VIII AMCTM2008

PROFICIENCY TESTING FOR CALIBRATION LABORATORIES

EDUARDA FILIPE

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Outline

1. General concepts
2. ILC evaluation
3. Laboratories uncertainty validation
4. ILCs examples
5. Conclusion

Proficiency Testing definition

ISO/IEC Guide 43-1:1997 - Proficiency testing by interlaboratory comparisons

and

ISO 13528:2005 - Statistical methods for use in proficiency testing by interlaboratory comparisons

> is the use of interlaboratory comparisons (ILCs) for purpose of the determination of laboratory testing performance

Introduction

Participation in PT schemes provides laboratories with objective means of assessing and demonstrating the reliability of the data they are producing”

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.

Introduction

- In order that an ILC is a reliable tool for a laboratory to validate their measurement capability it is needed that the NMI provides a travelling standard better – in terms of accuracy class or uncertainty, than the accredited best measurement capability (BMCs) of the laboratories

- There are cases were the resolution of the measuring instruments does not permit the requirement of a reference value uncertainty inferior to one third of the uncertainty of the participating laboratories

ILC evaluation

Performance statistics as defined by the ISO Guide 43 and ISO 13528 standard

- Scores Z, Z', ξ

Ratio of the differences between the participating laboratory results and the reference or assigned values and the combined uncertainty

- E_n numbers

Ratio of the differences between the participating laboratory results and the reference or assigned values and the combined expanded uncertainty

assigned value: value attributed to a particular quantity and accepted, sometimes by convention, as having an uncertainty appropriate for a given purpose

General Concepts

The new VIM:2007 defines measurement result (result of measurement) as the

"set of quantity values being attributed to a measurand together with any other *available relevant information* ...

Note 2 "A measurement result is generally expressed as a single measured quantity value and a measure-ment uncertainty …

previous edition → measurement result was defined as a value attributed to a measurand

Performance Statistic for Calibration Laboratories

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

X is the assigned value given 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

Critical value: 1,0

Satisfactory result $|E_n| < 1$ Unsatisfactory result $|E_n| > 1$

Reference Laboratory should guarantee that laboratories uncertainties are reported it in a uniform way and are consistent with the published documents

Laboratories uncertainty validation

If (ISO 13528) $u_{ref} < 0{,}3\ u_{lab}$

u_{ref} will not influence significantly the comparison uncertainty for a specific laboratory

$U_{comp} = \sqrt{U_{lab}^2 + U_{ref}^2}$ independence between x and X

→ its BMC (CMC) can be validated by the ILC

by the difference of its value from the assigned value and the "comparison uncertainty"

Laboratories uncertainty validation

- 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

→ NMIs have to buy the MIs, perform its characterization, stability and calibration

Laboratories uncertainty validation

→ Industrial MIs usually have a fixed resolution

→ Major component of uncertainty (case of digital balances, callipers, etc…)

→Even with the best instrumentation the NMI is not able to "decrease" this intrinsic component of uncertainty

→The reference value will have an uncertainty that will "penalise" the laboratory comparison uncertainty

→ The BMC's laboratories cannot be validated

Laboratories uncertainty validation

It is proposed to use the "Procedure A" as described by Cox M. for the evaluation of NMIs key comparison data

Reference value → the weighted mean of the laboratories values

Weights → the inverse of the squares of the associated standard uncertainties.

Laboratories uncertainty validation

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

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

Laboratories uncertainty validation

Weighted mean: "assigned value" of the ILC if all the results are consistent statistically

Consistency test

Statistic

$$\chi^2_{obs} = \sum_i \frac{(x_i - X)^2}{\psi_i} \rightarrow \chi^2_{\alpha,n-1}$$

(it is assumed the "normality of the data")

Null hiphoteses is true for $\chi^2_{\alpha,n-1} > \chi^2_{obs} \therefore \alpha = 5\%$

Reference value with the corresponding uncertainty
→ Assigned value for the ILC

Laboratories uncertainty validation

Null hypothesis is false $\chi^2_{\alpha,n-1} < \chi^2_{obs} \quad \therefore \quad \alpha = 5\%$

Not all measurement results belong to the same population

→ This situation is typical of most ILCs data sets

5. Identify discrepant values $|x_i - X| > U_{comp}$
6. Discard the discrepant values
7. Calculate the weighted mean the remaining values
8. And the assigned value is found.

Laboratories uncertainty validation

due to the dependence between x and X

$$U_{comp} = \sqrt{U_{lab}^2 - U_{ref}^2}$$

E_n changes to

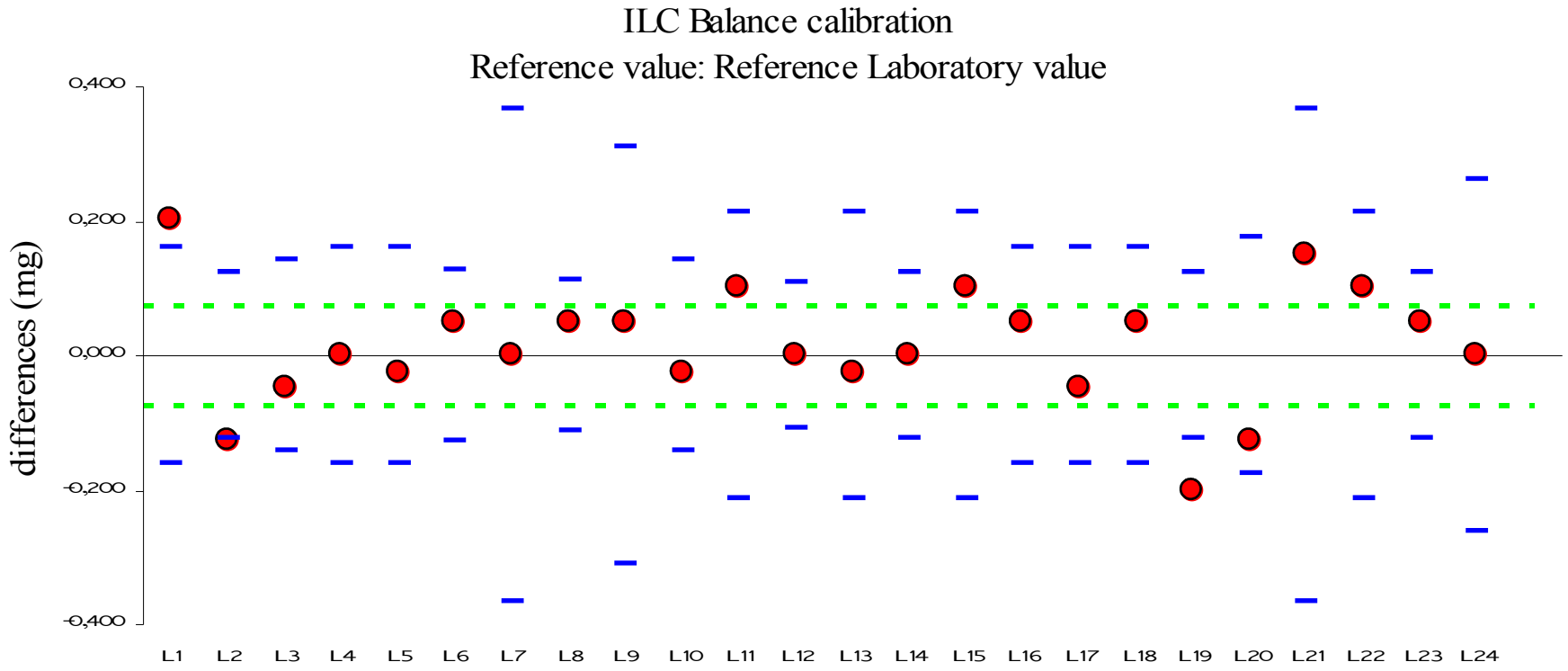
$$E_n = \frac{x - X}{\sqrt{U_{Lab}^2 - U_{ref}^2}}$$

$\rightarrow X$ will have an uncertainty compatible for the ILC

$\rightarrow$ BMC validation as U_{comp} is smaller then U_{lab}

ILC - Balance calibration (m = 50 g)

Digital balance with a 0,1 mg resolution; 24 participants laboratories asked to calibrate a balance that was in the NMI laboratory under stable conditions.



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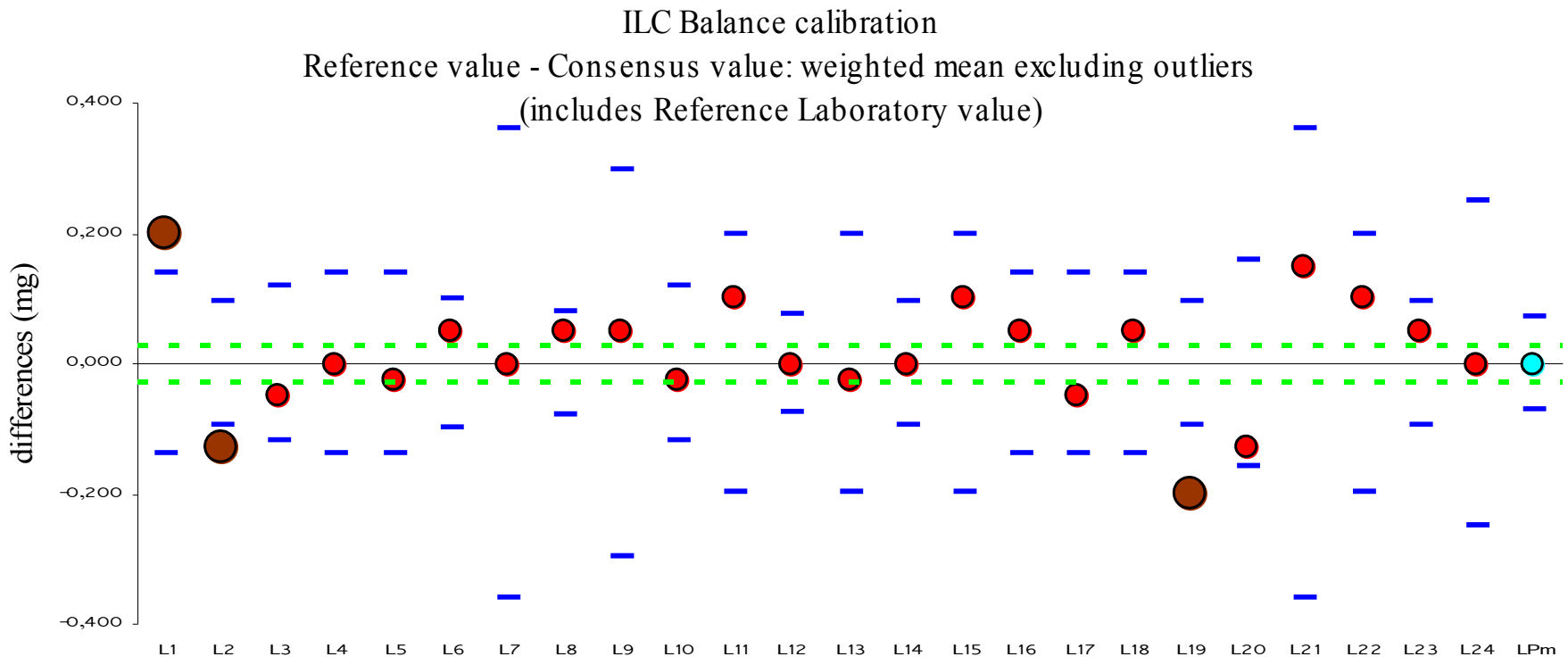

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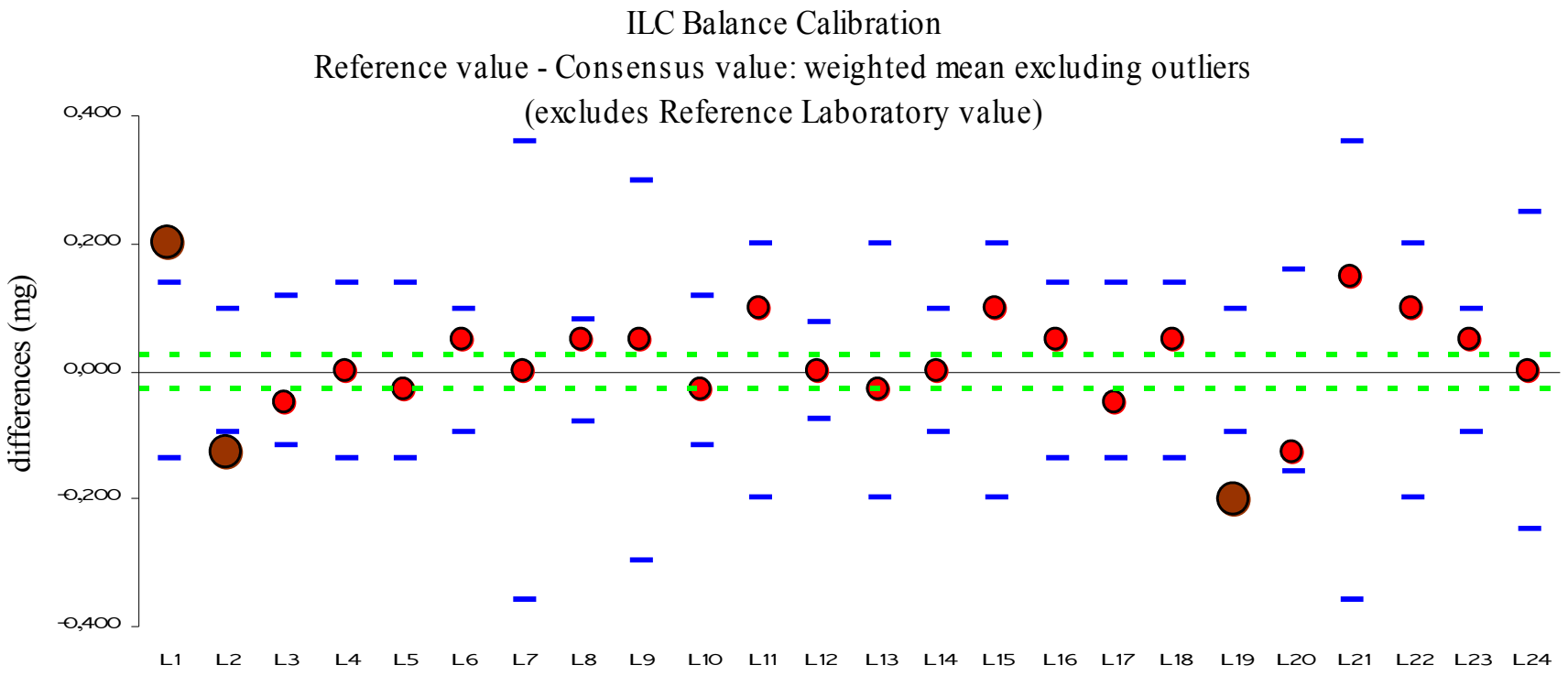
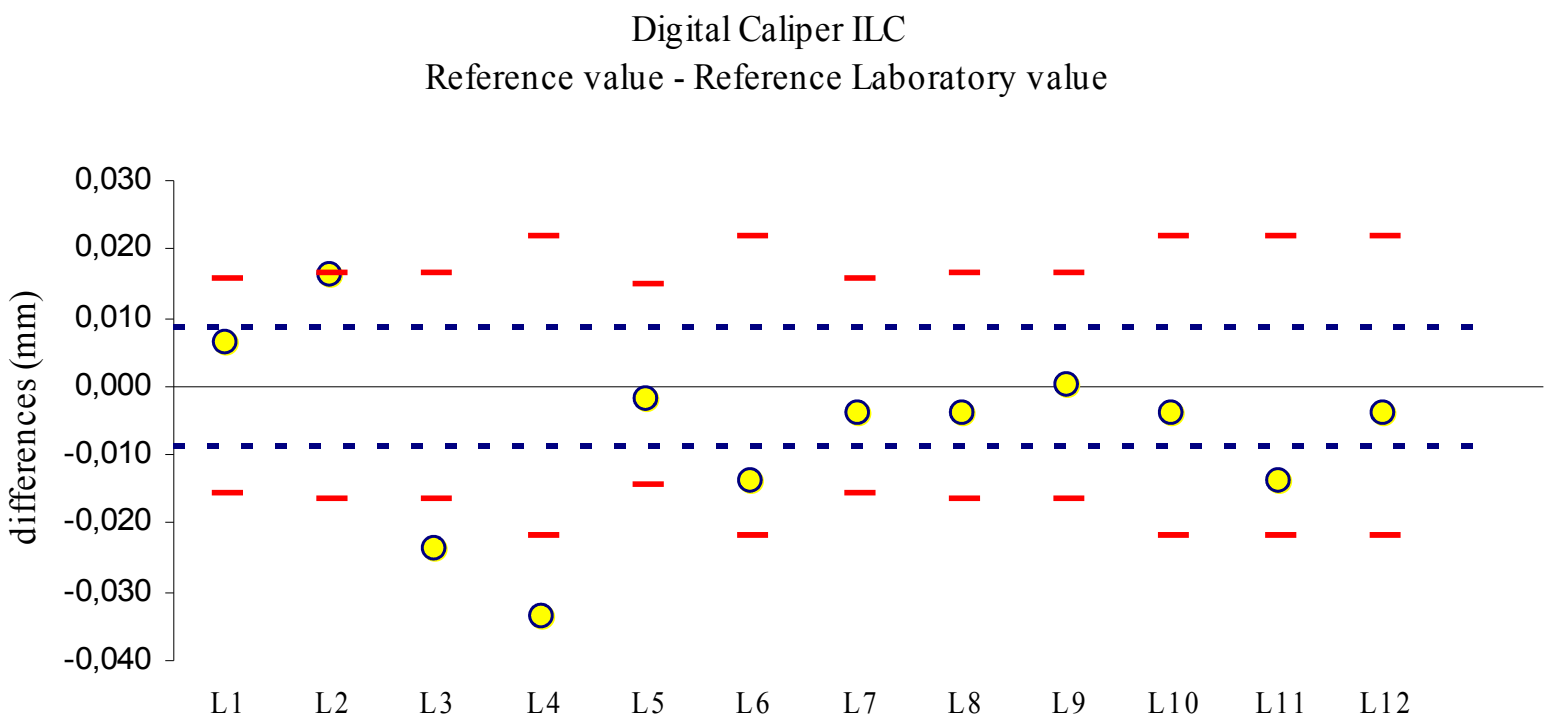


ILC - Digital Caliper calibration (*l* = 300 mm)

Digital caliper with a 0,001 mm resolution; Circulation among 12 participant's laboratories



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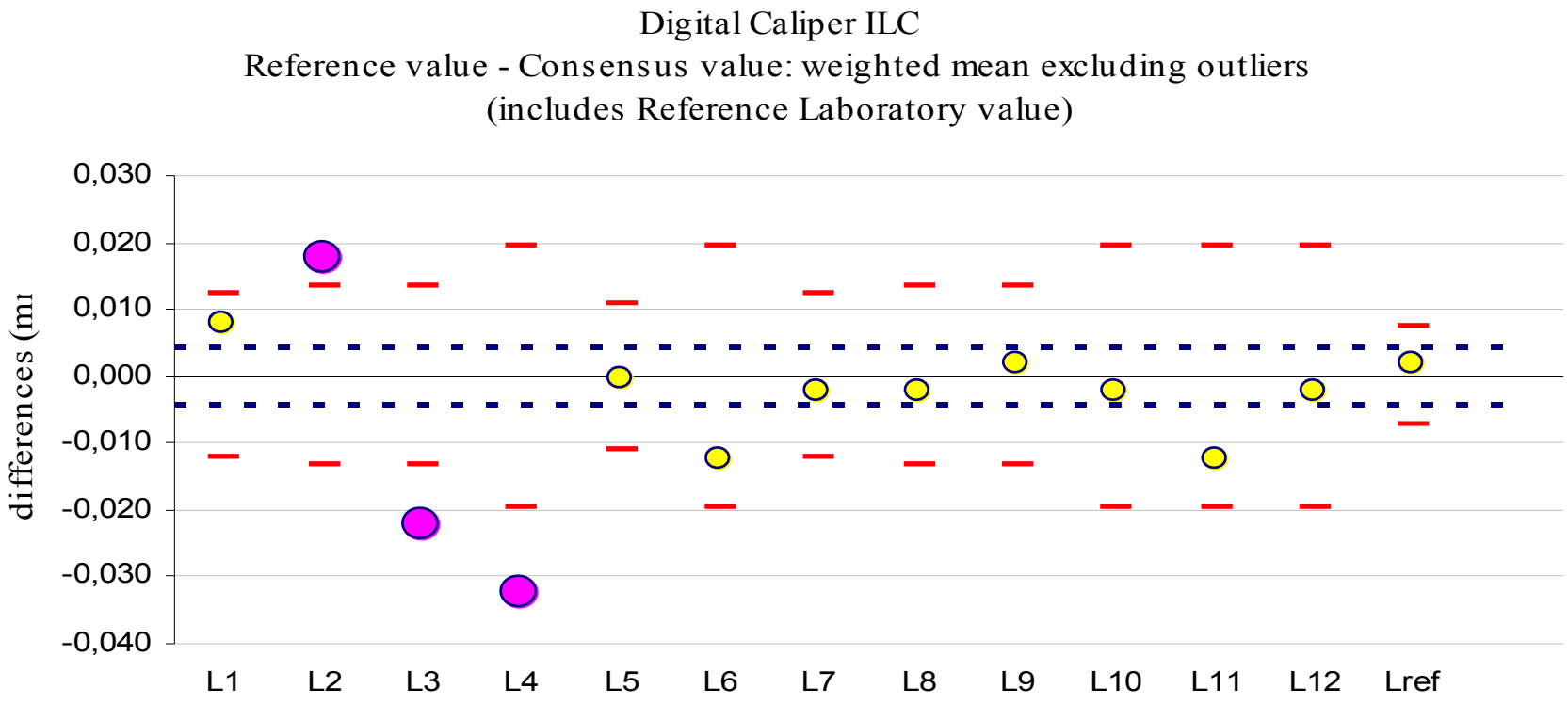

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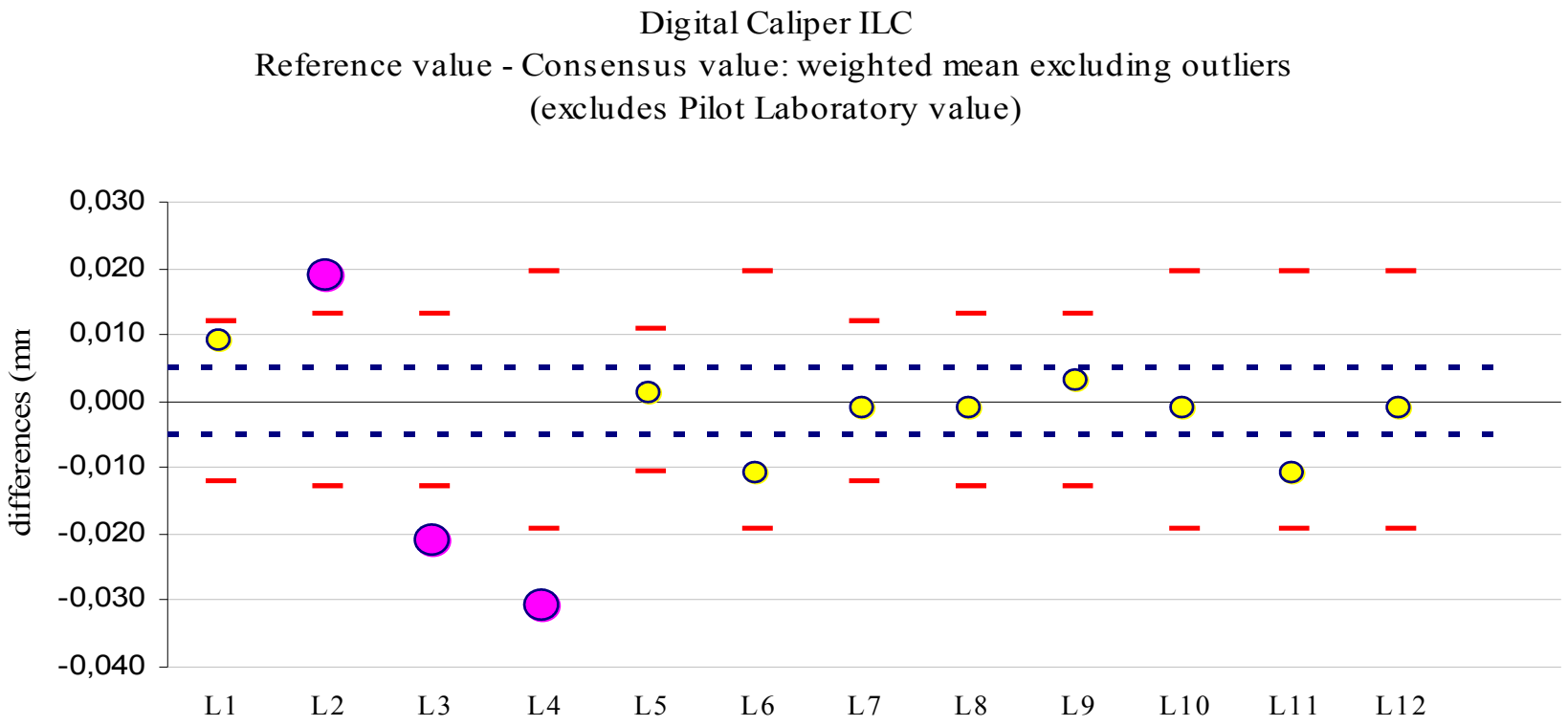

Conclusion

It was proposed:

- a procedure for the evaluation of Proficiency testing by ILCs performed with calibration laboratories for the cases where the limited resolution of the measuring instruments does not permit the requirement of a reference value uncertainty inferior to one third of the uncertainty of the participating laboratories.

References

- ISO/IEC Guide 43-1:1997 - Proficiency testing by interlaboratory comparisons. Part 1: Development and operation of proficiency testing schemes
- ISO 13528:2005 - Statistical methods for use in proficiency testing by interlaboratory comparisons
- ISO/IEC GUIDE 99:2007 - International vocabulary of metrology - Basic and general concepts and associated terms (VIM)
- Cox M.G. The evaluation of key comparison data. *Metrologia*, 2002, 39, 589-595

Thank you!