΩ, 10 kΩ. An original method and automated system developed by R. Malaric, [4], was used during the procedure of inter-comparison. One of the goals of the joint work was to practically transfer the reference standards values and to develop a model for estimation and validation of the uncertainty. The Example S3 given in EA-4/02 "Expression of the Uncertainty of Measurement in Calibration", [5] is chosen to compare the calibration characteristics. In that example the resistance of a standard resistor is determined by resistance comparison using a digital multimeter (7½ digits DMM), and a calibrated standard resistor of the same nominal value as reference standard. The resistors are immersed in an oil bath at a temperature of 23 °C, monitored by a mercury-in-glass thermometer. The four-terminal connectors of each resistor are connected in turn to the terminals of the DMM. The resistance R_2 of the unknown resistor is obtained from the relationship:

$$R_2=(R_1+\delta R_{d1}+\delta R_{t1})\, r_c\, r-\delta R_{t2} \qquad (1)$$

where: R_1 - resistance of the reference, δR_{d1} - drift of the resistance of the reference since its last calibration, δR_{t1} - temperature related resistance variation of the reference, $r = R_{i2}/R_{i1}$ - ratio of the indicated resistance (index i means 'indicated') for the unknown and reference resistors, r_c - correction factor for parasitic voltages and instrument resolution δR_{t2} - temperature-related resistance variation of the unknown resistor.

II. Inter-laboratory Comparison of Resistance Standards

The measuring arrangement of the improved resistance comparison method has already been presented in [4]. It is a comparison method of unknown resistance to a resistance with known value by using the method of two digital voltmeters (DV1 and DV2), measuring the voltage drop over the two resistances. The influence of thermoelectric EMFs is annulated through the change of measuring current polarity. The digital voltmeters are PC-controlled via GPIB bus. Two voltages measured with DV1 and DV2 are measured simultaneously using GET command, which enables synchronous integration of DC voltages on both voltmeters. The use of two voltmeters with simultaneous readings increases accuracy due to immunity of the measuring circuit to the current drift. It should be emphasized that the resistance of the short lead between the compared resistors does not affect accuracy of the measured ratio since it is placed outside of the voltmeters' measuring circuits. Relays contact resistances are found to be less then 0,1 mΩ, and can be easily neglected if compared to the teraohm input resistances of the DV1 and DV2. The measurement system is controlled by a computer connected to the voltmeters HP 3458A and Keithley 199 (for temperature measurement) through GPIB bus, and to the switch device via input/output 8255 card. Each of the voltmeters is measuring dc voltage across one of the compared resistors, so the relative errors can be assigned in accordance to the general relation:

$$Umeasured = Utrue\ (1 + p) \tag{2}$$

The resistance of the unknown standard can be calculated by the following relation:

$$R_2 = \frac{R_1}{\sqrt{\frac{U_{1a}U_{2b}}{U_{2a}U_{1b}}}\cdot(1+p)} = \frac{R_1}{M_{12}(1+p)} \tag{3}$$

where M_{12} stands for the absolute value of the measured ratio, which is determined by means of the automated measurement procedure. U_{1a} is the voltage measured by voltmeter DV1 across the resistor R_1, U_{2a} is the voltage measured by voltmeter DV2 across the resistor R_2, U_{1b} is the voltage measured by voltmeter DV1 across the resistor R_2, (after voltmeter's positions interchange), U_{2b} is the voltage measured by voltmeter DV2 across the resistor R_1, (after voltmeter's positions interchange), p is the relative error of the ratio, caused by the errors of the voltmeters' readings. The estimation of the uncertainty of measurement in the calibration is done by using a more detailed and improved model compared with the model given by the equation (1). From the basic relation (3) it follows that the resistance of the calibrated standard will be calculated from the known value of resistance R_1 and measured ratio M_{12}. The influence of relative error of the ratio p can be neglected because, when the measuring ratio is 10:1 it is equal to $5 \cdot 10^{-8}$, and for ratio 1:1 is even smaller. The resistance of reference R_1 can be expressed in the following form

$$R_1 = R_{1N} + \delta R_{d1} + \delta R_{t1}\ , \tag{4}$$

where δR_{d1} is the difference to the nominal value R_{1N} of the used standard R_1, at the moment (day) of comparison. To calculate this value for reference resistance standards (for 1 Ω-RR1, for 10 kΩ-RR10k) according to the regression function from calibration history, the associated program is used. The values calculated in that way are expressed for a reference temperature of 23 °C. The actual values of the used standards on calibration date were: (0,999992131±0,000000065) Ω, and (9999,9942±0,0093) Ω, δR_{t1} is the correction due to the temperature, calculated as follows:

$$\delta R_{T1} = (R_{1N} + \delta R_{d1})(1 + \alpha_1(T_1 - 23) + \beta_1(T_1 - 23)^2) \tag{5}$$

where α_1 and β_1 are temperature coefficients of the standard. The standard R_1 is placed into the ultra-thermostat and the average temperature value from NTC1 is used. The absolute value of measured ratio M_{12} can be expressed in the following form:

$$M_{12} = M_N(1 + m_{12}) \tag{6}$$

where m_{12} is the measured ratio, expressed as relative deviation from the nominal value, with variation due to the effects of cables, leakages, interferences, as well as non-corrected influences of temperature and power dissipation. The resistance of calibrated standard can be expressed in the form:

$$R_2 = (R_{1N} + \delta R_{d1})(1 + m_{12})(1 + \alpha_1(T_1 - 23) + \beta_1(T_1 - 23)^2)/(1 + \alpha_2(T_2 - 23) + \beta_2(T_2 - 23)^2) \tag{7}$$

The reference standard of 1 Ω (RR1) and 10 kΩ (RR10k) are stored in the ultra-thermostat. The thermostat maintains temperature at a reference value of 23 °C. Each of the reference standards is "equipped" with an NTC resistor. So, for the measurement of temperature, that can be reasonably used as the value of temperature of the standard. The standards under calibration were placed outside at ambient temperature, measured with additional NTCR, placed at appropriate place near the standard under calibration. The measurement uncertainty of the temperature is ±0,03 K. Differently from the Example S3, [5] the reference standard and the standard under calibration are on different temperatures. The uncertainty of the temperatures T_1 and T_2 is estimated from the measurement as:

$$\overline{T}_1 = \frac{\sum_{1}^{10} T_{1i}}{10}, \qquad u_{T_1} = \sqrt{\frac{\left(u_{T_1}^{\,2} + u_{NTC}^{\,2}\right)}{10}} \tag{8}$$

In the estimation of the uncertainty of R_2, the temperatures T_1 and T_2 are taken as correlated variables with coefficient of correlation calculated from the measured values. One of the specifics of the comparison was the change of the ambient temperature (the temperature T_2). Due to the presence of the staff in the laboratory and the non adequate clime system, the ambient temperature increased. During the first series of calibration measurement it was T_{2SR}=23,033 °C, while for the second series it was T_{2SR}=24,03 °C. The value of 1 Ω standard under calibration, having two series of measurement, is calculated as:

$$R_2=(R_2'+R_2'')/2,\ \ u_{R2}=((u'^{\,2}_{R2}+u''^{\,2}_{R2})/2)^{1/2} \tag{9}$$

The model (7), differently from (1), does not contain the factor r_c. Here it is assumed that measured variable m_{12} includes the influence of parasitic voltages, connections, polarity etc., while the uncertainty of the used multimeters should be added.

Table 1 Uncertainty budget calculated by GUM procedure of the 10 kΩ reference resistance

Quantity X_i	Estimate x_i	Unit	Standard Uncertainty $u(x_i)$	Probability Distribution	Sensitivity Coefficient c_i
R_{1n}	10.000,000000	Ω	0,004649	Normal	1,000220
δRd_{1n}	-0,005834	Ω		Constant	1,000220
T_1	22,999987	ºC	0,017321	Rectangular	0,050010
T_2	24,033000	ºC	0,017321	Rectangular	-0,100020
m_{12}	-0,000230		0,000000	Normal	-9.999,894090
a_1	0,000005			Constant	0,000000
a_2	0,000010			Constant	-10.002,095220
R_2	10.002,189765	Ω		u_{R2}=	0,005349

Table 2 Monte Carlo Simulation Summary

Measure	R2	R1n	m12	T1	T2
Observations	10.000	10.000	10.000	10.000	10.000
Mean	10.002,1929920945	9.999,9999752734	-0,0002298882	22,9999651636	24,0000582392
Standard Deviation	0,005111574	0,0047957099	0,000000179	0,017358801	0,0174331623
Posterior STD	0,0000511157	0,0000479571	0,0000000018	0,000173588	0,0001743316
Variance	0,0000261282	0,0000229988	3,203046192e-14	0,000301328	0,0003039151
Minimum	10.002,1735050732	9.999,9802882606	-0,0002306056	22,9700030615	23,9700086146
5th Percentile	10.002,1848282437	9.999,9922521369	-0,0002301788	22,9729861504	23,9728820235
Median	10.002,1930118813	9.999,9999990598	-0,0002298885	22,9998644577	24,0001744871
95th Percentile	10.002,2012352629	10.000,0076039533	-0,0002295929	23,0271194642	24,0272764283
Maximum	10.002,211625357	10.000,0165096132	-0,0002292131	23,0299942083	24,0299973184

Similarly, for the 1 Ω standard resistor, it is calculated: R_2=1,000219 Ω and u_{R2}=0,3699 µΩ. The same model (7) may be used for the calculation of uncertainty by Monte Carlo simulation. However, it is obvious that the variables T_1 , T_2, and m_{12} are not independent. The coefficients of correlation are calculated for the measured values of T_1, T_2 and m_{12}. The Monte Carlo simulation model takes into account the correlation of the variables T_1, T_2, and m_{12}. The Monte Carlo simulation results, shown in Table 2, serve as a verification of the GUM mainstream estimation. The simulated value of R_2 on 23 ºC is the same as it was calculated by GUM, [5] main stream procedure, R_2=10002,189765 Ω, but calculated uncertainty is u=0,00511172 Ω and it is smaller. The calculated sensitivity shows that R_s has the most important contribution in the R_2 uncertainty. Compared with the

Example S3 from the EA 4/02 publication, this results showed that the calibration of FEIT resistance standards uncertainty is 40 % lower, despite the laboratory conditions weren't so good.

III. DC Voltage Inter-laboratory Comparison

The simple and precise method to compare two DCRS, when they are connected in series opposition, is previously described in [1]. The voltages of the sources A and B are marked as U_A and U_B, and their internal resistances as R_{inA} and R_{inB}, respectively; the nano-voltmeter is marked with nV, while different TEMFs in the circuit are marked with ε. The voltage difference between the two standards is:

$$U_A - U_B = \delta_{AB} + \sum \varepsilon_1 - \varepsilon_{nV1} \tag{10}$$

where δ_{AB} represents the voltage difference measured by the nano-voltmeter; $\sum \varepsilon_1$ is the sum of residual TEMFs at the connection terminals of DCRS, and ε_{nV1} is residual TEMF of nano-voltmeter. The R_{inA} and R_{inB} are approx. 1 mΩ, and for this analysis their influences can be neglected due to the high impedance of nano-voltmeter (>10 GΩ), and low circuit currents (~10 pA). Therefore, the errors due to the voltage drop on internal resistances of DCRS can be neglected. The voltage differences are measured using a self-constructed digital nano-voltmeter **η***PEL* operated in its 10 mV range. This nano-voltmeter is calibrated using the output of Josephson voltage standard. The zero stability of **η***PEL,* with short plugs applied to its input, is appropriate (standard deviations between 8 nV and 10 nV for a measurement period up to 5 minutes). The minimum achievable Allan variance and the time necessary to attain it for **η***PEL* [6] is 0,3 nV for a period of 1000 s. The linearity on 10 mV range is better than 1 μV/V. The most important factors which limit the precision of digital and analog nano-voltmeters are the stability of ambient temperature and source resistance. For the used type of nano-voltmeter no specially temperature-stabilized enclosure is necessary (laboratory temperature conditions (ϑ = 23 °C ± 2 °C)). The application software is in the graphical language LabVIEW. The whole measurement process is performed automatically under GPIB and RS232 control. The inter-comparison measurements were done, after environmental stabilization of the FEIT reference standard FLUKE 732A. The measurements are made for the 3 outputs of the reference standard: 1 V, 1,018 V and 10 V. The actual values of the PEL reference standard were: U_{RU1V}= **0,9999894037** V ($U_{RU1V} + d\cdot\Delta t$), u_p = 664 nV, ΔU=**-10,596** μV/V deviation from nominal value (1 V). On the following figure are presented graphically the measurement results for the 1 V value inter-comparison. The figure show the difference $\delta U = U_{MAC} - U_{RU.\,V,}$ where U_{MAC} is the value of the FEIT standard and $U_{RU.\,V}$ is the value of the FER standard. The results of the inter-laboratory comparison of the 10 V and 1,018 V value standards will be given in the full version of the paper. The equation for the estimation of the combined uncertainty is:

$$u_C(U_X) = \sqrt{u_{A1}^2 + u_{AX}^2 + u_{A2}^2 + u_{DV}^2 + u_f^2} \tag{11}$$

The contributions are: u_{A1} – the uncertainty of standard A calculated for the chosen day according to the weighted regression line (depends of the date, but is near to 0,8 μV); u_{AX} – the uncertainty of the voltage difference ΔU_{AX}, a value less than 1 μV should be taken; u_{A2} – the value 0,2 μV should be taken, determined from the past calibration of standard A; u_{DV} – the uncertainty of the used digital nano-voltmeter that is calculated from the data in [3], the value u_{DV} = 0,4 μV should be taken; u_f – estimation of the remained errors in measurement procedure (thermal emfs, zeroing, etc.); the value u_f = 0,1 μV should be taken. The expanded uncertainty is calculated by the expression $U_C(U_X) = k \cdot u_C(U_X)$, where the coverage factor k = 2 should be used:

$$U_{MAC1V} = 0{,}9999904441\ \text{V}\ ,\ u_c(U_{MAC}) = \sqrt{u_{A1}^2 + u_{AX}^2 + u_{A2}^2 + u_{DV}^2 + u_f^2} = \sqrt{0{,}7^2 + 0{,}02^2 + 0{,}17^2 + 0{,}4^2 + 0{,}1^2} = 1\mu V \tag{12}$$

The inter-comparison measurement process shown in the Figure 1 is stable and precise. After the correct sampling time τ was chosen, nano-voltmeter deviation was much smaller than overall measurement uncertainty. The Allan variance is related to the power spectral density and can reveal correlations in time series. The Allan variance provided a way of characterizing measurement processes in the time domain [6, 7], and served as control of the measurement process quality. For the **η***PEL* nano-voltmeter the characteristic time bandwidth B has been deduced over which the measurement noise is white and Allan variance decreases. The key issue in measurement setup was to attain that the standard deviation of the measured mean is just the standard deviation divided by the square root of the number of measurements. Only for white noise time series the Allan deviation is equal to the standard deviation of the mean. In Fig. 2 the Allan deviation as a function of the sampling time is presented. Digital nano-voltmeter **η***PEL* in Fig. 2 has white noise process up to 1000 s measurement bandwidth. For sampling times below 1000 s, the Allan deviation decreases nearly as $\tau^{-1/2}$, indicating that in this range the noise is white. The Allan variation increase with the increase of the sampling time is not allowed, which indicates an incorrect measurment process. The measurement results on Figure 1 show that the voltage difference of the reference Zener DC voltage standards of the FEIT Metrological Laboratory and the FER accredited Primary Electromagnetic Laboratory is small, and slightly decreases with the measurement time. The slight decrease of voltage difference may be explained with the short environmental stabilization of the FEIT reference standard prior the measurement. The uncertainty of measurement in the inter-comparison was also small, around 1 μV, with the main contribution of the uncertainty of the output value of PEL standard, calculated for the day of inter-comparison according to the weighted regression line (smaller than 1 μV).

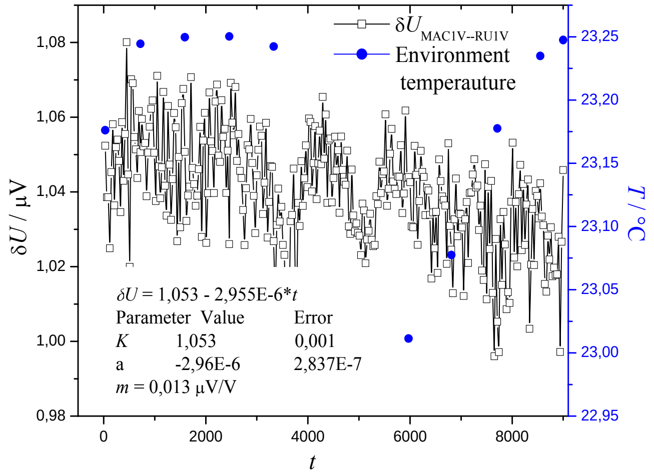

Figure 1. 1 V-Voltage difference versus temperature and time

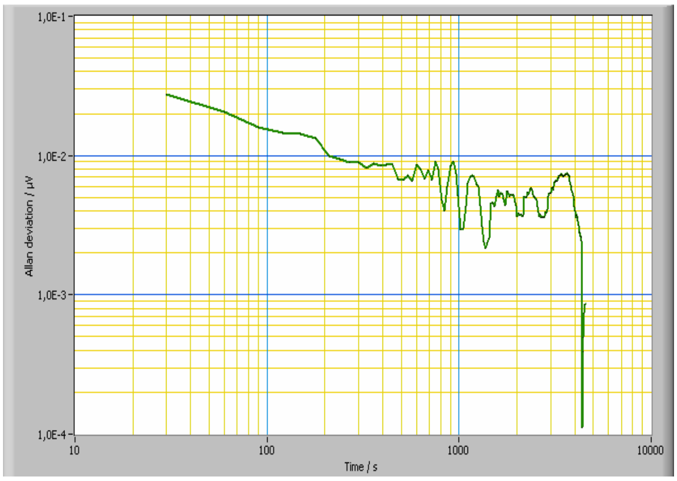

Figure 2. Allan deviation versus sampling time τ

IV. Conclusions

The inter-laboratory comparison of the DC reference standards of 1 V and the resistance standards of 1 Ω and 10 kΩ of the FEIT – Skopje and the FER-Zagreb was described. The measurement results show that the voltage difference of the reference FEIT and the FER Zener DC voltage standards is small. The uncertainty of measurement in the calibration of the voltage was estimated in order of 10^{-6} V, what is comparable with the direct calibrations of electronic voltage standards via a Josephson voltage standard, were the achievable uncertainties of some 10^{-8} V, are reduced to the order of 10^{-6} V when the short-term random fluctuations of the output voltages are taken into account. The used improved resistance comparison method for calibration of resistance standards, the calibration system, and laboratory conditions, as well as the experienced staff, contributed to the high precision of the calibration measurements and small uncertainty. The improved expanded model for uncertainty estimation and the calculations by GUM, mainstream procedure and Monte Carlo method, showed small calibration uncertainty-high calibration quality. For the future work, it is planned a regular inter-laboratory comparison and research of the factors influencing the stability of the resistance and voltage standards and the quality of the calibration.

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