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INTERLABORATORY COMPARISON IN THE RANGE FROM 0 TO 0,1 MPa FOR CROATIAN ACCREDITED LABORATORIES

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Abstract: This paper describes procedure and performance of the interlaboratory comparison (ILC) of pressure gauges calibration results in the range from 0 to 1 bar. ILC was organised by Croatian national pressure laboratory between four accredited Croatian pressure calibration laboratories. Transfer standard was pressure transducer with digital calibrator. Objective of this comparison was to assess calibration performance of participant laboratories compared to reference laboratory. Deviations for all measurement points will be listed in this paper. Assessment of the measurment results will be given by calculating E_n values.

Key words: interlaboratory comparison, pressure, calibration, measurements, measurement uncertainty

1. INTRODUCTION

Procedure of interlaboratory comparison (ILC) of pressure gauges calibration results in the range from 0 to 1 bar that was conducted by The Laboratory for process measurement (LPM) and four participant laboratories is described in this paper. Purpose of this ILC is to determine precision of measurement methods and to assess competence and capability of reference and participant laboratories to perform given measurement method. The standard for calibration of pressure gauges was provided by LPM.

LPM at Faculty of mechanical engineering and naval architecture (FSB - FAMENA) is developing and maintaining national pressure, temperature and humidity standards, and is part of Croatian Metrology Institute (HMI). It is also accredited by PTB (Physikalisch-Technische Bundesanstalt – Physical-Technical Federal Berau) with a DKD (Deutscher Kalibrierdienst - German Calibration Service) accreditation in accordance with EN ISO/IEC 17025 [1].

Other participant laboratories in this ILC were: Petrokemija d.d., MARUS–ATM d.o.o., Metron Instruments d.o.o., BMB Laboratorij Brcković d.o.o., all accredited by Croatian Accreditation Agency (HAA). Participant laboratories and their pressure standards are shown in Table 1.

Table 1: Reference standards used by participant laboratories

	Participating Laboratory	**Pressure standard 0 to 1 bar**
1.	Petrokemija d.d.	Pressure balance
2.	MARUS-ATM d.o.o.	Pressure calibrator
3.	Metron Instruments d.o.o.	Pressure transducer
4.	BMB laboratorij Brcković d.o.o.	Pressure balance

2. PROCEDURE

2.1. Object of Calibration and Calibration Procedure

Object of calibration was pressure transducer with digital calibrator Druck DPI 615 with resolution of 0,001 bar. It was circulated in four laboratories from May till September 2012, transported by car. All measurements and calibrations were conducted in accordance with DKD-R 6-1 guidelines [2]. Reference laboratory decided to perform type B of calibration sequence.
Every laboratory has made two increasing, and one decreasing measurement series in previously agreed eleven measurement points (0, 100, 200, 300, 400, 500, 600, 700, 800, 900, 1000 mbar). Load change and waiting time was 30 seconds. Aimed measurement uncertainty is between 0,1 and 0,6 percent of measurement span.
The calibration was performed according to type B procedure as follows:

1. Reading is made after opening the reference side of the pressure transducer to the atmosphere.
2. Ambient pressure and ambient temperature are recorded.
3. Two preloadings are made (highest value of pressure is applied for 30 seconds two times) with waiting time of 30 seconds between them.
4. After preloading, and after steady state conditions are reached, indication of the calibration item is set to zero, and zero point value is recorded.
5. Increasing measurement series is preformed. Pressures of 100 to 1000 mbar are produced with 30 seconds waiting time between measurement points. All pressure values in all measurement points are read and recorded. Before

proceeding to down measurement series, waiting time at upper limit of measurement range is two minutes.

6. Down measurement series is performed. Procedure is same as for up series, but in reverse order, pressures are produced from 1000 to 100 mbar.
7. Final up measurement series is preformed, same as in step 5.
8. Pressure is led off. Reading of zero point is made after 30 seconds. Reading of ambient pressure and temperature is repeated.
9. Participant laboratories are required to record their results in enclosed data sheets and to issue their standard calibration certificate.

Type of ILC procedure was circling, which means that measurements started in the reference laboratory (FSB - LPM), they were continued throughout participant laboratories, and comparison ended with last measurement in reference labratory.

2.2. Measurement uncertainty estimation

The measurement uncertainty of laboratory demonstrates the capability of laboratory to perform accurate measurements, and is equally important as measurement itself. Standard and expanded measurement uncertainties are calculated in accordance with protocol guidelines [2] and [3].
Combined standard uncertainty, *u*, for each point of measurement series was calculated as follows:

$$u(y) = \sqrt{u(stand)^2 + u(res)^2 + u(b')^2 + u(f_0)^2 + u(h)^2} \qquad (1)$$

Where:

u(y)	is combined standard uncertainty
u(stand)	is uncertainty of the standard
u(res)	is resolution uncertainty caused by pressure transducer (DUT)
u(b')	is uncertainty caused by repeatability of pressure transducer (DUT)
$u(f_0)$	is zero point uncertainty
u(h)	is uncertainty caused by hysteresis of pressure transducer (DUT)

Expanded uncertainty, *U*, is calculated as follows:

$$U(y) = k \cdot u(y) \quad (2)$$

Where:

U(y)	is expanded standard uncertainty
k	is coverage factor, and its value is 2
u(y)	is combined standard uncertainty

3. RESULTS

3.1. Deviations for all laboratories

In this section results from all laboratories will be displayed, namely deviations in calibrated range for all laboratories in Figure 1, and deviations with stated measurment uncertainties for each pressure point in Figures 2 and 3. Reference laboratory is depicted as LPM, and participant laboratories are numbered from 1 till 4, without showing individual laboratory name.

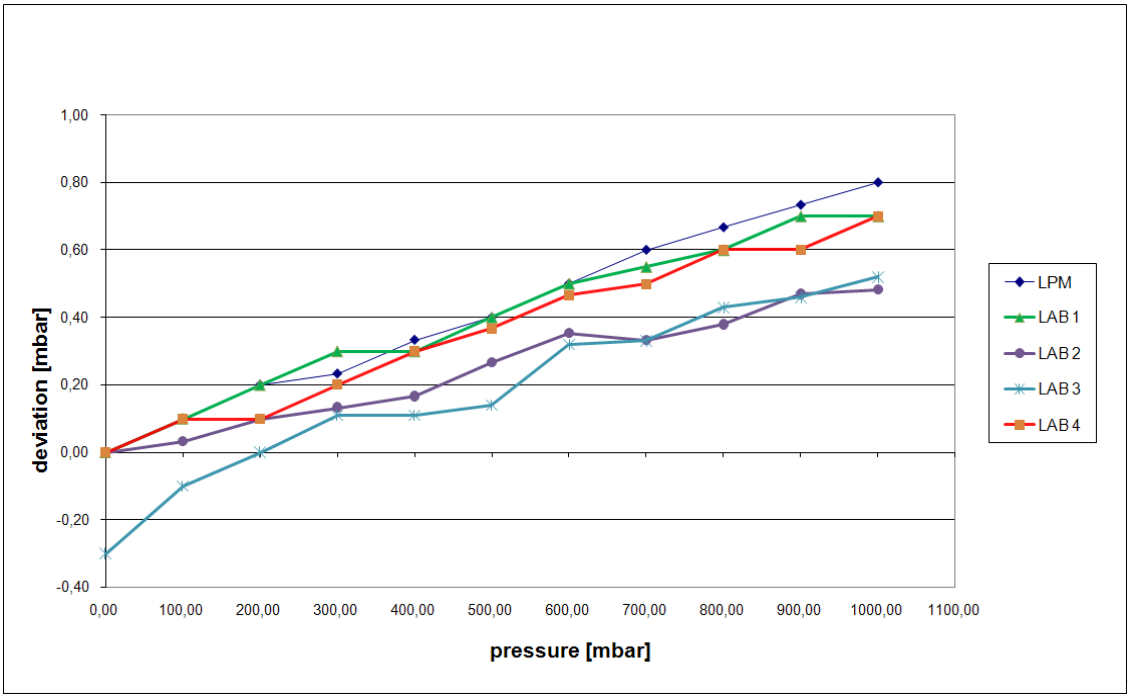


Figure 1: Deviations in calibrated range for all laboratories

Deviations with stated measurement uncertainties for each pressure point are shown individually:

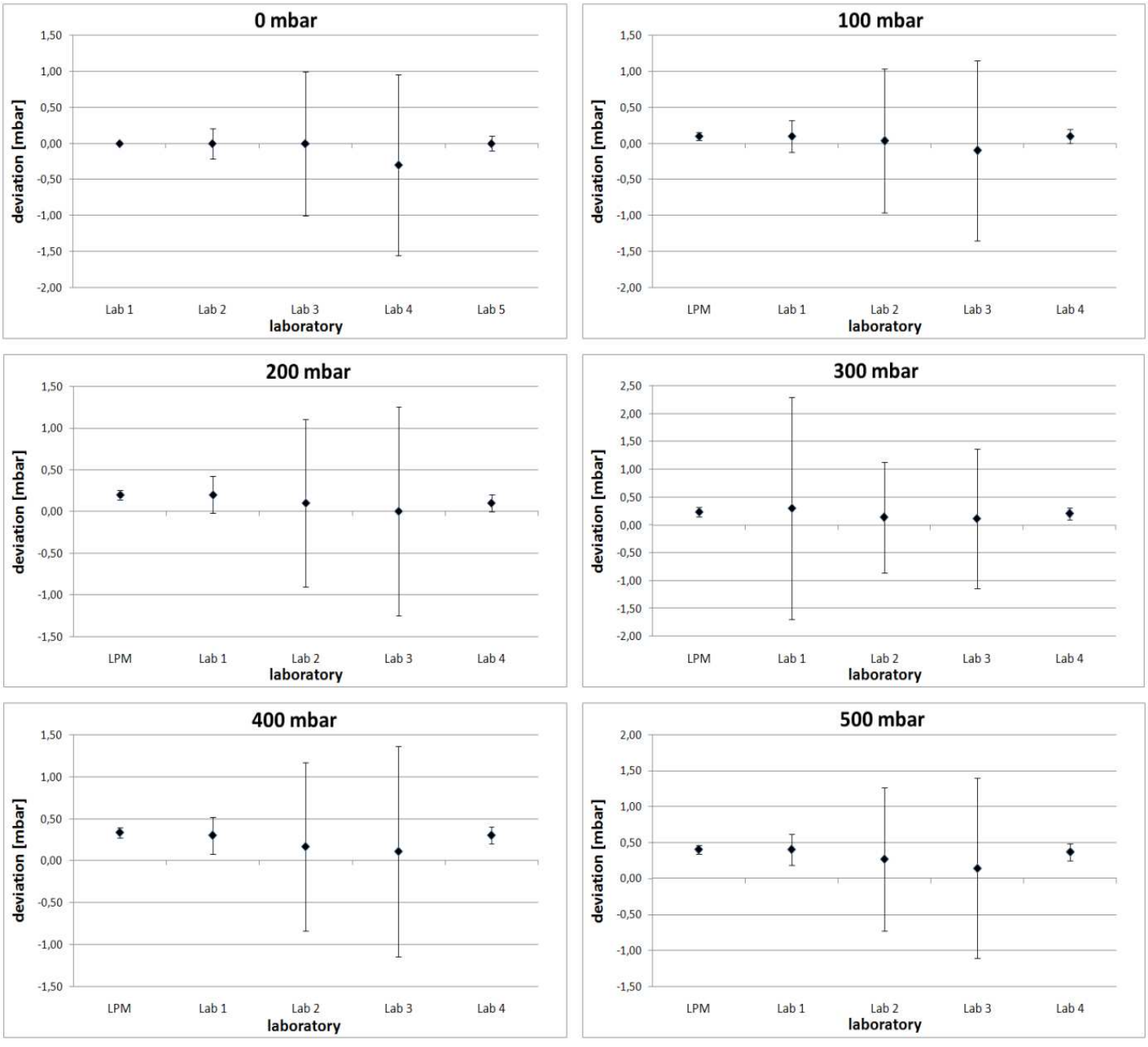


Figure 2: Deviations for all participating laboratories at 0 - 500 mbar

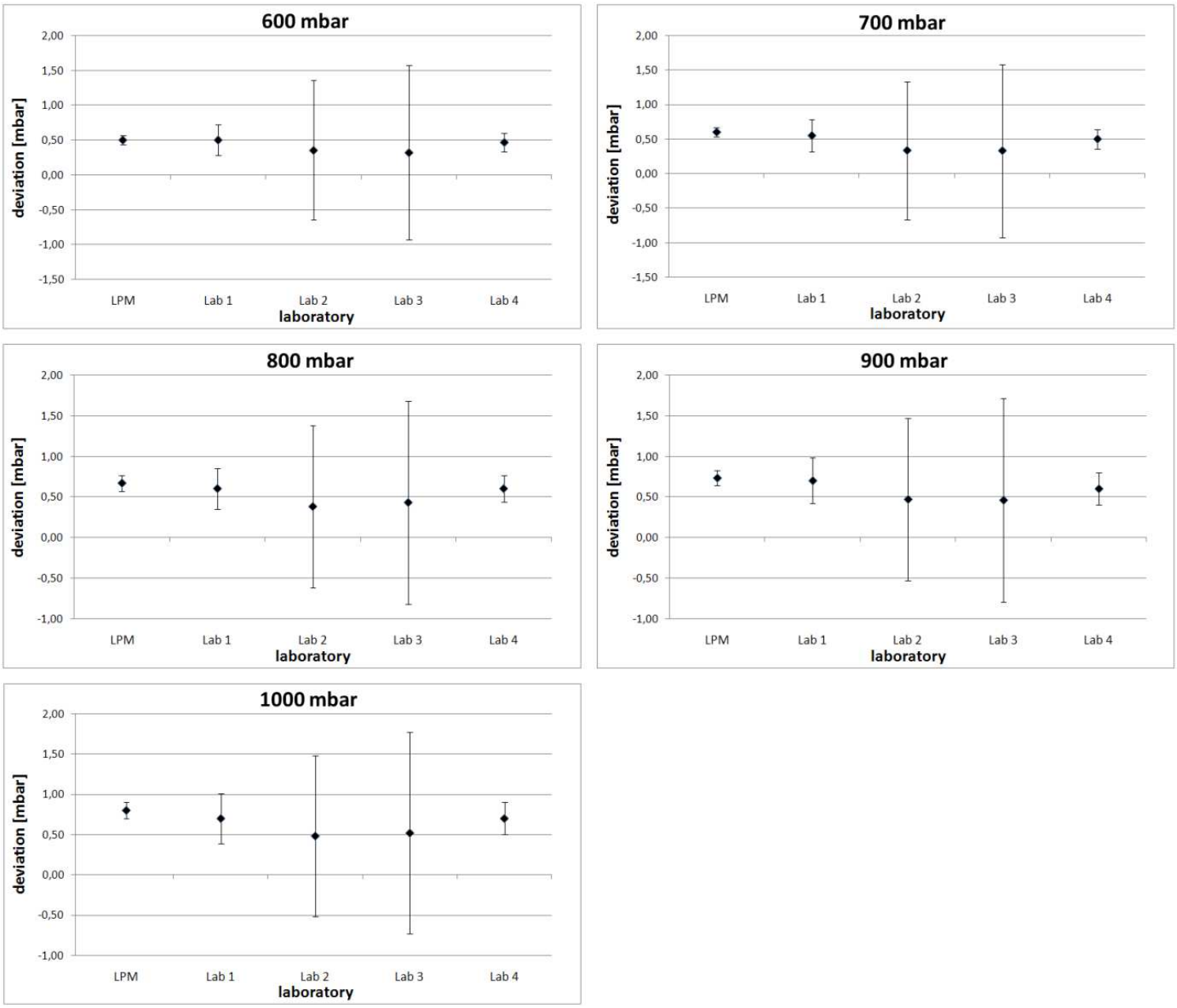
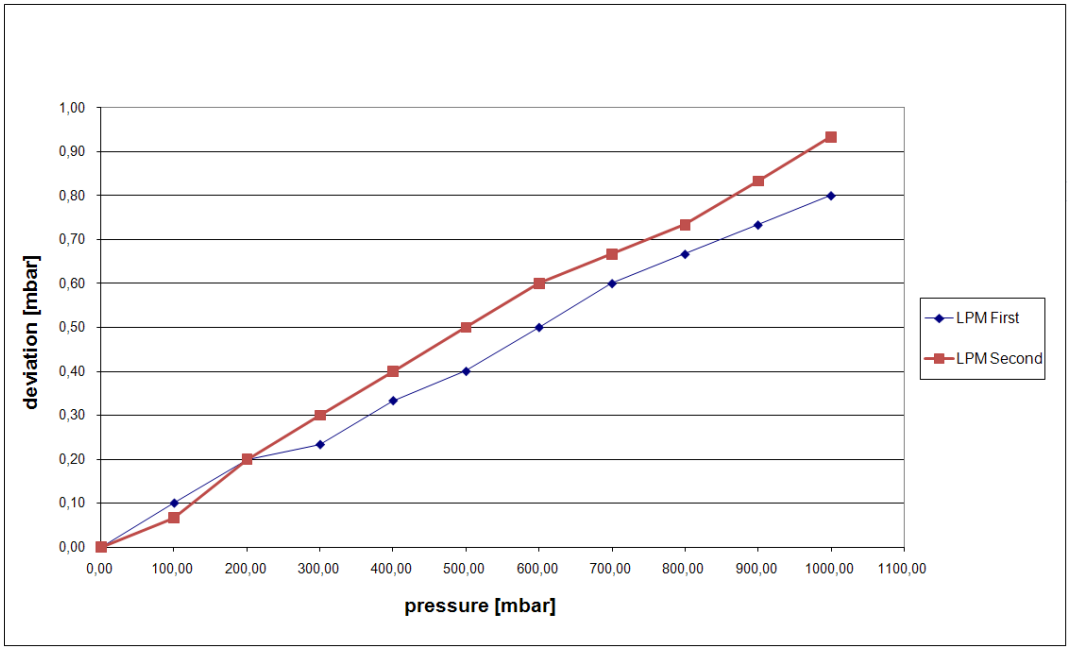


Figure 3: Deviations for all participating laboratories at 600 - 1000 mbar

3.2. Calibration in reference laboratory

ILC started with a calibration in reference labratory, and it also ended with calibration in reference laboratory. Purpose of the second calibration at the end of the ILC is to show the stability of the object of calibration. As seen in Figure 4, maximum deviation in calibrated range before and after comparison is 0,13 mbar. Second calibration was used as a reference.



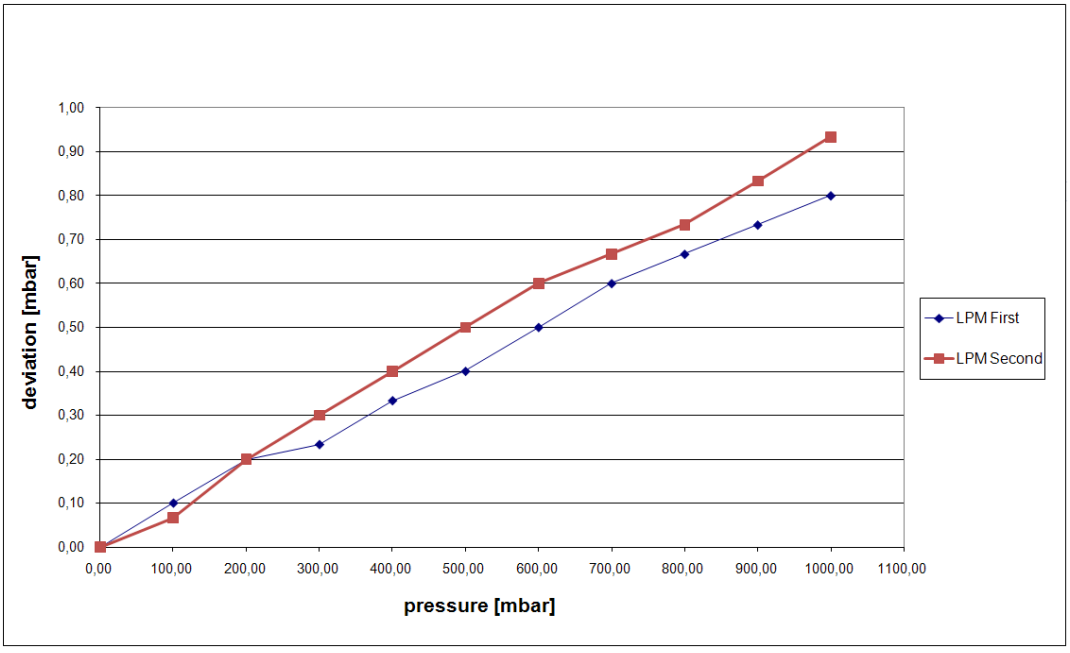


Figure 4: Deviations in calibrated range before and after comparison

In order to assess participant laboratories performance, E_n scores [4] were calculated. E_n scores provide a measure of how closely a reported laboratory result agrees with the assigned value, taking account of uncertainties in both the result and assigned value. Where a laboratory does not report an uncertainty estimate, an uncertainty of zero is used to calculate the En score. The E_n-score is an objective measure of whether or not an individual result is consistent with the assigned value. E_n-scores do not require the setting of a target standard deviation, and they are calculated as follows:

$$E_n = \frac{x - X}{\sqrt{U_{lab}^2 + U_{ref}^2}} \qquad (3)$$

Where:

E_n	is E_n score
x	is individual laboratory result
U_{lab}	is expanded uncertainty of the individual laboratory result
X	is assigned value
U_{ref}	is expanded uncertainty of the assigned value

E_n scores are interpreted as follows:

- $|E_n| \leq 1$ satisfactory
- $|E_n| > 1$ questionable

Resulting E_n scores of participant laboratories are given in Table 2.

Table 2: E_n scores of participating laboratories compared with LPM

Pressure	$\|E_n\|$ Lab 1	$\|E_n\|$ Lab 2	$\|E_n\|$ Lab 3	$\|E_n\|$ Lab 4
[mbar]				
0	0,00	0,00	0,24	0,00
100	0,00	0,07	0,16	0,00
200	0,00	0,10	0,16	0,98
300	0,32	0,10	0,11	0,28
400	0,14	0,17	0,18	0,27
500	0,00	0,13	0,21	0,26
600	0,00	0,15	0,14	0,23
700	0,21	0,27	0,22	0,64
800	0,24	0,28	0,19	0,34
900	0,11	0,26	0,22	0,63
1000	0,31	0,32	0,22	0,45

Detailed comparisons of each participant laboratory are shown in Tables 4 to 7. In mentioned tables one can compare expanded uncertainties of the individual laboratory result to the expanded uncertainty of the assigned value for each measurement point. Deviations are compared by substracting deviations of reference laboratory from deviations of participant laboratories.

Table 3: Deviations of laboratory 1 compared with LPM

Pressure	Standard (LPM)	Standard (Lab 1)	Deviation M_{LAB1}	Deviation M_{LPM}	M_{LAB1}-M_{LPM}	U_{LAB1}	U_{LPM}	$\|E_n\|$
[mbar]			[mbar]	[mbar]	[mbar]	[mbar]	[mbar]	
0			0,00	0,00	0,00	0,21	0,0020	0
100,00			0,10	0,10	0,00	0,22	0,0102	0
200,00			0,20	0,20	0,00	0,22	0,0200	0
300,00			0,30	0,23	0,07	0,2	0,0651	0,32
400,00	Pressure Balance	Pressure Balance	0,30	0,33	-0,03	0,22	0,0702	0,14
500,00			0,40	0,40	0,00	0,22	0,0500	0
600,00			0,50	0,50	0,00	0,22	0,0600	0
700,00			0,55	0,60	-0,05	0,23	0,0700	0,21
800,00			0,60	0,67	-0,07	0,25	0,1143	0,24
900,00			0,70	0,73	-0,03	0,28	0,1069	0,11
1000,00			0,70	0,80	-0,10	0,31	0,1000	0,31

Deviations with stated measurement uncertainties for all pressure points of participant laboratories are compared to deviations of reference laboratory in Figures 14 to 17.

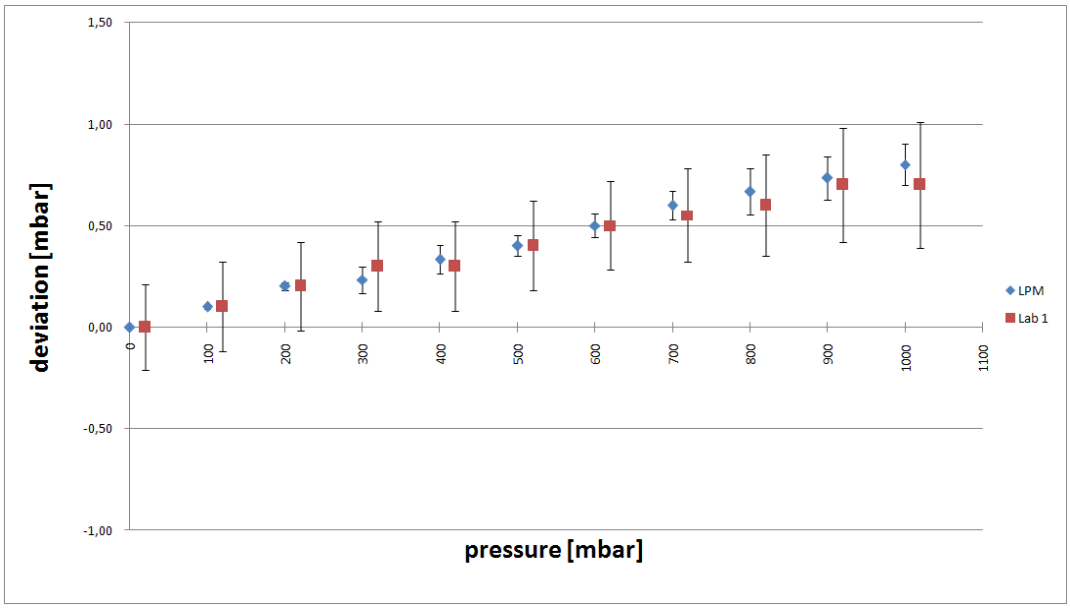


Figure 5: Deviations of laboratory 1 compared with LPM

Table 4: Deviations of laboratory 2 compared with LPM

Pressure	Standard (LPM)	Standard (Lab 2)	Deviation M_{LAB2}	Deviation M_{LPM}	M_{LAB2}-M_{LPM}	U_{LAB2}	U_{LPM}	$\lvert E_n \rvert$
[mbar]			[mbar]	[mbar]	[mbar]	[mbar]	[mbar]	
0	Pressure Balance	Pressure Calibrator	0,00	0,00	0,00	1,00	0,0020	0
100,00			0,03	0,10	-0,07	1,00	0,0102	0,07
200,00			0,10	0,20	-0,10	1,00	0,0200	0,1
300,00			0,13	0,23	-0,10	1,00	0,0651	0,1
400,00			0,17	0,33	-0,17	1,00	0,0702	0,17
500,00			0,27	0,40	-0,13	1,00	0,0500	0,13
600,00			0,35	0,50	-0,15	1,00	0,0600	0,15
700,00			0,33	0,60	-0,27	1,00	0,0700	0,27
800,00			0,38	0,67	-0,29	1,00	0,1143	0,28
900,00			0,47	0,73	-0,26	1,00	0,1069	0,26
1000,00			0,48	0,80	-0,32	1,00	0,1000	0,32

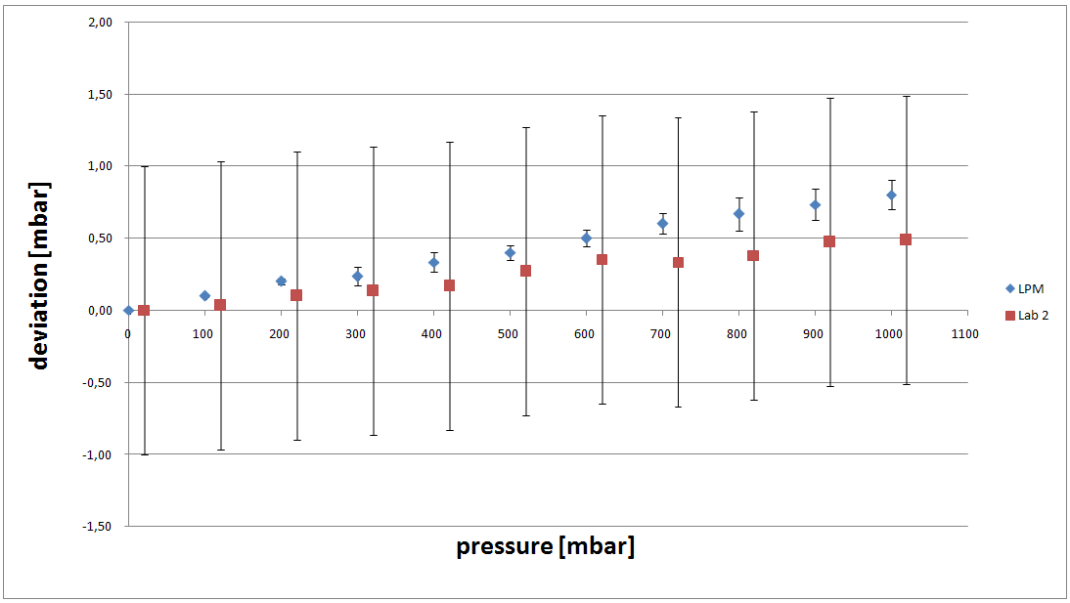


Figure 6: Deviations of laboratory 2 compared with LPM

Table 5: Deviations of laboratory 3 compared with LPM

Pressure	Standard (LPM)	Standard (Lab 3)	Deviation M_{LAB3}	Deviation M_{LPM}	M_{LAB3}-M_{LPM}	U_{LAB3}	U_{LPM}	$\lvert E_n \rvert$
[mbar]			[mbar]	[mbar]	[mbar]	[mbar]	[mbar]	
0	Pressure Balance	Pressure Transducer	-0,30	0,00	-0,30	1,25	0,0020	0,24
100,00			-0,10	0,10	-0,20	1,25	0,0102	0,16
200,00			0,00	0,20	-0,20	1,25	0,0200	0,16
300,00			0,11	0,23	-0,12	1,25	0,0651	0,11
400,00			0,11	0,33	-0,22	1,25	0,0702	0,18
500,00			0,14	0,40	-0,26	1,25	0,0500	0,21
600,00			0,32	0,50	-0,18	1,25	0,0600	0,14
700,00			0,33	0,60	-0,27	1,25	0,0700	0,22
800,00			0,43	0,67	-0,24	1,25	0,1143	0,19
900,00			0,46	0,73	-0,27	1,25	0,1069	0,22
1000,00			0,52	0,80	-0,28	1,25	0,1000	0,22

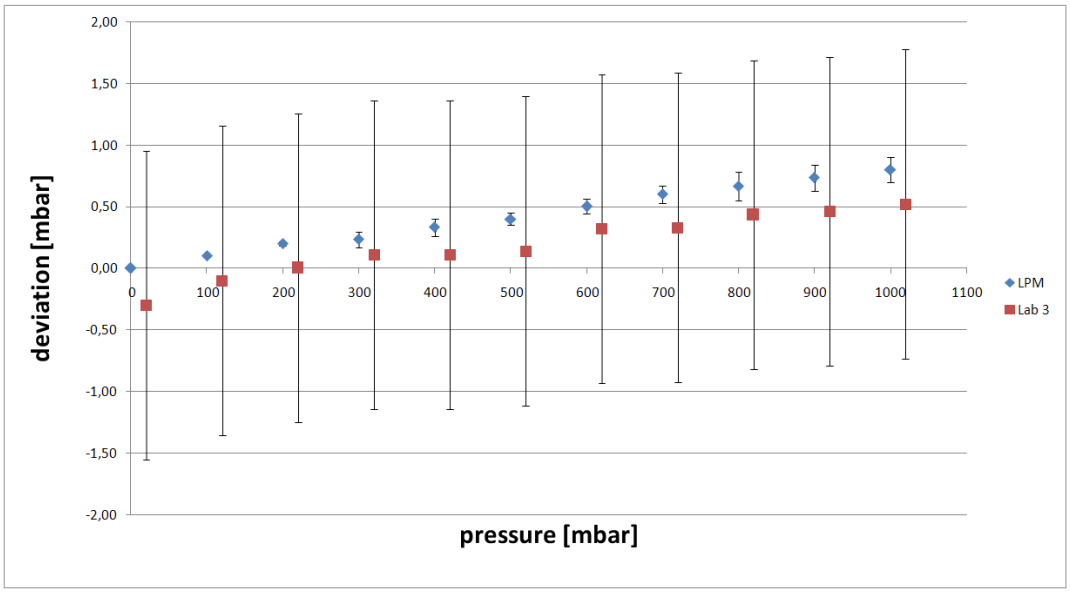


Figure 7: Deviations of laboratory 3 compared with LPM

Table 6: Deviations of laboratory 4 compared with LPM

Pressure	Standard (LPM)	Standard (Lab 4)	Deviation M_{LAB4}	Deviation M_{LPM}	M_{LAB4}-M_{LPM}	U_{LAB4}	U_{LPM}	$\lvert E_n \rvert$
[mbar]			[mbar]	[mbar]	[mbar]	[mbar]	[mbar]	
0	Pressure Balance	Pressure Balance	0,00	0,00	0,00	0,10	0,0020	0
100,00			0,10	0,10	0,00	0,10	0,0102	0
200,00			0,10	0,20	-0,10	0,10	0,0200	0,98
300,00			0,20	0,23	-0,03	0,10	0,0651	0,28
400,00			0,30	0,33	-0,03	0,10	0,0702	0,27
500,00			0,37	0,40	-0,03	0,12	0,0500	0,26
600,00			0,47	0,50	-0,03	0,13	0,0600	0,23
700,00			0,50	0,60	-0,10	0,14	0,0700	0,64
800,00			0,60	0,67	-0,07	0,16	0,1143	0,34
900,00			0,60	0,73	-0,13	0,18	0,1069	0,64
1000,00			0,70	0,80	-0,10	0,20	0,1000	0,45

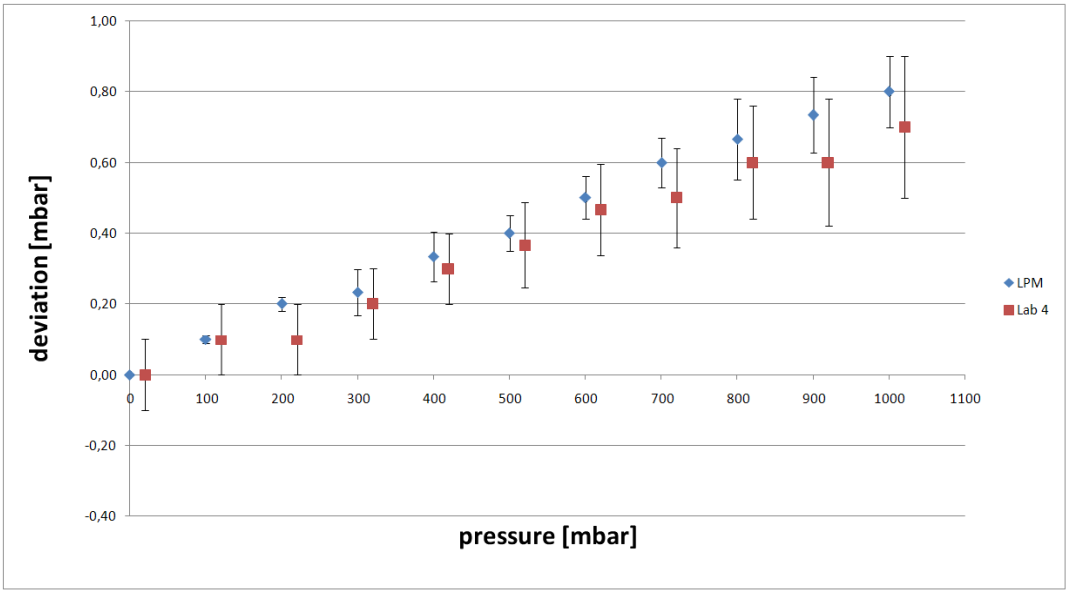


Figure 8: Deviations of laboratory 4 compared with LPM

4. CONCLUSION

ILC procedure presented in this paper was conducted between four Croatian accredited laboratories in accordance with DKD-R 6-1 guidelines. All laboratories have performed the necessary measurements and submitted measurement results. Comparison began in the reference laboratory on 07.05.2012., and ended in the same laboratory on 05.09.2012. The analysis of measurement results from all laboratories has shown that all the results are satisfactory, because the E_n scores for all participant laboratories are smaller than one.

In the case that one or more participant laboratories would have had one or more of their E_n scores greater than 1, it is important to analyze the possible causes for such scores and to initiate corrective action such as calibrating existing equipment, checking the method with control sample and to participate in repeated interlaboratory comparison.

REFERENCES

[1] International standard ISO/IEC 17025 –General requirements for the competence of testing and calibration laboratories, 1999.
[2] DKD-R-6-1, Guideline (Calibration of Pressure Gauges), Edition 01/2003

[3] EURAMET/cg-17/v.01 Guidelines on the Calibration of Electromechanical Manometers, July 2007

[4] International standard ISO/DIS 13528 - Statistical Methods for use in proficiency testing by interlaboratory comparisons.