To participants

Report on an interlaboratory comparison (ILC) concerning the calibration of an extensometer used in tensile testing machines.



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Abstract

This report presents the outcome of an intercomparison/ILC in calibrating an extensometer.

The calibrated object is an Extensioneter with a range of 12,5 mm, MTS 10638748D. The comparison focused on the stated indication errors found by the participants with respect to their own reference and their reported measurement uncertainties.

The outcome of this comparison is presented by showing those data in tables and diagrams thus giving a qualitative picture of the conformity between the laboratories work. It is, however, custom to also inform about quantitative measures how close or distant the various results are from a reliable reference incorporating the stated uncertainties as well. This is typically accomplished using the En-criteria for each participant and each measuring point.

In a comparison of this kind, it is not possible to provide such a neutral reference from a laboratory with high credibility. The only way to achieve asked En-values is to determine a refence value for every indication error as a consensus value based on the available calibration data delivered by the participants.

In the current comparison the number of results is small n=4. The participants coming from Denmark, Sweden, Estonia, Rumania, and Kenya. The calibrations were made on 7 points.

The number of measuring points are 28 and 7 got a En-value above1.

Purpose and implementation of the comparison

This interlaboratory comparison serves as a tool to verify results from the calibrations carried out by calibration laboratories. It is an effective method to demonstrate the technical capacity of the participant and serves as a technical base for accreditation as required by ISO/IEC 17025:2017 (SS-EN ISO/IEC 17025:2018) as specified in point 7.7.2.

Advisory group

A part of the work as an accredited organiser of proficiency testing schemes (PT/ILC) is to establish professional reference groups related to the actual subject.

The advisory group in this case consists of Aykurt Altintas, Denmark, Peter Lau MNE Konsult and Håkan Källgren Swedish Metrology and Quality.

Date	Lab	Extensometer
Week 16		
2023-04-17	Preparations	
2023-04-18	Force Technology, Denmark	Х
2023-04-19		
2023-04-20	MTS System AB, Sweden	X
2023-04-21	RISE Research Institutes of Sweden	
Week 17		
1023-04-24	AS Metrocert Estonia	Х
2023-04-25-27	KENYA BUREAU OF STANDARDS,	
	Kenya	
2023-04-26-27	SARTOROM IMPEX SRL, Romania	X

Participants in the intercomparison and time schedule.

Calibration instructions

The basic instructions to participate in the intercomparison were found here: <u>ILC in force, torque,</u> <u>hardness, and related areas – SMQ Conference (smquality.se)</u>

The laboratories were advised to use their own calibration procedures with focus on agreed calibration points described below. They should use their own mechanical equipment and the software they normally use.

Agreed calibration points.

Extensometer length, mm
1
2
3
4
5
10
12,5

Planning and instruction details

For protocolling the participants received excel sheets (enclosed in Annex) to fill in and deliver to the organizer before leaving the site.

The participants were asked to send calibration certificates to the organiser within one week after finishing the calibration.

Administrative information

Address to send the required documents:

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Analysis of the calibration results using En-values

For a quantitative description of the comparison outcome En-values were calculated for all participants (eq. 1) and all calibration points. This measure uses the deviation of a reported indication values from a derived reference value. By dividing this deviation for each calibration point with the uncertainty in this difference it is possible to set limits for a reasonably allowed discrepancy with respect to the reported measurement uncertainty.

$$E_n = \left| \frac{x_i - x_{ref}}{\sqrt{U_i^2 + U_{ref}^2}} \right| < 1$$
 (eq. 1)

x_i: Single measurement result (error of indication); the index i counts the various participants.

x_{ref}: Calculated inter-comparison reference value – based on a chosen consensus.

Ui: The estimated expanded uncertainty (k=2) stated by each laboratory for each calibration point.

 U_{ref} : The estimated expanded uncertainty (k=2) of the reference value for the same calibration point.

The indication error is the difference between the individually recorded instrument readings and the records from the reference equipment used by the participants for their calibration. A calibration result is generally accepted if its En-value is between -1 and +1.

Assigned inter-comparison reference value for consensus.

The preferred option to assign a reference value is to use the weighted mean among the presented calibration results for each measurement point. The main argument is that it in contrast to the arithmetic mean also considers the stated uncertainty with each result. Consequently, results having low uncertainties can dominate this reference drastically over those results stated with larger uncertainties. This is a fair decision if all uncertainties are credible. A prerequisite, however, is that results with low uncertainty need to lie closer to the reference they form as results with larger uncertainties. If they do not fulfil this claim, they must be excluded from taking part in building the consensus value. The decision whether a result must be excluded is based on a chi-square test that determines if all values in the tested data sample belong to the same distribution with a certain probability. If this is not the case one results must be removed and the test be repeated. Eventually more than one results must be discarded from the data sample until the rest is regarded as consistent. For a large sample this method is very useful and convincing. For small samples as in this comparison it is not transcendent.

Another way to form a consensus value disregarding the reported uncertainties is to use the arithmetic mean. In this case its uncertainty depends on the spread in the sample, meaning the reference uncertainty can be too large for a reliable En-calculation. Before using the arithmetic mean of a sample an eventual outlier too must be detected and removed. This is decided using Grubb's outlier test. Here a result lying most distant from the mean is statistically tested based on the samples standard deviation. Failing the test means it must be as well excluded from the calculation of the mean and eventually a new test is performed on the rest. Even here small samples do not provide reliable statistics and the uncertainty of such a calculated consensus value is mainly due to the spread in the remaining sample. In this inter-comparison the decision was in favour of the arrhythmic mean due to a lower reference uncertainty, which in turn is due to lower spread compared to the uncertainties in the remaining results after the Grubb's test was performed.

Traceability of reference values

The traceability of the reported values was demonstrated by the participants via documenting their reference equipment in their calibration certificates. The laboratories equipment was calibrated by accredited laboratories or National Metrology Institutes.

Extensometer calibration—Machine MTS 10638748D

Four participants P1, P3, P4 and P5 had the capacity to perform this calibration at 7 measurement points on equipment/ machine MTS 634.12F.51. Two of them P1 and P5 adjusted the object (extensometer) and two P3 and P4 the reference equipment to even extension values. The headlines presented in the calibration certificates does not make it self-evident which is the object and which the reference. Only one P1 explicitly expressed the error (in relative units). The others did not give this information at all. The evaluation had to interpret what exactly was meant with *Length of standard measure, Calibration result, Average of measured value, Displacement, True value, and Extension* to calculate error values for comparison. Diagram 1 shows an overview of these interpretations in absolute numbers, with three participants achieving positive and one negative error values. Please se also **Corrections/Changes** after the participants comments/finding to the Draft report.



Diagram 1. Summary of 4 calibration results with belonging uncertainty bars. Those of participant P1 are within the size of the used symbol.

Comment: Due to the obscureness of the not expressed errors no reference values are included in this draft.

Looking to the width of uncertainties (factor 3 to 37 between minimum and maximum extension) the most reasonable way to define a consensus value seemed using the arithmetic mean. It then also immediately becomes clear that the P1 data will be excluded in this calculation (Grubbs outlier test) except for the first extension point at 1mm.

Table 1. Reported error at 1 mm.			
Partici-	Stated indi-	Stated	En-
pant	cation error	uncertainty	value
	[%]	[%]	
P1	-0,18	0,06	-1,40
P3	0,14	0,10	0,47
P4	0,15	0,30	0,28
P5	0,10	0,15	0,22
R	0,054	0,154	

Table 1. Reported ciror at 1 min



Comment: For the first extension point the Grubbs outlier test states that all results still belong to the same normal distribution. The standard deviation, however, produces a large reference uncertainty.

rable 2. Reported error at 2 mm				
Partici-	Stated indi-	Stated	En-	
pant	cation error	uncertainty	value	
	[%]	[%]		
P1	-0,18	0,04	-6,30	
Р3	0,14	0,05	0,05	
P4	0,16	0,30	0,08	
P5	0,11	0,15	-0,18	
R	0,137	0,031		

Table 2. Reported error at 2 mm



Ρ4

Ρ5

R

Comment: In the following extension points with P1-results judged as outliers the spread between the remaining sample leads to lower reference uncertainties. However, they are not considerably lower than those of two of the participants.

Table 3. Reported error at 3 mm

Partici-	Stated indi-	Stated	En-
pant	cation error	uncertainty	value
	[%]	[%]	
P1	-0,18	0,04	-6,44
P3	0,14	0,03	0,00
P4	0,17	0,30	0,09
P5	0,11	0,15	-0,18
R	0,140	0,032	

Table 4. Reported error at 4 mm

Partici-	Stated indi-	Stated	En-
pant	cation error	uncertainty	value
	[%]	[%]	
P1	-0,19	0,04	-6,38
P3	0,14	0,05	-0,08
P4	0,18	0,30	0,11
P5	0,12	0,15	-0,17
R	0,145	0,034	

Table 6. Reported error at 5 mm

Partici-	Stated indi-	Stated	En-
pant	cation error	uncertainty	value
	[%]	[%]	
P1	-0,19	0,04	-6,69
Р3	0,15	0,04	0,02
P4	0,18	0,30	0,10
P5	0,12	0,15	-0,19
R	0,153	0,034	



Ρ1

Ρ3



Diagram 5.







	1		
Partici-	Stated indi-	Stated	En-
pant	cation error	uncertainty	value
	[%]	[%]	
P1	-0,26	0,05	-7,92
P3	0,21	0,03	-0,02
P4	0,25	0,30	0,12
P5	0,18	0,15	-0,21
R	0,216	0,040	

Table 7. Reported error at 10 mm

Table 9. Reported error at 12,5 mm

Partici-	Stated indi-	Stated	En-
pant	cation error	uncertainty	value
	[%]	[%]	
P1	-0,33	0,04	-9,94
P3	0,27	0,03	-0,06
P4	0,32	0,30	0,13
P5	0,24	0,15	-0,23
R	0,278	0,043	









Corrections/changes after participant comments to the draft report

After publishing the draft participant P1 discovered that the Extensioneter data were inserted in the wrong column of the used application for documentation, which led to a negative sign of the indication error. In this stage of the comparison process this cannot be changed. However, had this error been avoided or discovered and reported before publishing the draft report it could have been corrected in the final report. A considerably better consensus in all points had followed. Changed reference values would have influenced all En-values. Thus of 21 extensioneter En-values 7 would have been lower and 14 slightly higher with a maximum of En = 0,33. The P1 values would have improved from large negative values to a maximum of En = 0,70.

Due to the low uncertainty of participant P3 compared to the reference uncertainty the En values for P3 are too optimistic (U_{ref} should be 1/3 of U(P1) or lower) but still far below critical.

Certificates

-- not a part of the intercomparison

Extensioneter results are documented in one case with the mean of 3 measurements. Another participant presented 2 result tables without a calculated mean (as described in ISO 9513:2012).

Final conclusions

In this inter comparison some of the participants could demonstrate a capacity to calibrate and give relevant values in relationship to their uncertainties.

As a result of this intercomparison the following can be pointed out:

Results on extensioneter is that 7 En-values of 28 are higher than 1. But they all are due to an undetected protocol error. The participants shall evaluate their results according to the requirement in EN ISO 17025:2017 point 7.7.3 in relation to the En-values and their CMC values.

<u>Acknowledgement</u>

Kungliga Tekniska Högskolan,KTH made this intercomparison possible as their machines could be used as the machine in the intercomparison.

A special thanks to Martin Öberg that supported all laboratories install equipment to run the machines and support in arranging he equipment in a safe way.

We gratefully thank the member of the advisory board and expert in pressure calibrations Aykurt Altintas, Denmark as well as the main evaluator of the results Peter Lau (Aykurt did not participate in evaluation of the result as Force was one of the participants).

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Annex

References:

- ISO/IEC 17043:2010 Conformity assessment General requirements for proficiency testing
- ISO/IEC 17025:2017 General requirements for the competence of testing and calibration laboratories
- ISO 13528 Statistical methods for use in proficiency testing by interlaboratory comparison.
- Evaluation of measurement data Guide to the expression of uncertainty in measurement, GUM (JCGM 100:2008)

- EA-4/02 M:2013 Evaluation of Uncertainty of Measurement in Calibration
- International Vocabulary of Metrology Basic and General Concepts and Associated Terms (VIM)
- ISO 7500-1:2018 calibration of force machines
- ISO 9513:2012 calibration of extensometers