2025 IMEKO Joint Conference
TC8 - TC11 - TC25
Torino, Italy, September 14-17, 2025

Assessing Measurement Consistency of Reference Standards through Intra-Laboratory E_n Criteria Analysis

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***Abstract* – Adherence to ISO/IEC 17025:2017 requires inter-laboratory comparisons and proficiency testing, alongside other measures, for ensuring test and calibration data validity. This study explores intra-laboratory comparison, as an additional quality assurance method. The presented procedure is conducted in an accredited calibration laboratory, by comparing results sourced/measured with multiple reference standards, including a high-voltage decade resistor, 8 ½ and 6 ½ digit multimeters, and a multifunction calibrator with a high-resistance measurement adapter, all of them covering the range of high resistance, above 100 MΩ. By applying the ISO/IEC 17043:2023 statistical methods and E_n criteria, the quantitative assessment of the measurement consistency is achieved, thus enhancing the confidence in the laboratory's standards.**

Keywords: Intra-laboratory comparison, calibration of electrical quantities, ISO/IEC 17043:2023, E_n-criteria, laboratory quality assurance.

I. INTRODUCTION

One of the basic requirements for quality assurance, introduced in the international standard ISO/IEC 17025:2017 [1], which calibration and testing laboratories are supposed to fulfill, is a periodic participation in inter-laboratory comparisons and proficiency testing schemes [2]. These participations enable comparison between results from calibration proceedings and test procedures conducted in two or more laboratories that cover the same or similar scope of accreditation. By comparing the measurement results, laboratories provide an enhanced level of confidence in the data they provide to their customers.

According to [1], laboratories are committed to introduce other measures for quality assurance as well [3]. Such a procedure may include comparison between calibration, or testing, results performed by several different performers, using the same equipment. Other example is a periodic checkup of the laboratory's equipment with a lower accuracy class instruments or testing artifacts with prior known characteristics. In this paper, an innovative concept for quality assurance will be presented, referred to as intra-laboratory comparison. Similar to the inter-laboratory comparison, an intra-laboratory comparison is a concept that relies on weighting up the measurement performance of multiple measuring units that cover the same or similar measurement ranges, by implementing a pre-defined criteria [4].

The results of an intra-laboratory comparison will be presented, which is performed in an accredited laboratory for calibration of instruments and reference standards for electrical quantities [5], the Laboratory for Electrical Measurements (LEM) [3]. The laboratory is within the Faculty of Electrical Engineering and Information Technologies (FEEIT) at Ss. Cyril and Methodius University in Skopje (UKIM), and it maintains international traceability to BIPM [6] intrinsic reference standards. The laboratory possess a documented history of participation in both inter-laboratory comparisons and proficiency testing schemes [7-10], in most of its accreditation scope.

In 2023, LEM extended its accreditation scope in domain of extreme electrical resistance and several other electrical quantities. For the purposes of this work, the introduction of a high resistance decade resistor IET Labs, Inc. HRRS-Q-4-1M-5KV [11], into the laboratory's list of equipment is important, in such a manner a capability for calibration of instruments that measure resistance up to 1 TΩ, most notably insulation testers, was ensured. The extension of the accreditation scope and the introduction of new calibration protocols is already presented in several scientific papers [12-14]. The following methodology, is supposed to complement the previous research with the introduction of intra-laboratory comparison in scope of high-resistance calibrations. Beside the decade resistor [11], which serves as a primary reference standard (RS) in LEM, other instruments are implemented as well, such as high resolution digital multimeters and a multifunction calibrator with additional adapter for low current/high resistance measurement. The results that are going to be presented are obtained during a 24 h time period. For calculation of the measurement uncertainty, the concept provided in "Guide to the expression of uncertainty in measurement"-GUM [15] is adopted. For quantitative analysis of the intra-laboratory comparison results, the E_n criteria, introduced in the international standard ISO/IEC 17043:2023 [16], is implemented.

II. MEASUREMENT EQUIPMENT AND SELECTION OF MEASUREMENT POINTS

For the realization of the intra-laboratory comparison three other RSs are used, beside the high voltage decade resistor IET Labs, Inc. HRRS-Q-4-1M-5KV [11], shown in Fig. 1. The decade resistor [11] is traceable to BIPM [6] through the national standards of USA, via NIST [17]. First, there is the primary RS of LEM for both DC and AC voltages and currents, as well as electrical resistance up to 100 MΩ, an 8 ½ digit multimeter, Agilent 3458 A [18], given in Fig. 2. Additional digital multimeter, with a resolution up to 6 ½ digits, that serves as a secondary RS for the aforementioned quantities, FLUKE 8846 A [19], presented in Fig. 3. is also used. Both digital multimeters are traceable to BIPM [6] via the Directorate for Measures and Precious Metals [20], the National Metrological Institute of R. Serbia. The third RS, introduced in the intra-comparison, is the multifunction calibrator Transmille 4015 [21], with adapter for low current/high resistance measurement, EA 008 [22], presented in Fig. 4. This calibrator is traceable to BIPM [6], through the national standards of NPL – UK [23].

Fig. 1. High voltage decade resistor HRRS-Q-4-1M-5KV.

Fig. 2. Agilent 3458 A, 8 ½ digit multimeter.

Fig. 3. Fluke 8846 A, 6 ½ digit multimeter.

Fig. 4. Transmille 4015 multifunction calibrator with high resistance measurement adapter EA 008

Taking into account the different types of measuring instruments used in the intra-comparison, the results are supposed to provide quality assurance, not only in the performances of the single RSs, but in the different methods implemented for high resistance measurement as well.

The lowest resistance that may be sourced from the IET Labs decade resistor [11] is 100 MΩ. On the other hand, the highest resistance that may be recorded with both digital multimeters [18-19] is 1 GΩ. This implies that the intra-laboratory comparison has to be conducted in a very small range of high resistance values, between 100 MΩ and 1 GΩ. The calibrator [21] and its adapter EA 008 [22], have wider measurement range, in such a way they do not have an influence on the selection of measurement points. In Table 1, the best 1 year's specification of all aforementioned RSs is presented, in the range between 100 MΩ and 1 GΩ. The best 1 year's specification is given as a percentage of the sourced/measured value.

Table 1. Best 1 year's specification of the four reference standards in the range between 100 MΩ and 1 GΩ

Reference standard	Best 1 year's specification
IET Labs, Inc. HRRS-Q-4-1M-5KV	±0.1 %
Agilent 3458 A	±0.1 % to ±1 %
FLUKE 8846 A	±0.8 % to ±2 %
Transmille 4015 with EA008 adapter	±0.5 %

Based on the data presented, it may be highlighted that the decade resistor [11] will serve as a reference (pilot) instrument, while the indications of all other measuring units will be evaluated using the E_n criteria according to the international standard ISO/IEC 17043:2023 [16]:

$$E_n = \frac{R_{par} - R_{ref}}{\sqrt{U_{C,par}^2 + U_{C,ref}^2}}, \tag{1}$$

where R_{par} is the recorded resistance with one of the digital multimeters [18-19], or the multifunction calibrator [21-

22], R_{ref} is the reference resistance, set on the decade resistor [11], $U_{C,par}$ is the expanded combined uncertainty accompanied to the result obtained with one of the RSs that participate in the intra-laboratory comparison, while $U_{C,ref}$ is the reference uncertainty attributed to the set up value on the decade resistor [11]. According to [16], if the calculated E_n criteria value in a single measurement point is between -1 and +1, the result of the intra-comparison is denoted as PASS, otherwise it stands as FAIL.

III. MEASUREMENT UNCERTAINTY EVALUATION

The uncertainty accompanied to the measured value, recorded with any of the instruments participating in the intra-comparison, as well as the uncertainty of the reference resistor, are calculated according to the concept presented in GUM [15]. The overall uncertainty, accompanied to a measurement result, is evaluated as standard combined uncertainty, calculated by taking into account multiple influencing factors, which may be regarded as Type A or Type B uncertainties. The parameter used in the evaluation of the E_n criteria is the expanded uncertainty, U_C, calculated by multiplying the standard combined uncertainty, u_C, with a coverage factor, k, that corresponds to a pre-defined level of probability, according to the adopted distribution:

$$U_C = k \cdot u_C \,. \tag{2}$$

For evaluation of the reference uncertainty, i.e. the uncertainty from the set up resistance on the IET Labs. decade resistor [11], the approach for mutually uncorrelated input influence factors [15] is used:

$$U_{c,ref} = k \cdot \sqrt{u_{acc}^2 + u_{st}^2 + u_{temp}^2 + u_{cal}^2} \,, \tag{3}$$

where, u_{acc} is the uncertainty component emerging from the specification of the reference standard [11], i.e. the accuracy declared by the manufacturer, u_{st} is the component that carries information about its long-term stability, u_{temp} is uncertainty component that represents temperature fluctuations' influence on the resistor's performance and u_{cal} is the traceability component, i.e. value obtained from the level up calibration of IET Labs, Inc. HRRS-Q-4-1M-5KV [11].

The participating instrument's result, R_{par} in (1) is calculated as a mean value, R_m, of 10 measurements:

$$R_{par} = R_m = \frac{1}{N}\sum_{i=1}^{N} R_{par,i} \,, \tag{4}$$

where $R_{par,i}$ is the single resistance recording and N=10 is the number of measurements. Multiple recordings are performed in order for the statistical random variations of the measured quantity, i.e., Type A uncertainty, u_A [15], to be evaluated. Other influencing factors that contribute to the overall uncertainty budget in the resistance measurement with any of the RSs include:

- instruments' finite resolution, u_{res},
- instruments' accuracy class, i.e. error limits presented by the manufacturer, u_{acc},
- instruments' long-term stability, once again obtained from the technical datasheet, u_{st},
- instrument's level up calibration, i.e. component related to single unit's traceability, u_{cal},
- eventual temperature-related fluctuations that affect the performance of the instrument, u_{temp}.

As mentioned earlier the overall uncertainty is calculated as expanded combined uncertainty, regarding all input influencing factors as mutually uncorrelated [15]:

$$U_{C,par} = k \cdot \sqrt{u_A^2 + u_{res}^2 + u_{acc}^2 + u_{st}^2 + u_{cal}^2 + u_{temp}^2} \,. \tag{5}$$

Taking into account the multiple influencing factors affecting the measurement, the expanded uncertainty is evaluated by assuming Gaussian (Normal) distribution, with approximately 95.46 % probability, therefore k=2.

IV. MEASUREMENT RESULTS AND DISCUSSION

The intra-laboratory comparison is conducted by means of resistance measurement, set up on the reference decade resistor [11], with the presented three RSs. Measurements in three measuring points, corresponding to a set up value of 100 MΩ, 500 MΩ and 1 GΩ are conducted, according to the predefined protocol. In Table 2, the reference uncertainty of the decade resistor [11], $U_{C,ref}$, is presented for the specified resistance values, R_{ref}.

Table 2. Expanded combined uncertainty of the IET Labs. HRRS-Q-4-1M-5KV decade resistor

R_{ref}	$U_{C,ref}$
100 MΩ	±0.12 MΩ
500 MΩ	±0.61 MΩ
1 GΩ	±0.0029 GΩ

Only one data set of 10 recordings is obtained with both digital multimeters [18-19], while regarding the calibrator [21] and its high resistance measurement adapter [22], measurements with three test voltages of 100 V, 500 V and 1000 V are performed. The measurement results, in the form of mean resistance values, as well as the corresponding uncertainties, are presented in Table 3. As can be seen from the table, in the measurement point of 100 MΩ, the 8 ½ digit multimeter [18] provides the best result in terms of both measured value, as well as lowest measurement uncertainty. The expanded uncertainty, attributed to the recorded mean value is almost equal to the reference uncertainty in the same measurement point, taking into account that both artifacts act as primary RSs in LEM for the 100 MΩ resistance value. For the other two measurement points, this multimeter [18], provides higher deviation results in comparison to the recordings performed with the Transmille 4015 calibrator [21], using its high resistance adapter EA008 [22]. The presented expanded uncertainty, for both 500 MΩ and 1 GΩ measurement points, regarding both RSs, has the same order of magnitude value. The uncertainty accompanied to the measured resistance with Transmille 4015/EA008 [21-22] is strongly correlated to the test voltage. As can be seen from Table 3, in the same measurement point, for different test voltages, different uncertainty values are obtained. This is due to the fact that the accuracy related uncertainty component, u_{acc}, as depicted in (5), is related to both the measured value and the measurement range. The measurement ranges vary with the alteration of the test voltage. The working RS of LEM, FLUKE 8846 A [19], provides the measurement results that differ more significantly in relation to the set up reference resistance. These deviations are however covered by the expanded uncertainty attributed to its measurement performance, which is much more significant, in comparison to the other two artifacts that participate in the intra-comparison.

In Table 4 the results from the intra-comparison, in terms of calculated E_n criteria values, are presented. From the data it may be concluded that all three RSs successfully passed the intra-comparison, taking into account that the E_n criteria value is between -1 and +1, in every measuring point, regarding all the participating measuring units.

Table 3. Results from resistance measurements with the three RSs.

RS	R_{ref}	R_{par}	$U_{C,par}$
Agilent 3458 A	100 MΩ	100.0034 MΩ	±0.13 MΩ
	500 MΩ	499.7681 MΩ	±6.2 MΩ
	1 GΩ	1.0015016 GΩ	±0.013 GΩ
Transmille EA008 100 V	100 MΩ	100.001 MΩ	±0.9 MΩ
	500 MΩ	500.1251 MΩ	±6.5 MΩ
	1 GΩ	1.0006 GΩ	±0.009 GΩ
Transmille EA008 500 V	100 MΩ	100.0122 MΩ	±2.5 MΩ
	500 MΩ	500.035 MΩ	±4.5 MΩ
	1 GΩ	1.001 GΩ	±0.025 GΩ
Transmille EA008 1000 V	100 MΩ	99.993 MΩ	±0.9 MΩ
	500 MΩ	500.0425 MΩ	±6.5 MΩ
	1 GΩ	1.0003 GΩ	±0.009 GΩ
FLUKE 8846 A	100 MΩ	99.949 MΩ	±0.94 MΩ
	500 MΩ	497.762 MΩ	±13.5 MΩ
	1 GΩ	0.992633 GΩ	±0.027 GΩ

Table 4. Results from the intra-comparison, E_n criteria

RS	R_{ref}	E_n-criteria	Status
Agilent 3458 A	100 MΩ	0.019	PASS
	500 MΩ	-0.037	PASS
	1 GΩ	0.11	PASS
Transmille EA008 100 V	100 MΩ	0.0011	PASS
	500 MΩ	0.019	PASS
	1 GΩ	0.063	PASS
Transmille EA008 500 V	100 MΩ	0.0049	PASS
	500 MΩ	0.0077	PASS
	1 GΩ	0.039	PASS
Transmille EA008 1000 V	100 MΩ	-0.0077	PASS
	500 MΩ	0.0065	PASS
	1 GΩ	0.027	PASS
FLUKE 8846 A	100 MΩ	-0.054	PASS
	500 MΩ	-0.17	PASS
	1 GΩ	-0.27	PASS

The intra-comparison results, in the form of deviation between the measured and the set up values, with the attributed uncertainties, are illustrated in Fig. 5, Fig. 6 and Fig. 7, for the three measurement points.

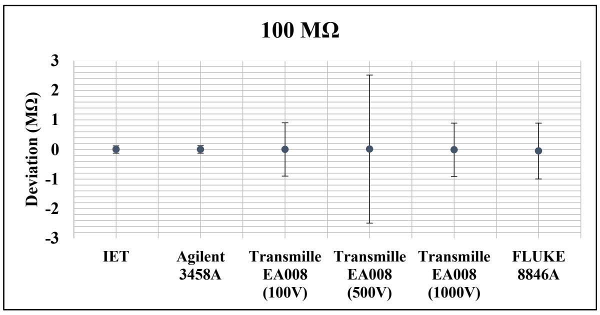

Fig. 5. Intra-laboratory comparison results for 100 MΩ.

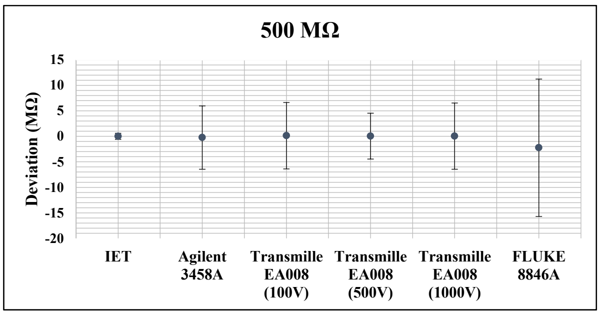

Fig. 6. Intra-laboratory comparison results for 500 MΩ.

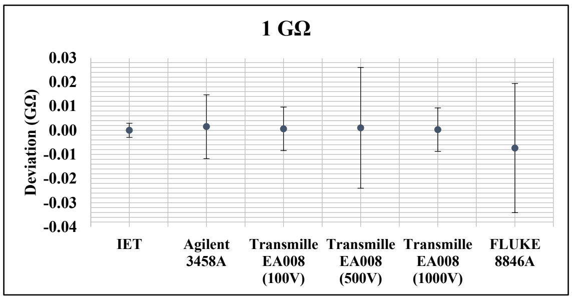

Fig. 7. Intra-laboratory comparison results for 1 GΩ.

V. CONCLUSION

In the paper, results of an intra-laboratory comparison, as an additional quality assurance measure in an accredited calibration laboratory, are presented. The study evaluates the performance of several, high accuracy class, reference standards, in the scope of high resistance measurements.

The performed analysis, based on a pre-defined E_n criteria, indicate that all measuring instruments pass the intra-laboratory comparison, in the selected range. The analysis provides enhanced confidence in the calibration results of the laboratory, indicating that the examined artifacts may be used on equal terms for maintaining an unbroken traceability chain for instruments of lower accuracy class. Taking into account that the reference standards are based on different measuring principles, this protocol provides quality assurance for usage of diferent methods for high resistance measurements, as well.

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