The Strategic Role of Inter-Laboratory Comparison Among International Institutions to Assure Confidence: Report of the INRIM and the European Spatial Agency Comparison on

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ABSTRACT

Inter-laboratory comparisons (ILCs) are an effective mean to establish the compatibility of the measurements among laboratories also belonging to different countries and so belonging to different measurement systems. As consequence, they are a mean to assure confidence in the competence and in the correctness of the unit dissemination processes from national Standards in different countries. This paper refers on an ILC between the National Institute of Metrological Research (INRIM-Italy) and the European space research and technology Centre (ESA- ESTEC), belonging therefore to two different measurement systems, on DC Voltage, DC Resistance and Electrical Capacitance carried out in 2016. The comparison had satisfactory results.

Keywords: Measurement system, measurement compatibility, inter-laboratory comparison, measurement uncertainty, normalized error, low frequency electrical quantities.

1. INTRODUCTION

Nowadays, high level technical products, mainly in aero-spatial sectors, are developed in more than a country. For this reason, to assure that the various parts of the products are correctly assembled the production systems in each country must be under control and the measurements made in the different countries must be compatible. A physical quantity (i.e. the measurand) may be modelled with an estimate measurement value *m* and an estimate standard measurement uncertainty *u* obtained through a measurement process $\mathcal{MP}$ [1]. Every country has its own measurement system $\mathcal{MS}$ that normally involves a National Metrology Institute (NMI), a network of calibration laboratories, a larger network of testing laboratories and a larger number of organizations for which the traceability of the measuring devices to national measurement standards must be assured [2]. In a $\mathcal{MS}$ all the measuring devices are traceable to national Standards, the lines of traceability are under control and defined in traceability diagrams. Thus, the measurement results obtained in the successive steps of the traceability diagrams can be evaluated in terms of the relevant national Standards. A concept connected with that of uncertainty is the measurement compatibility, which plays a central role in the comparison of the measurement results of the same measurand, in well-defined conditions, obtained through measurement processes $\mathcal{MP}_i$ based on different methods and instruments or performed by different laboratories. The measurement compatibility, as defined in [3] is:" Property of a set of measurement results for a specified measurand, such that the absolute value of the difference of any pair of measured quantity values from two different measurement results is smaller than some chosen multiple of the standard measurement uncertainty of that difference". Based on this concept the inter-laboratory comparisons (ILCs) are performed. ILCs can be carried out among NMIs (key comparisons) to establish their degree of equivalence, expressed in terms of deviations from the Key Comparison Reference Value (KCRV) and uncertainty of these deviations. To obtain the KCRV the measurements and uncertainties of all the participating Laboratories are considered, formally treating all the participating Laboratories at the same technical level. For this case, the rules to evaluate the results were given by Cox in [4] and applied for example in the comparisons [5-7]. Another kind of ILCs involves a NMI and secondary Laboratories of the same country, so belonging to the same *MS*. Normally, these Laboratories are accredited by national Accreditation Bodies. To determine the reference values of these ILCs are taken into account only the measurements of the NMI that is considered at an upper metrological level with respect the other Laboratories [8-11]. In the evaluation of these ILCs it is important to correctly calculate the possible correlation between the measurements of the NMI and the secondary Laboratories as often the Laboratories send their reference standards to the same NMI for periodical calibration. Suggestions for this evaluation can be found in [1,8-10]. Further criteria are reported in [12] in which it is argued that the correlation coefficient r, can be defined for each measurement point of an ILC between a NMI and a secondary Laboratory that takes its traceability from it as:

$$r(m_L, m_N) = \frac{u_B^2(std\_{Q1})}{u(m_N) \times u(m_L)} \tag{1}$$

where m_L m_N, $u(m_L)$ and u (m_N) are the measurement results and the standard uncertainties obtained respectively by a Laboratory and by the NMI, while $u_B(mstd\_{Q1})$ is the type B component of the standard uncertainty due to the national Standard of the relevant physical quantity Q_1 and to the unit dissemination process to the reference standards of Q_1. If a measurement needs the traceability from more national standards (i.e. the electrical current needs the traceability from DC Resistance and from DC Voltage) the (1) becomes:

$$r(m_L, m_I) = \frac{u_B^2(std\_{Q1}) + ... + u_B^2(std\_{Qn})}{u(m_N) \times u(m_L)} \tag{2}$$

In this paper an ILC, made according to the EN ISO/IEC 17043 international Standard [13] between the National Institute of Metrological Research (INRIM-Italy) and the European space research and technology Centre (ESA- ESTEC), on DC Voltage, DC Resistance and Electrical Capacitance is discussed.

2. INRIM AND ESA-ESTEC

INRIM is the Italian National Metrology Institute. It realizes and compares with other Institutes primary measurement standards for all SI units except for the ionizing radiation field. It carries out and promotes scientific research focused on metrology, materials science and innovative technologies, as NMI underpins the SI system, disseminates and transfers scientific results, technology and know-how to scientific, industrial and service users. Furthermore, it produces, and coordinates, even within the European Union programs and international organizations, scientific and technological research activities, both through its own facilities or in collaboration with universities and other public and private entities, national and international.

The job of the European Space Agency (ESA) is to draw up the European space programs. ESA's programs are designed to find out more about Earth, our Solar System, as well as to develop space technologies and services, and to promote European industries. ESA also works closely with space organisations outside Europe. In the European Space Research and Technology Centre (ESTEC) most ESA projects are born and guided through the various phases of development. These regard: science, exploration, telecommunications, human spaceflight, satellite navigation and Earth observation. The European Space Research and Technology Centre (ESTEC) develops and co-ordinates most ESA projects on science, exploration, telecommunications, human spaceflight, satellite navigation and Earth observation. In ESTEC operates a metrology laboratory performing mechanical, opto-mechanical and dimensional measurements for the ESTEC Test Centre, ESA laboratories and ESA projects. The Laboratory has primary electrical standards traceable to national and international standards. The Laboratory is ISO 17025 accredited for DC voltage, resistance, electrical capacitance, temperature/humidity by the Dutch Accreditation Council RvA. This Laboratory made the measurements concerning the ILC described in the paper.

3 TRAVELLING STANDARDS OF THE ILC

The traveling standard for DC Voltage was a J. Fluke mod. 732 B Reference standard no. 5650305, belonging to INRIM, to be calibrated at 1.018 V and 10 V (see Fig. 1).
The traveling standards for DC Resistance were respectively: a J. Fluke model 742A-1 Ω no. 437003 (Fig. 2), a 10 Ω Leeds & Northrup mod. 4030-B. no. 1710511 (Fig. 3) and a 1 MΩ Leeds & Northrup mod. 4050-B. no. 1867621 (Fig. 4) standard resistors belonging to INRIM.
The traveling standard for electrical capacitance was an ESI mod. SC1000 1000 pF standard capacitor belonging to ESA to be calibrated at 1 kHz (Fig. 5).

Fig.1. J. Fluke mod. 732 B DC Voltage standard.

Fig. 2.: J. Fluke model 742A 1 Ω standard resistor.

Fig. 3. 10 Ω Leeds and Northrup 4030-B standard resistor.

Fig. 4. 1 MΩ Leeds and Northrup 4050-B high value standard resistor.

Fig. 5. 1 nF ESI SC1000 standard capacitor.

4 INSTRUCTIONS FOR THE CALIBRATION OF THE STANDARDS OF THE ILC

DC Voltage

The Fluke mod. 732B DC Voltage standard had to be calibrated at 1.018 V and 10 V. The calibrations had to be performed at the following conditions:

- At a laboratory temperature of 23.0 °C ± 1 °C (or better stability);
- With sinusoidal AC power supply with RMS voltage of 230 V ± 5 % (or better) and frequency 50 Hz ± 1 % (or better);
- the power supply had to be applied to the instrument at least 48 h before any measurement;

The measurements had to be started after 1 h since the switching the instrument supply from mains to its rechargeable batteries. In these conditions, the standard outputs (at 1.018 V and 10 V), the R_t values provided by the instrument and the measurement time for each output had to be reported.

DC Resistance

The 1 Ω e 10 Ω resistors had to be placed either in a stable oil bath controlled at a temperature of 23.0 °C to insure a short-term instability of 0.1 °C or better, or in free air at a temperature of 23.0 °C with a short-term stability of 0.3 °C or better. The resistors had to be calibrated in the four-terminal configuration at the following measurement currents: if the laboratory used a current comparator method: 30 mA and 100 mA for the 1 Ω resistor and 10 mA and 30 mA for the 10 Ω resistor. The resistors had to be connected by means of shielded and insulated cables. If the laboratory used different methods, it had to report the actual measurement current(s) at which the calibrations were performed and the resistance values when a thermal equilibrium was achieved. The 1 MΩ resistor had to be placed in free air or in an air bath at 23.0 °C ± 1 °C (or better). It had to be calibrated at 10 V in four (or five) terminal configuration. As the resistor fifth terminal, the aluminum ring on its upper external part could be used.

Electrical capacitance

The standard capacitor is equipped with two coaxial connectors respectively marked with H and L. The external conductor of the H terminal is insulated while that of the L terminal is connected to the shield. The calibration had to be performed in the following conditions:

- at laboratory temperature of 23.0 °C ± 1 °C (or better stability);
- With the external conductor of the H and L terminals shorted together with the inner conductor of the L terminal set at the shield potential, but electrically-insulated from it.
- The capacitor power supply had to be a sinusoidal AC voltage of 1.5 V at 1 kHz.

In these conditions, the capacitance between the H and L terminals had to be determined.

5. MANAGEMENT OF THE TRAVELLING STANDARDS

No events took place influencing the ILC and /or the results obtained by the laboratories. The travelling standards were transported by a reliable express courier with a suitable packaging. Upon receipt and before the successive transfer, suitable checks, as established in the protocol, were carried out.

6. REFERENCE VALUES

As ILC reference values were considered the mean values of the INRIM calibrations obtained before and after the calibration at the ESA-ESTEC laboratory. The travelling standards were calibrated twice (in March-April and in June 2016) by INRIM also to check their stability and suitability to the aim of the ILC. ESA-ESTEC measurements were made in May 2016. The measurement results along with their uncertainties of the two laboratories were reported in regular calibration certificates according to their calibration procedures respectively approved by INRIM as signatory of the CIPM MRA[1] and by the Dutch Accreditation Council RvA.

7. EVALUATION OF THE RESULTS

The ILC results were evaluated by means of the normalized error E_n, according to the EN ISO/IEC 17043 international Standard [13]. Therefore, the INRIM reference values (as mean of two calibrations) and the values of the ESA-ESTEC laboratory were respectively defined as:

$$m_I \pm U_I \quad (3)$$

$$m_L \pm U_L \quad (4)$$

Where U_I and U_L are the corresponding expanded uncertainties. From U_I and U_L, the standard uncertainties were obtained: $u_I \cong \frac{1}{2}U_I$ and $u_L \cong \frac{1}{2}U_L$

The following differences were then calculated:

$$y = m_L - m_I \quad (5)$$

Whose standard uncertainties are:

$$u_y^2 = [u_L^2 + u_I^2 - 2u_L u_I \times r(m_L, m_I)] \quad (6)$$

Where $r(m_L, m_I)$ is the correlation coefficient between the measurements at INRIM and at the ESA-ESTEC laboratory. In this case r=0 as the measurements carried out by the two laboratories were independent as made by two laboratories belonging to two different *MSs* and the ESA-ESTEC reference Standards are calibrated at the Dutch NMI, VSL.

Finally, the normalized error E_n was evaluated as:

[1] The CIPM Mutual Recognition Arrangement (CIPM MRA) is the framework through which National Metrology Institutes demonstrate the international equivalence of their measurement standards and the calibration and measurement certificates they issue. The outcomes of the Arrangement are the internationally recognized (peer-reviewed and approved) Calibration and Measurement Capabilities (CMCs) of the participating institutes.

$$E_n = \frac{y}{U_y} \qquad (7)$$

An ILC result is considered satisfactory if $|En| \leq 1$. The ILC results are reported in in Table 1-3. The measurement results are reported as relative difference from the nominal values of the Standards in parts per million.

Table 1. Results of the ILC for DC Resistance. Measurement values and uncertainties are expressed in parts per million.

(Ω)	m_I	$U(m_I)$	m_L	$U(m_L)$	y	$U(y)$	E_n
1[2]	11.1	1.0	3.0	45	–8.1	45.0	–0.2
10^3[3]	43.6	1.0	44.4	45	0.8	45.0	0.0
1 M[4]	49.6	3.0	44	30	–5.6	30.1	–0.2

Table 2. Results of the ILC for DC Voltage. Measurement values and uncertainties are expressed in parts per million.

(V)	m_I	$U(m_I)$	m_L	$U(m_L)$	y	$U(y)$	E_n
1.018	126.3	3.0	126.7	5.0	0.4	5.8	0.1
10	2.8	0.5	3.0	5.0	0.2	5.0	0.0

Table 3. Results of the ILC for electrical Capacitance. Measurement values and uncertainties are expressed in parts per million

(F)	m_I	$U(m_I)$	m_L	$U(m_L)$	y	$U(y)$	E_n
1 n	49.2	7	47.0	10	–2.2	12.2	–0.2

In figure 6-8 the same results are reported in graphical way. Uncertainty bars are related to a 2σ confidence level.

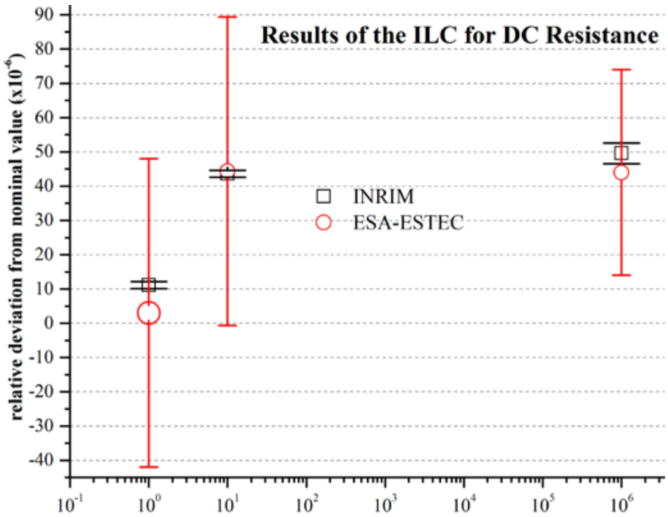

Fig. 6. Compatibility results for DC Resistance.

[2] The laboratory made the measurement at 10 mA. This value was compared with the INRIM measurement at 30 mA.
[3] The laboratory made the measurement at 1 mA. This value was compared with the INRIM measurement at 10 mA.
[4] The laboratory made the measurement at 5V. This value was compared with the INRIM measurement at 10 V.

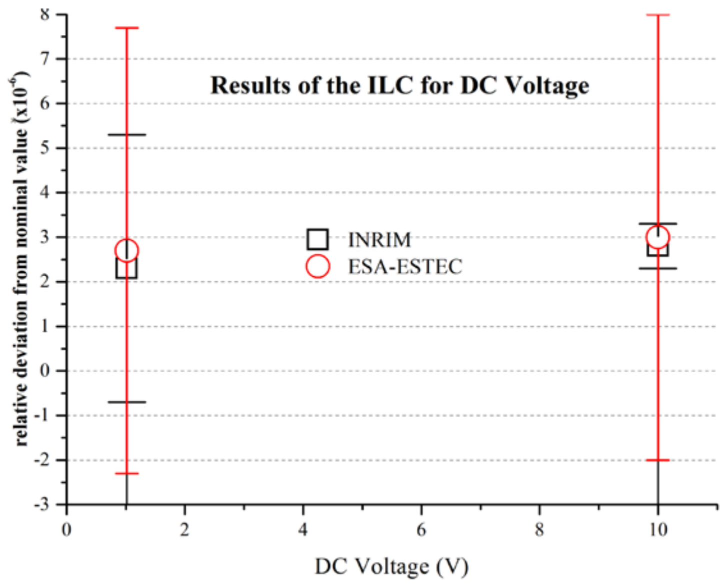

Fig. 7. Compatibility results for DC Voltage.

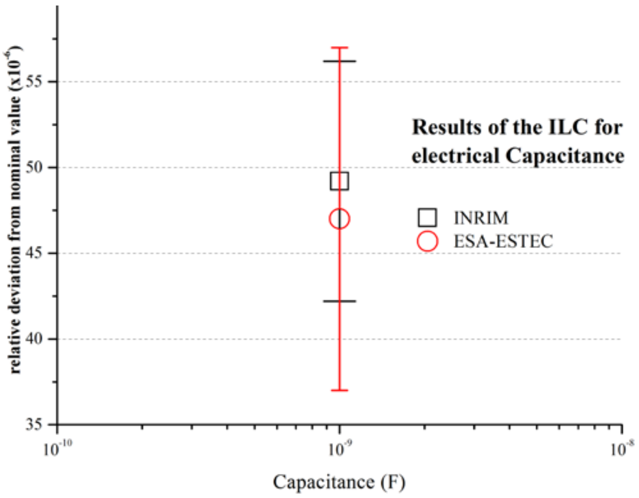

Fig. 8. Compatibility results for electrical Capacitance.

8. CONCLUSION

Tables 1 to 3 and Fig. 6-8 show that ILC had satisfactory results with very low values of the normalized errors E_n. This result means that the ESA-ESTEC laboratory has adequate competence, instrumentation and calibration procedures to sustain its capabilities as accredited by RvA. This result also shows that the agreement between the measurements of two important scientific institutions belonging to different countries and therefore to different *MSs* is satisfactory. These Institutions play a strategic role in addressing scientific and industrial communities and research programs. In addition, this result is a further demonstration that international agreements such as the CIPM MRA or the EA MLA[5] are correctly implemented. Future aim of the collaboration between INRIM and ESA could be an ILC involving as traveling Standard a DC Voltage high accuracy multi-value transportable Standard developed at INRIM [14].

[5] The EA Multilateral Agreement (EA MLA) is a signed agreement between the EA Full Members whereby the signatories recognise and accept the equivalence of the accreditation systems operated by the signing members, and also the reliability of the conformity assessment results provided by conformity assessment bodies accredited by the signing members.

ACKNOWLEDGMENT

The authors wish to thank R. Cerri, P. P. Capra, E. Gasparotto V. D'Elia of INRIM for their valuable contribution in making the measurements and A. Cozzani, Department-Head of Serco Nederland BV for ESA.

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