Int. J. Metrol. Qual. Eng. **6**, 307 (2015)

DOI: 10.1051/ijmqe/2015021

Interlaboratory comparison in the pressure range from 0 to 2 MPa for accredited calibration laboratories

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Received: 4 September 2015 / Accepted: 6 September 2015

Abstract. This paper describes procedure and results of the interlaboratory comparison that was organised by Croatian national pressure laboratory (HMI/FSB-LPM) between eight accredited pressure calibration laboratories. Comparison was performed in the gauge pressure range from 0 to 2 MPa with pressure transducer as transfer standard and using gas as transmitting medium. Basic aim of this comparison was to improve measurement methods and the measurement uncertainties of each participating laboratory. Analysis of the measurement results are given by calculating the deviation, E_n, normalized with respect to the stated measurement uncertainties.

Keywords: Interlaboratory comparison, pressure, measurement uncertainty

1 Introduction

The interlaboratory comparison (ILC) program described in this paper is designed to help calibration laboratories to achieve confidence in pressure calibration results, validate reliable uncertainty levels and meet ISO 17025 requirements [1]. Participation in interlaboratory comparisons also provides laboratories an opportunity for independent assessment of their measurement performance.

Programme is developed in the laboratory for process measurement (LPM) at University of Zagreb, faculty for mechanical engineering and naval architecture (FAMENA), which is part of distributed National Metrology Institute in Croatia (Croatian Metrology Institute-HMI).

Calibration in the range from 0 to 2 MPa in gauge pressure range is of great importance for all pressure laboratories because it covers many technical applications and because this range of pressure is mostly used when pressure transmitting medium is gas. The objective of described comparison is to determine the relative agreement between gas pressure standards.

2 Participants, transfer standard and measurement procedure

2.1 Pilot laboratory and participants

The reference values for calibration of pressure gauges, described in this paper were provided by the LPM. As pilot laboratory, LPM was responsible for preparing the measurement instructions, controlling the stability of the transfer standard, calculating the results and preparing the final report.

The activities of the LPM are in the area of measurement of thermal and process values, developing and maintaining national pressure, temperature and humidity standards with published CMC's (Calibration Measurement Capabilities) in the BIPM key comparison database. Laboratory is accredited according to the EN ISO/IEC 17025 for temperature pressure and humidity calibrations by the DAkkS (Deutsche Akkreditierungsstelle – registration number DKD-K-35601) from 2002 to 2012 and from 2013 by Croatian Accreditation Agency.

Laboratory participated in many EURAMET (European Association of National Metrology Institutes) projects and intercomparisons and start to develop national interlaboratory comparison schemes from 2010 [2].

Other participating laboratories and their pressure standards are shown in Table 1.

Table 1. Reference standards used by participating laboratories.

	Participating Laboratory (Town)	Pressure standard 0 up to 20 bar
1.	MARUS ATM d.o.o. (Zagreb)	Gas Pressure Balance
2.	Metron Instruments (Zagreb)	Pressure transducer
3.	Ravnoteža (Zagreb)	Pressure transducer
4.	Petrokemija d.d. (Kutina)	Gas Pressure Balance
5.	Inspekt Metrolab (Zagreb)	Pressure transducer
6.	BMB (Zagreb)	Gas Pressure Balance
7.	CEI-IETA (Zagreb)	Pressure transducer
8.	STSI (Zagreb)	Gas Pressure Balance

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2.2 Transfer standard and calibration procedure

Digital pressure transducer (DRUCK DPI 615) as transfer standard of high accuracy was provided by LPM. Measurement range of transfer standard was from 0 up to 20 bar with 0.001 bar resolution. Transfer standard was circulated in eight laboratories from January till June 2010, transported by car.

All participants agreed with following protocol:

- The pressure medium is nitrogen.
- More than one reference standard can be used.
- After setting up, there must be enough time for the pressure transducer to reach ambient temperature.
- The surface of the upper level of the adapters considered as reference level for pressure measurements.
- Readings of the digital measuring unit must be in bar.

The calibration was performed in three measurement series according to, type B procedure [3], as follows:

1. After having opened the reference side of the pressure transducer to atmosphere, the reading is made.
2. The calibrating unit of the digital measuring unit is used to set the reading to zero. The ambient pressure and the ambient temperature shall then be read and recorded.
3. A pressure corresponding to maximum pressure of the device under test, but not more than 25 bar, is summoned up within two minutes. Then, the pressure must be let out to $p_e = 0$ bar, this is repeated twice after two minutes. The zero-point value must be read and recorded.
4. Pressures of 2, 4, 6, 8, 10, 14, 16 and 20 bar must be produced, but not more than 25 bar.
5. Step 4 is repeated in reverse order.
6. Pressure is led off. Reading is made after 5 min. Reading and recording of the ambient pressure and ambient temperature is done. Read the pressure after 10 min for the second time.
7. The participating laboratories are requested to write down their results in the enclosed data sheets and to additionally issue one of their standard calibration certificates.

Depending on type of pressure standard, corrections of nominal pressure were applied. Calculations of effective pressure were done by each laboratory according to their own procedure.

3 Measurement uncertainty estimation

The uncertainty of laboratory measurement is equally as important as the measurement itself and demonstrates the capability of laboratory to make accurate measurements. For the uncertainty analysis in this intercomparison it was agreed that participants can follow DKD [3] or EURAMET [4] recommendations for calibration of pressure gauges, both based on ISO-GUM method [5].

Evaluation model for one calibration point is described in continuous. Following exactly the references [3, 4] recommendations, sources of uncertainty are divided in three groups: uncertainties of the pressure standard (usually standard piston/cylinder assembly or standard pressure transducer), uncertainties of the calibration method (including hydrostatic deviations from reference level and current measurements) and uncertainties associated with device under test, DUT (resolution, repeatability and hysteresis). Since we have only three readings on each pressure point, Type A uncertainty can not be calculated from statistics but repeatability and hysteresis, which can be dominant influence quantities, are taken into account as Type B uncertainties. Repeatability was calculated as difference between two ascending measurements and hysteresis as difference between ascending and descending readings, taking into account rectangle probability distribution as described in [3]. Uncertainty of pressure standard was estimated from calibration report as Type B taking into account normal probability distribution. All uncertainty contributions can be expressed in the unit of pressure (bar) and there was no need for determination of sensitivity coefficients.

Combined standard uncertainty, u, for each calibration point was calculated after corrections as:

$$u^2 = u_{PC}^2 + u_{Corr}^2 + u_H^2 + u_r^2 + u_{f_0}^2 + u_b^2 + u_h^2, \qquad (1)$$

where:

- u_{PC} is P/C unit uncertainty estimated from manufacturer's calibration report,
- u_{Corr} is P/C unit uncertainty under conditions of calibration,
- u_H is uncertainty caused by difference in height,
- u_r is resolution uncertainty caused by DUT,
- u_{f_0} is uncertainty caused by zero deviation of pressure transducer (DUT),
- u_b is uncertainty caused by repeatability of pressure transducer (DUT),
- u_h is uncertainty caused by hysteresis of pressure transducer (DUT).

Expanded uncertainty, U, for each point was calculated as:

$$U = ku, \qquad (2)$$

where coverage factor $k = 2$.

4 Results

Calibration results for 9 measurement points including deviations from standard pressure and measurement uncertainties are reported to LPM and analysed to ensure uniform treatment for all participants. Deviations shown in Figure 1 pertains to all participating laboratories, depicted as LAB 1, LAB 2, LAB 3. . . . without showing the names of the laboratory.

Deviations with stated measurement uncertainties for each pressure point are shown individually (Figs. 2–10).

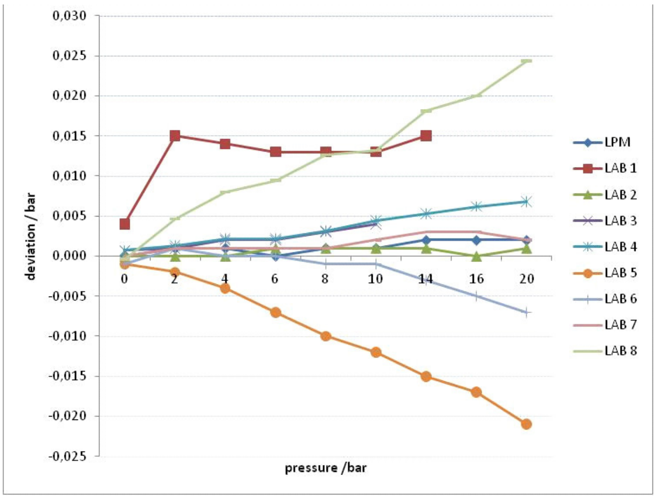

Fig. 1. Deviations in calibrated range for all laboratories.

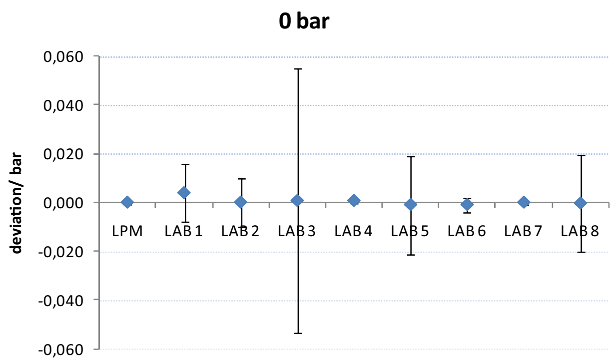

Fig. 2. Deviations for all participating laboratories respective to associated expanded uncertainty at 0 bar.

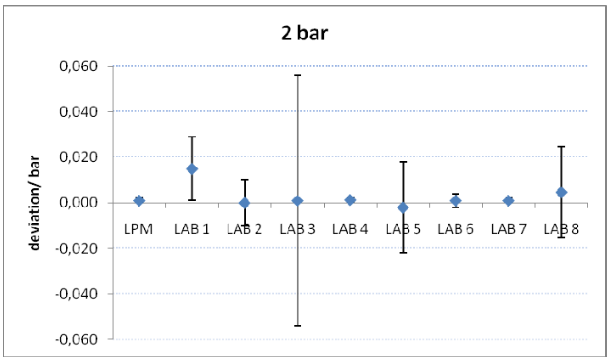

Fig. 3. Deviations for all participating laboratories respective to associated expanded uncertainty at 2 bar.

4.1 Stability of the standard

Stability of the standard in this case was assessed by re-calibration of transfer standard. LPM has calibrated transfer standard before comparison and six months later after all measurements were performed. No intermediate calibrations were performed. Stability of the standard was within 1 mbar (Fig. 11).

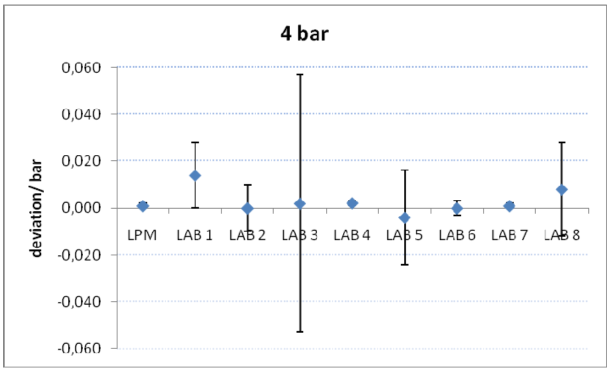

Fig. 4. Deviations for all participating laboratories respective to associated expanded uncertainty at 4 bar.

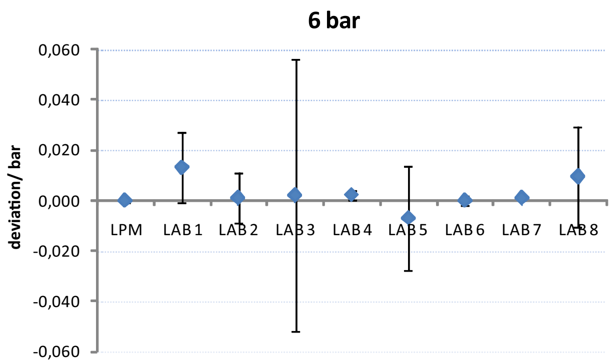

Fig. 5. Deviations for all participating laboratories respective to associated expanded uncertainty at 6 bar.

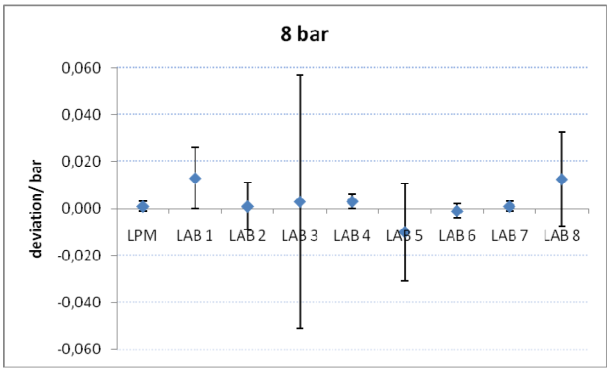

Fig. 6. Deviations for all participating laboratories respective to associated expanded uncertainty at 8 bar.

4.2 Reference value and E_n numbers

The most widely accepted method of data analysis compares each laboratory's measured data and reported measurement uncertainties against established reference value. The result is measure of agreement between participants, E_n. When the resultant E_n for any measurement is between $+1$ and -1 no corrective action is usually required [6]. If the resultant E_n is greater than $+1$ or -1 this provides a signal that something might be wrong with a laboratory's calibration process, equipment or standards,

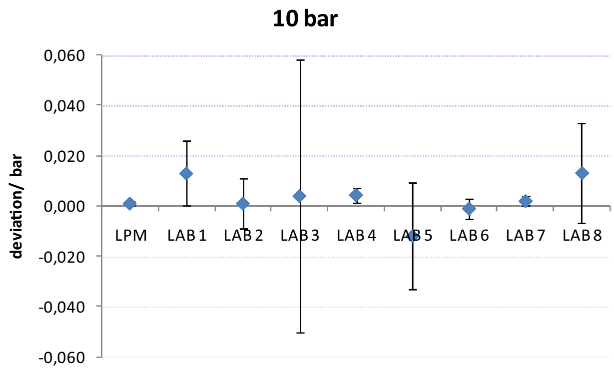

Fig. 7. Deviations for all participating laboratories respective to associated expanded uncertainty at 10 bar.

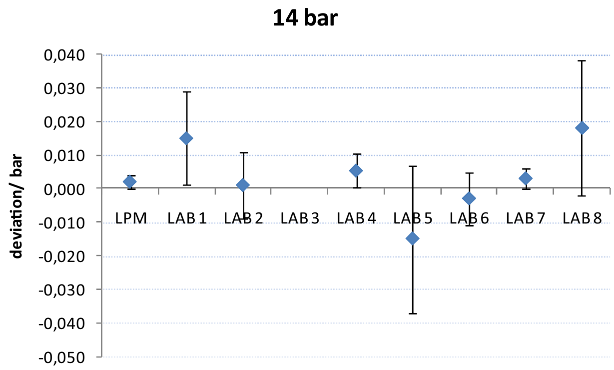

Fig. 8. Deviations for all participating laboratories respective to associated expanded uncertainty at 14 bar.

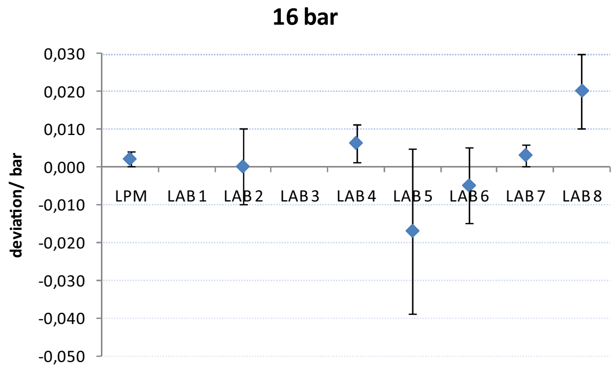

Fig. 9. Deviations for all participating laboratories respective to associated expanded uncertainty at 16 bar.

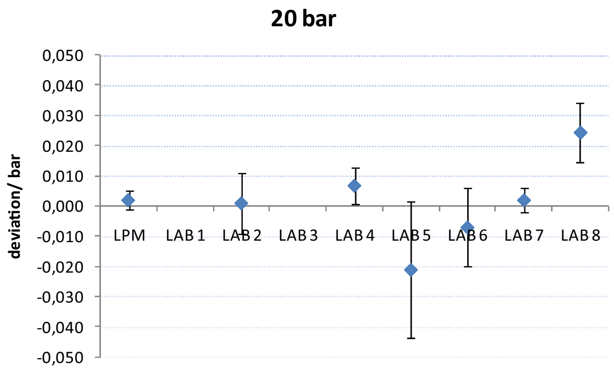

Fig. 10. Deviations for all participating laboratories respective to associated expanded uncertainty at 20 bar.

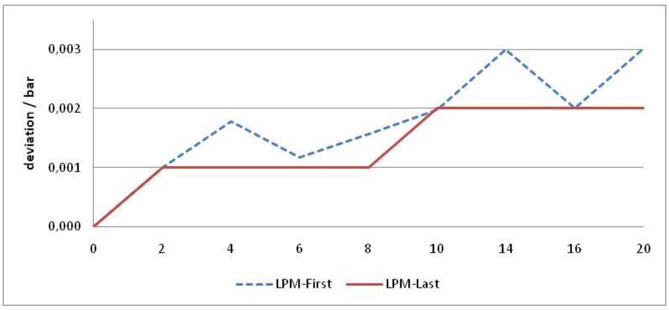

Fig. 11. Deviations in the calibrated range before and after comparison.

Table 2. E_n numbers of participating laboratories 1 to 4 compared with LPM.

Presssure bar	E_{nLAB1}	E_{nLAB2}	E_{nLAB3}	E_{nLAB4}
0	0.33	0.00	0.01	0.49
2	1.00	0.10	0.00	0.21
4	0.93	0.10	0.02	0.85
6	0.93	0.10	0.04	0.98
8	0.91	0.00	0.04	0.58
10	0.92	0.00	0.06	1.08
14	0.92	0.10		0.61
16		0.20		0.78
20		0.10		0.72

competence of personnel, stated uncertainties, etc.

$$E_n = \frac{|M_{LPM} - M_{lab}|}{\sqrt{(U_{lab}^2 + U_{LPM}^2)}}, \tag{3}$$

where M_{LPM} is measurement result of LPM, M_{lab} is measurement result assigned by participated laboratory, U_{LPM} is uncertainty of M_{LPM}. U_{lab} is the uncertainty of M_{lab} and should include an allowance for the performance of the measurement service. E_n is displayed using chart and graph for one real testing.

Resulting E_n values are given in Tables 2 and 3.

5 Conclusion

Eight Croatian accredited laboratories compared their calibration results and estimated measurement uncertainties using the same procedure. Equivalence respective to reference value is fully realized by 4 participants. From the results it can be seen that number of occurrence of $E_n \geq 1$ is 4 but in different pressure points. Those laboratories should analyse their measurements or increase calibration measurement capabilities (CMC values).

It is clear that participation in such programme has significant educational element. Basically every part of

Table 3. E_n numbers of participating laboratories 5 to 8 compared with LPM.

Presssure	E_{nLAB5}	E_{nLAB6}	E_{nLAB7}	E_{nLAB8}
bar				
0	0.05	0.32	0.00	0.02
2	0.15	0.00	0.00	0.18
4	0.25	0.32	0.00	0.35
6	0.34	0.00	0.67	0.47
8	0.53	0.55	0.00	0.58
10	0.62	0.49	0.45	0.61
14	0.78	0.61	0.28	0.80
16	0.86	0.69	0.28	0.90
20	1.02	0.67	0.00	1.11

Laboratory quality system comes into play during participation in a ILC.

Participating laboratories can use such intercomparison:

- For cross train of their personnel.
- To demonstrate technical competence.
- To identify potential areas for improvement.
- Data can be analysed to prevent incorrect results from being reported (ISO 17025: 5.9.2).

References

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