EVALUATION OF INTERLABORATORY COMPARISONS ON PRESSURE MEASUREMENT IN THE PHILIPPINES OF 6 YEARS INTERVAL

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Abstract:
This paper presents the evaluation of two intercomparisons on pressure measurement, six years apart using the same artefact. These intercomparisons aimed to provide local laboratories access to proficiency testing for their compliance with the ISO/IEC 17025 requirements. Improved awareness and commitment to quality of the participants and the enhanced competence of the pilot laboratory are a few of the enumerated factors instigating the increase in the number of participants as well as those obtaining satisfactory results. The PT schemes offered aim to sustain and continuously develop this service to support further progress in the calibration and measurement capabilities of local laboratories.

Keywords: Measurement; Intercomparison; Interlaboratory, Proficiency Testing; Pressure

1. INTRODUCTION

The National Metrology Laboratory (NML) of the Industrial Technology Development Institute (ITDI) under the Department of Science and Technology (DOST) conducted interlaboratory comparisons in the field of hydraulic pressure measurement among the local calibration laboratories in the Philippines. The provision of interlaboratory comparison, otherwise known as proficiency testing (PT), is a program to strengthen the NML relationship with the said laboratories in establishing scientific metrology in the country.

This PT program aims to: (a) determine the technical capabilities and performance of the laboratories; (b) assess the reliability of their measurement results and validate their calibration measurement capabilities; (c) disseminate a harmonized and validated calibration procedure; (d) demonstrate metrological equivalence to the NML and most importantly, (e) provide access to interlaboratory comparisons for their compliance to ISO/IEC 17025 [3] requirements. The Pressure Standards Section (PSS) of the NML acted as the program coordinator and reference laboratory, which is accredited under the terms of ISO/IEC 17025:2005 (2017 at present). The PSS is responsible for providing the artefact, the reference value and its measurement uncertainty, the monitoring of the program as a whole, and preparation of written reports for the two intercomparisons being compared, one was conducted in 2010 while the other is in 2016.

2. COMPARISON PROCESS

The two intercomparisons followed an almost similar process, differences are, however, emphasized in this paper specifically the contributing factors that affected the results of the two schemes. It is herein referred the first PT scheme as 2010 and the second PT scheme as 2016 throughout the discussion.

The interlaboratory comparison program is designed as a cycle where the NML as a reference or pilot laboratory calibrates the artefact at the beginning, middle, and end of the program.

One main difference between 2010 and 2016 is the conduct of a preparatory workshop before the start of the PT program. This workshop proved to be essential in getting to know each lab's capabilities, what standards they have and what procedure they follow in the calibration of pressure gauges. Also, in this workshop, an agreement between the participants and the NML was reached, fulfilling the objective to disseminate a harmonized and validated calibration procedure and the identification of limitations of the participants and the PT program in general.

2.1 Participants

Participants in the two PTs are local calibration laboratories with NML as the reference lab. In 2010, the five (5) participants were all private laboratories in Metro Manila while in 2016, the 16 participants were composed of private and government laboratories including some DOST regional metrology laboratories.

2.2 Artefact

The artefact used in 2010 and 2016 is a Bourdon-tube type pressure gauge. It is noted that in 2010, two artefacts of different ranges were measured by the participants but in this paper, comparisons will be based on only one artefact used on both PTs and the description is as follows:

Figure 1: The artefact

Table 1: Technical Specification of the Artefact

Manufacturer	Ashcroft
Serial Number / Identification	S2-W-006
Capacity	25 000 kPa
Graduation	100 kPa
Accuracy	0.25 %
Medium	Liquid

The artefact was subjected to initial and subsequent characterization before the PTs and maintained a regular interval of calibration and intermediate checks when not used as an artefact.

2.3 Calibration method

The participants were asked to calibrate the artefact through direct comparison to their standard. In 2010, each participant used the typical calibration procedure in their laboratory, this means their laboratory-developed method. Meanwhile, in 2016, after the earlier mentioned preparatory workshop, it was agreed among the participants to follow an international guideline, the DKD-R 6-1 Calibration of Pressure Gauges [4] which not only guided the calibration procedure but the computation of measurement uncertainty as well. The said guideline is used by the NML.

2.4 Measurement Scheme

The measurement scheme is chosen in order to monitor the metrological quality of the artefact throughout the whole PT process.

In 2010, the artefact was calibrated before and after a trip to a participant. It is hand-carried to and from each participating lab by its representative. In 2016 however, since there were more participants and some are outside Metro Manila, the artefact was calibrated by the NML before and after a group of participants, usually 2 to 3 labs, strategically chosen based on location so the sending back and forth to the NML of the artefact are made in the most efficient manner.

2.5 Report of the Participants

In 2010, the participants were only asked to submit the filled-out NML-provided measurement datasheet and the calibration certificate they usually issue to their customers. Other information such as uncertainty budget was only known when required by the NML. This practice was changed in 2016 wherein the participants should submit documents such as the measurement datasheets, a copy of the calibration certificate of their standard proving valid traceability, their usual calibration report, and the uncertainty budget. The transparency of the participants' data to NML led to an improved evaluation process of measurement results.

2.6. Reference Values

In both PTs, the reference values used in evaluating the normalized error *(En)* for each participant were based on the values nearest the participant's reported results, which is either before or after the calibration of the reference laboratory. In 2016, this result was given to participants in an Interim Report, showing only the specific participant's results compared to NML's results. This interim report was very useful for participants having to prove their competence to technical peers during their assessment while the intercomparison is not yet completed. It should be noted however that in the final report, the reference values reflected are the weighted average of all the measurement results of the NML.

3. MEASUREMENT RESULTS

The measurement results of participating laboratories are evaluated using the earlier mentioned normalized error or the *En* ratio [5], calculated using the equation:

$$E_n = \frac{x_{lab} - x_{ref}}{\sqrt{\left(U_{lab}^2 + U_{ref}^2\right)}} \quad (1)$$

x_{lab}: Measured value of the participating laboratory
x_{ref}: Reference value
U_{lab}: Expanded Uncertainty (k = 2)of participant
U_{ref}: Expanded Uncertainty (k = 2)of reference value

The reference value in this equation is the deviation of the artefact reading from the NML's applied pressure at the nominal calibration points. Similarly, the measured value of the participating laboratory is the deviation of their reported value to the nominal calibration points. This ensures the uniformity of values to be compared. Moreover, the expanded uncertainties were reported with a coverage factor of $k = 2$ indicating a confidence level of approximately 95%.

Figure 2 shows the performance of participants in the two PTs. More participants joined in 2016 with 88% (14 out of 16) satisfactory performance compared to the 40% (2 out of 5) in 2010. Two participants joined both the PTs and one participant performed better in 2016 while the other still failed.

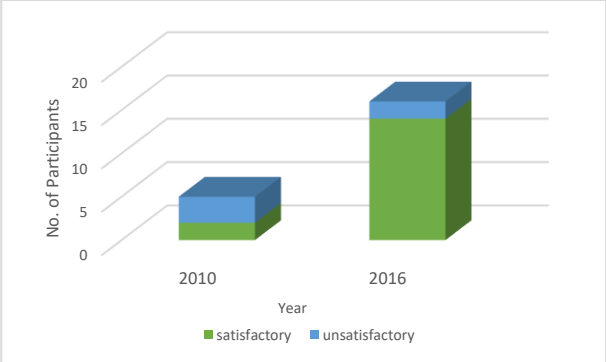

Figure 2: Performance percentages

In 2010, four out of the five participants were able to calibrate the artefact in its full capacity while one participant did not submit a result in the last two highest points. Meanwhile, in 2016, all the participating laboratories were able to calibrate the artefact as a whole.

Tables 2 and 3 show the *En* value of the participants in 2010 and 2016 respectively.

Table 2: Summary of 2010 participants' *En* values relative to the nominal pressure value

Nominal Pressure	LAB A	LAB B	LAB C	LAB D	LAB E
	En	En	En	En	En
kPa					
0	0.00	0.00	0.00	0.00	0.00
2500	0.21	-0.16	-0.14	0.04	0.26
5000	0.14	-0.11	-0.25	0.13	0.18
7500	0.52	0.05	-0.15	0.41	0.87
10000	0.32	0.09	-0.86	-0.07	0.40
12500	-0.21	-0.26	-2.11	-0.87	-0.69
15000	-0.38	0.12	-2.77	-0.64	-0.79
17500	-0.77	-0.36	-3.06	-1.31	-1.82
20000	-0.74	-0.32	-3.27	-1.36	-1.77
22500	-0.83	-0.15	-3.27	-1.42	
25000	-0.97	-0.42	-3.61	-1.55	

4. DISCUSSION OF RESULTS

The satisfactory performance of laboratories is determined when $|E_n| \leq 1$ in all the prescribed calibration points. In 2010, only 95% of the total calibration points with $|E_n| \leq 1$ was required to be considered as a satisfactory performance of a laboratory but this was later on corrected to 100% of the calibration points in 2016. Some participants interpreted that the 95% confidence level in the uncertainty budget estimate may also be applied in the inter-laboratory comparison, thus, assuming that they don't need to perform well in all the measurement points since there is a 5% margin of error. The NML had to explain the rationalization that the 5% margin of error cannot be tolerated in the measurement procedure since the basic requirement of the PT was to calibrate the artefact as a whole and any doubt in their procedure must well be accounted for in their uncertainty budget and not on their measurement value. Also, the 95% satisfactory performance will not be possible in the calibration points prescribed since any one point is already 10% of the artefact's ten calibration points.

For illustration purposes, Figures 3 and 4 show the participant's deviation from the reference value with its corresponding uncertainties in the minimum and maximum calibration points, respectively. The 2010 participants are represented by the blue markers and labelled alphabetically (LAB A to LAB E) while participants in 2016 are of green markers and are labelled with numbers (LAB1 to LAB16). Comparing the participants' performance in the two PTs, all the participants performed satisfactorily in the minimum calibration point in 2010 as opposed to those in 2016 with 1 participant whose value is already lying beyond the limit if not with its uncertainty. Differences in the computed uncertainty values depended mainly on the standard they used, mostly a digital pressure gauge, only a few used a pressure calibrator and a deadweight tester. Moreover, in 2010, while the NML prescribed a guideline for measurement uncertainty calculations, most participating laboratories estimated the expanded uncertainties using their laboratory procedure and technique with the notion that declaring a low uncertainty means better performance. Contrastingly, in 2016, all the participating laboratories followed the agreed guideline on procedure and uncertainty estimates, with the lower limit as the accuracy of the artefact. This ensured that measurement uncertainties were neither over nor under-estimated.

Table 3: Summary of 2016 participants' *En* values relative to the nominal pressure value

Nominal Pressure	LAB1	LAB2	LAB3	LAB4	LAB5	LAB6	LAB7	LAB8	LAB9	LAB10	LAB11	LAB12	LAB13	LAB14	LAB15	LAB16
	En	En	En	En	En	En	En	En	En	En	En	En	En	En	En	En
kPa																
0	0.00	-0.05	0.00	0.00	0.00	0.00	0.00	0.00	0.00	-0.43	0.00	0.00	0.00	0.00	0.00	0.00
2500	0.03	-0.05	0.08	-0.25	-0.27	1.00	-0.02	-0.14	0.57	-0.06	0.43	0.34	0.01	-0.37	0.16	0.11
5000	0.02	-0.20	0.05	-0.25	-0.46	0.50	0.06	-0.17	0.62	0.29	0.19	0.20	0.02	-0.28	0.22	-0.14
7500	-0.17	-0.48	-0.12	-0.33	-0.33	0.14	-0.14	-0.48	0.28	-0.27	-0.06	-0.02	-0.03	-0.26	0.17	0.11
10000	-0.21	-0.53	-0.11	-0.16	-0.45	-0.52	-0.01	-0.49	0.53	-0.38	0.15	0.16	-0.11	-0.23	0.18	0.07
12500	-0.16	-0.55	0.03	-0.14	-0.65	-1.14	-0.13	-0.76	0.06	-0.42	0.19	-0.22	-0.28	0.12	0.31	-0.22
15000	-0.63	-0.88	-0.39	-0.41	-1.43	-0.72	-0.01	0.32	0.25	-0.61	0.15	-0.02	-0.27	0.26	0.14	-0.40
17500	-0.49	-0.79	-0.16	-0.22	-1.17	-0.85	-0.01	0.14	0.09	-0.77	-0.04	0.13	-0.16	0.29	0.45	-0.69
20000	-0.33	-0.73	0.00	0.09	-1.30	-1.19	-0.02	0.04	0.12	-0.80	0.56	0.47	-0.43	0.09	-0.08	-0.86
22500	0.18	-0.78	-0.12	0.08	-1.07	-1.01	0.02	0.00	0.06	-0.62	0.84	0.05	-0.48	-0.99	-0.42	-0.97
25000	0.31	-0.43	-0.19	0.27	-0.96	-1.28	-0.27	-0.41	-0.11	-0.49	0.71	0.38	-0.71	-0.89	-0.32	-0.96

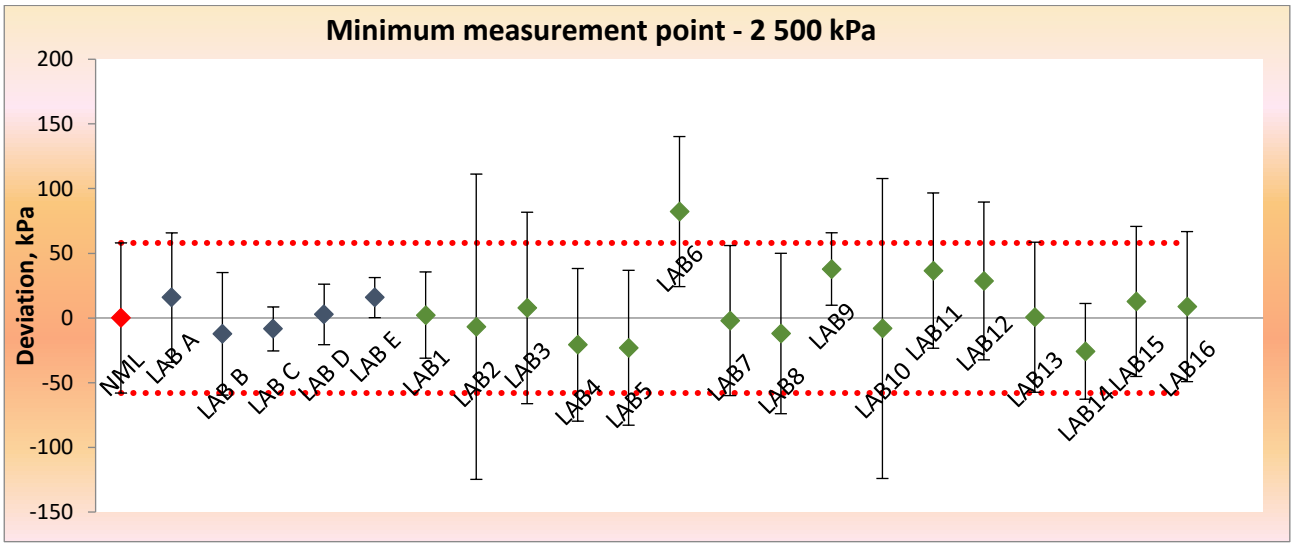

Figure 3: Participant's deviation from the reference value and its corresponding uncertainties at 2 500 kPa

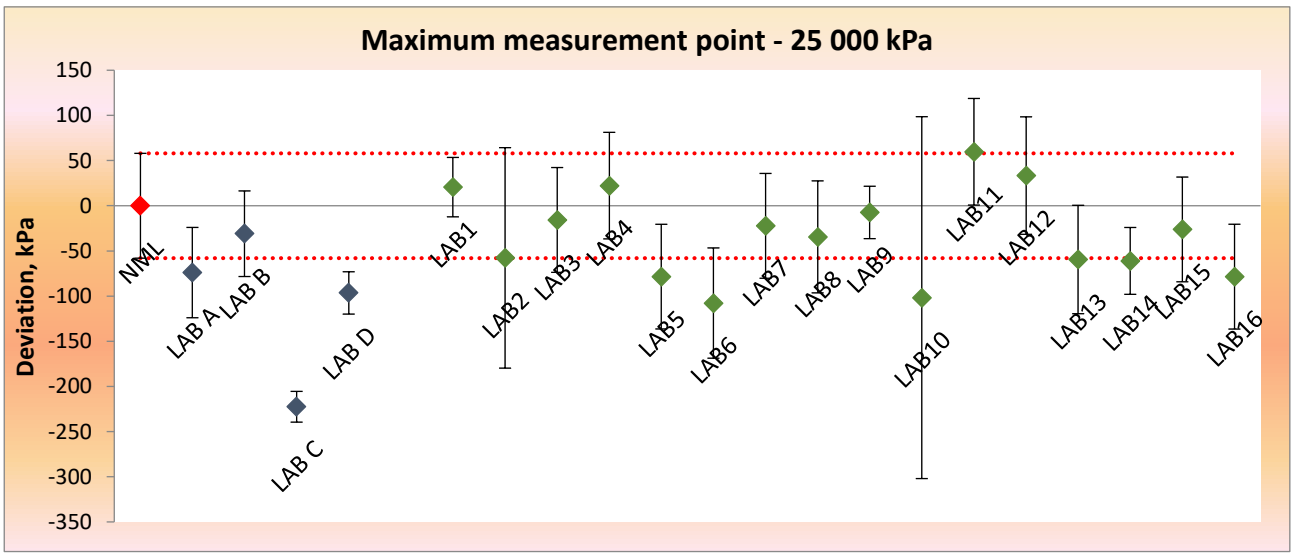

Figure 4: Participant's deviation from the reference value and its corresponding uncertainties at 25 000 kPa

5. EVALUATION OF COMPARISONS

Comparing the two PTs as a whole, defined factors affecting the performance of participating laboratories are summarized in Figure 5.

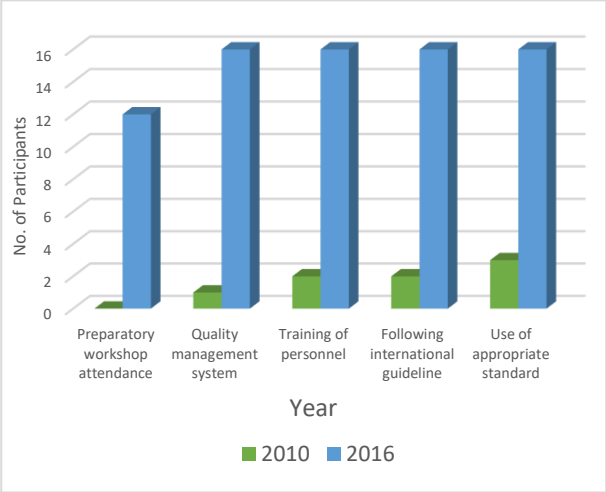

Figure 5: Defined factors affecting lab performance

The earlier mentioned preparatory workshop not done in 2010 but is conducted in 2016 proved to be one key factor that led to the increased satisfactory performance of participants. In 2016, four laboratories was not able to attend this workshop, however 2 laboratories asked for details after the event and strictly followed the agreed procedure, 1 lab with satisfactory result but did not follow the agreed limit of uncertainty and 1 that did not perform satisfactorily. In both PTs, the main objective of the participation of laboratories is mostly to fulfill the ISO/IEC 17025 requirement for PT participation. The urgency of this requirement however was not fully realized by the 2010 participants since the accreditation to ISO/IEC 17025 was fairly new in the country during that time. Moreover, most 2010 participants have inexperienced or untrained personnel and are unfamiliar with the calibration method used by the NML. Contrastingly, in 2016, most laboratories have trained personnel and have proven competencies in scopes other than the pressure field. Knowledge on the estimation of uncertainty budget also significantly improved through training and as seen by their submitted results. An improved selection of standard and upgraded facilities by the laboratories was also more evident in the latter PT.

It is most probable that the 2010 PT was considered as a test run by the participating laboratories, with 2 labs participating satisfactorily on both, 1 lab that improved on 2016, 1 that still unsatisfactorily performed, and 1 that did not continue to be a calibration laboratory, this is also the participant that did not complete the measurement due to inappropriate standard. The 2016 participants on the other hand are mostly maintaining their ISO/IEC 17025 accreditation already or are in the process of acquiring their accreditation in the pressure scope, supported by this intercomparison. In both PTs, all the laboratories with unsatisfactory performance were recommended to review their calibration method and uncertainty budget analysis, investigate sources of error leading to the unsatisfactory results, and initiate necessary corrective actions.

The NML on the other hand as the reference laboratory continuously improves as a PT provider learning from experience, from the handling of the artefact to the analysis of data that is most appropriate to all participants. Consequently, the NML extended its PT offering regularly, with different pressure ranges and other fields of measurement, also the conduct of concluding workshop is planned succeeding PTs. Furthermore, coordination with the local accreditation body as the channel to know the demands for PT in the country for NML's plan on PT provision and in return, the laboratories are made aware of the NML PT offerings. Availability of artefact is still the biggest limitation of the PT provision but is hoped to be resolved to cope-up with ongoing demands on PT.

At present, the laboratories' commitment to quality, supported by courses and training which are not only technical but also in the quality management systems are added contributors to the laboratories' handling of intercomparison prominent to satisfactory performance. The participants have become more aware of good laboratory practices and are encouraged to continuously improve through a refresher and new Metrology awareness courses.

6. SUMMARY AND CONCLUSION

The intercomparisons, 2010 and 2016 are two independent PTs and are generally considered successful in terms of results, coordination, and the experience gained by the participants and the reference laboratory. Measurement results revealed the calibration and measurement capabilities of each participating laboratory. Based on the requirements of ISO/IEC 17043:2010 [5], the performances are mostly satisfactory, in terms of the *En* values, especially in the 2016 PT. This also indicates that the measurement practices of these participants greatly improved and are aligned and complying with an internationally validated method. The PT schemes offered by the NML will be of continuous improvement to support further progress of the local calibration laboratories in the Philippines.

7. REFERENCES

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