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LABORATORIES BEST MEASUREMENT CAPABILITY VALIDATION

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Abstract – The Accredited Laboratories at their Certificate have described its best measurement capabilities (BMCs)[1] for the more or less routine calibrations. This BMCs were usually evaluated by interlaboratory comparisons (ILCs), the proficiency testing (PT) schemes as described by the ISO Guide 43. The NMIs traditionally organize the ILCs for the NABs providing the travelling standards, the reference(s) value(s) and at the end perform the statistical analysis of the laboratory results. The goal of this article is to discuss the existing approaches for ILCs evaluation in the calibration laboratories and propose a basis for the validation of the laboratories' BMCs.

Keywords (up to three): interlaboratory comparison, measurement capability

1. BASIC INFORMATION

The proficiency testing (PT), as described by the ISO Guide 43 [1] "is the use of interlaboratory comparisons (ILCs) for purpose of the determination of laboratory testing or measurement performance and one of the main uses of PT schemes is to assess laboratories' ability to perform tests competently. … Participation in PT schemes provides laboratories with an objective means of assessing and demonstrating the reliability of the data they are producing". All the PT schemes have in common the comparison of test and measurement results obtained by two or more laboratories.

The proficiency testing policy and plan are designed by the National Accreditation Boards (NABs) in collaboration with the National Metrology Institutes (NMIs). The type of ILCs are identified in order to comply with the requirements of the regional and international organizations, namely the European Accreditation (EA) and the International Laboratory Accreditation Cooperation (ILAC), needed for the maintenance of these Boards at the multilateral agreements (MLA) of recognition of the calibration certificates or reports issued by a signatory country.

The NMIs traditionally organize the ILCs providing the travelling standards, the reference(s) value(s) and at the end perform the statistical analysis of the laboratory results. These results are evaluated by performance statistics usually the E_n numbers and the different types of scores as defined by the ISO Guide 43 and more recently by the ISO 13528 standard [2]. These performance statistics are obtained by the ratio of the differences between the participating laboratory results and the reference or assigned values[2] and the combined expanded uncertainty (95 %) – E_n numbers, or the combined uncertainty (68%) – *Z, Z', ξ*…scores of these two values.

The best measurement capability (BMC) is defined at the EA-4/02 [3] as "*the smallest uncertainty of measurement that a laboratory can achieve within its scope of accreditation, when performing more or less routine calibrations of nearly ideal measurement standards intended to define, realise, conserve or reproduce a unit of that quantity or one or more of its values, or when performing more or less routine calibrations of nearly ideal measuring instruments designed for the measurement of that quantity*".

In order that an ILC is a reliable tool for a laboratory BMC validation the NMI should provide a travelling standard better – in terms of accuracy class or uncertainty, than the laboratory' BMC. Although this situation encloses the majority of the cases, there are cases were the resolution of the measuring instrument does not permit the requirement of a reference value uncertainty inferior to one third or less of the uncertainty of the participating laboratories.

The goal of this article is to discuss the calibration laboratories ILCs evaluation and propose a basis for the validation of the laboratories' BMCs.

2. GENERAL PRINCIPLES AND CONCEPTS

The new VIM:2007 [4] defines measurement result (result of measurement) as the set of quantity values being attributed to a measurand together with any other *available relevant information* …" and in Note 2 "A measurement result is generally expressed as a single measured quantity value and a measurement uncertainty …" . This is one of the changes from the previous edition where measurement result was defined as a value attributed to a measurand and will require the revision of the standards related with the ILCs as it will be needed that the laboratories indicate a reliable estimate of their uncertainties. This is already a practice in the generality of the calibration laboratories especially in the "physics area" and will be generalized to the other sectors.

[1] This acronym is usually used for Accreditation purposes. It is equivalent to the CMCs (Calibration and Measurement Capabilities) acronym used by the NMIs for MRA purposes.

[2] Assigned value: value attributed to a particular quantity and accepted, sometimes by convention, as having an uncertainty appropriate for a given purpose [2].

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The ISO 13528:2005 standard [2] defines proficiency testing as the determination of laboratory testing performance by means of ILCs and this performance can be described essentially by three properties: laboratory bias, stability and repeatability. For the case of calibration laboratories the ILCs are usually carried on to evaluate the laboratory bias. The travelling standard and assigned value are, as referred, provided by a Reference Laboratory, usually a NMI. The performance statistic preferred is the E_n numbers defined as:

$$E_n = \frac{x - X}{\sqrt{U_{Lab}^2 + U_{ref}^2}} \quad (1)$$

Where X is the assigned value determined by the Reference Laboratory; U_{ref} is the expanded uncertainty of X and U_{lab} is the expanded uncertainty of a participant's result x. The critical value is 1,0 so a result is considered satisfactory to $|E_n| \leq 1$ and unsatisfactory for $|E_n| > 1$. The laboratories uncertainty evaluation should be consistent with the published documents, namely the GUM [5] and the GUM consistent NABs Regional documents, and the Reference Laboratory should guarantee that their uncertainties are reported it in a uniform way.

When the uncertainty of the travelling standard obtained by the Reference laboratory is better than one third of the laboratories uncertainties this value will not influence significantly the comparison uncertainty for a specific laboratory and its BMC can be validated by the ILC by the difference of its value from the assigned value and the "comparison uncertainty" calculated by equation (2), as it is considered that there is independence between the laboratory value and the assigned value.

$$U_{comp} = \sqrt{U_{lab}^2 + U_{ref}^2} \quad (2)$$

The NABs ask frequently the NMIs to evaluate the performance of the accredited laboratories when calibrating industrial measuring instruments (MIs). These services usually are not provided by the NMIs and in these cases the NMIs have to buy the MIs, perform its characterization, specially its stability and finally to calibrate the instrument before delivering the travelling MI to the participant laboratories. The MI is re-calibrated at the end of the comparison and the reference value will be the mean of this two calibrations. Depending of the participant laboratories number and instrument type the travelling MI may return to the Reference Laboratory for other calibrations.

Normally these MIs have a fixed resolution that became the major standard uncertainty component as the case of digital balances, micrometers, etc.

Even with the best instrumentation the NMI is not able to "decrease" this intrinsic component of uncertainty and for this reason the reference value will have an uncertainty that will "penalise" more or less 40 % the laboratory comparison uncertainty[3]. The BMC's laboratory for this reason cannot be validated.

[3] For the cases where the participating laboratory have an uncertainty similar to the reference laboratory calibration certificate.

It is proposed in this cases to use the "Procedure A" as described in [6] for the evaluation of NMIs key comparison data. In this procedure it is calculated a reference value by the weighted mean of the laboratories values. This estimator uses as weights the inverse of the squares of the associated standard uncertainties.

$$X = \hat{\mu} = \frac{\sum_i \psi_i \cdot x_i}{\sum_i \psi_i} \quad \therefore \quad \psi_i = \frac{1}{u^2(x_i)} \quad (3)$$

$$\frac{1}{u(X)} = \sqrt{\sum_i \psi_i} \quad \text{or} \quad u(X) = \frac{1}{\sqrt{\sum_i \psi_i}} \quad (4)$$

This consensus value will be considered as the "assigned value" of the ILC if all the results are consistent statistically. So it is needed to perform a consistency test, assuming the "normality of the data" obtained, using the statistic:

$$\chi_{\text{obs}}^2 = \sum_i \frac{(x_i - X)^2}{\psi_i} \quad (5)$$

Under the null hypothesis that all n measurement results are consistent and independent of each other, this statistic follows a χ^2 distribution with n–1 degrees of freedom.

Calculating the critical value of this sampling distribution for a given significant level (usually $\alpha = 5\%$) we accept the null hypothesis if:

$$\chi_{\alpha,n-1}^2 > \chi_{\text{obs}}^2 \quad \therefore \quad \alpha = 5\% \quad (6)$$

and the reference value with the corresponding uncertainty will be the assigned value for the ILC.

If the null hypothesis is false, for the cases where the statistic χ_{obs}^2 is superior to the critical value, it means that not all measurement results belong to the same population.

This situation is typical of most ILCs data sets. The discrepant values should be identified by:

$$|x_i - X| > U_{\text{comp}} \quad (7)$$

After discarding the discrepant values the remaining ones will be used to calculate the weighted mean and the assigned value will be found.

In all cases where the assigned value is a calculated one the comparison uncertainty U_{comp} will be smaller due to the dependence between the laboratory value and the assigned value as described in [6] App. C.

$$U_{comp} = \sqrt{U_{lab}^2 - U_{ref}^2} \quad (8)$$

in this case the E_n formula should change to:

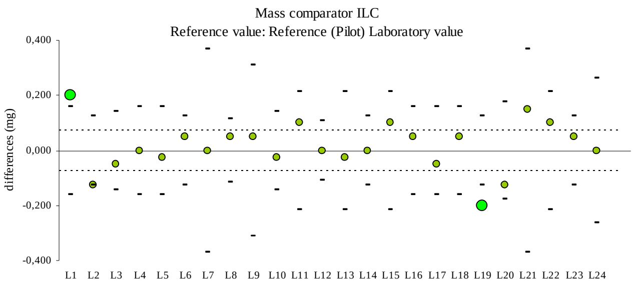
$$E_n = \frac{x - X}{\sqrt{U_{lab}^2 - U_{ref}^2}} \quad (9)$$

As it can be seen in the following example this reference value will have an uncertainty compatible to the laboratories BMC's validation.

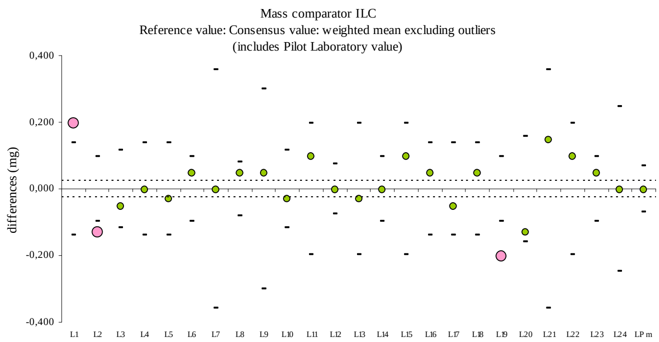
3. ILC EXAMPLE

3.1 ILC- Mass Comparator

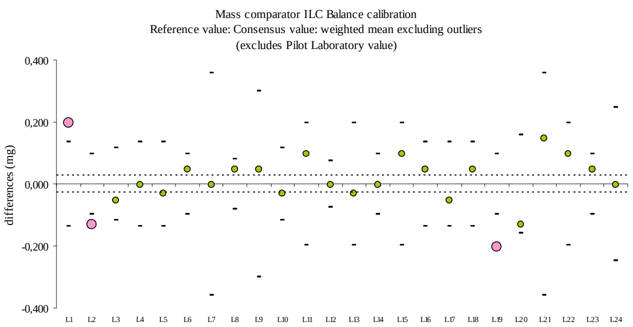
This ILC was performed with a digital balance with a 0,1 mg resolution. To the 24 participants laboratories was asked to calibrate a balance at four points within its measuring interval. The balance was at the NMI mass laboratory room under stable ambient conditions. The ILC results for m = 50 g are the following (Fig. 1).



(a)



(b)



(c)

Figure **1.** (a) Zero line: Reference (Pilot) Laboratory value *(X)*: 50,00017 g; Doted line: $U(X)$: 0,075 mg. Outliers green large dots. (b) Zero line: Calculated assigned value excluding outliers (rose dots) *(X)*: 50,00017 g; Doted line: $U(X)$: 0,026 mg. The Reference laboratory was included at the calculation (LPm) of the assigned value.(c) Zero line: Calculated assigned value excluding outliers (rose dots) *(X)*: 50,00017 g; Doted line: $U(X)$: 0,028 mg. The Reference laboratory was not included at the assigned value.

The MI drift was controlled daily by the Reference Laboratory during this three week exercise. A complete calibration was performed each week and the MI shown a very good stability during all the exercise.

From the figure 1 it can be noticed that the expanded uncertainty of the reference value in the b) and c) graphs is more or less one third of the Reference Laboratory calibration uncertainty (a) graph).

It can be noticed also the small influence of the Reference Laboratory value and uncertainty in the assigned value uncertainty, b) and c) graphs, as there is a significant number of participating laboratories.

If we take as example for the BMC validation the result of laboratory L12 that has an expanded uncertainty of 0,080 mg similar to the Reference Laboratory uncertainty, 0,075 mg, we verify that the comparison uncertainty calculated by eq. 1 is 0,11 mg and by eq. 8 is 0,080 mg.

In the first case we are not able to validate the L12 laboratory BMC even with a result practically at the “reference line”. Calculating by eq. 8 we are able to validate its BMC.

CONCLUSIONS

It was proposed a procedure for the evaluation of Proficiency testing by ILCs performed with calibration laboratories and BMC laboratory validation for the cases where the resolution of the travelling instrument is the major standard uncertainty component.

ACKNOWLEDGMENTS

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References

[1] ISO/IEC Guide 43-1:1997, Proficiency testing by interlaboratory comparisons. Part 1: Development and operation of proficiency testing schemes.

[2] ISO 13528:2005, Statistical methods for use in proficiency testing by interlaboratory comparisons.

[3] EA-4/02 Expression of the Uncertainty of Measurement in Calibration”, 1999.

[4] ISO/IEC GUIDE 99:2007, International vocabulary of metrology - Basic and general concepts and associated terms (VIM).

[5] BIPM et al, Guide to the Expression of Uncertainty in Measurement (GUM), 2nd ed., International Organization for Standardization, Genève, 1995.

[6] M. G. Cox, The evaluation of key comparison data. *Metrologia*, 39, pp. 589-595, 2002.