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PROFICIENCY TESTING (PT) PROGRAM UNDER NABL IN THE PRESSURE RANGE 7- 70 MPA USING DIGITAL PRESSURE CALIBRATOR (DPC)

This paper describes the proficiency testing of the seven laboratories, accredited by National Accreditation Board for Testing and Calibration of Laboratories (NABL), India using a Digital Pressure Calibrator (DPC) [Model No. – H540/101, Sl. No.- MC808, make-DH-Budenberg, U.K.] as an artifact in the pressure range 70 bar to 700 bar (7 MPa to 70 MPa). The primary objective for organizing this proficiency testing (PT) is to assess the laboratory's technical competence to perform measurements and also fulfilling the requirement of ILAC/APLAC in regards to the compatibility of results submitted by these laboratories. National Physical Laboratory (NPLI), New Delhi, India has the responsibility of coordinating this programme and also acted as a reference laboratory.

The stability of the artifact was assessed by measurements made at NPLI before and after the circulation of the artifact. The comparison was carried out at 11 arbitrarily chosen pressure points i.e. 0.7 MPa, 14 MPa, 21 MPa, 28 MPa, 35 MPa, 42 MPa, 49 MPa, 56 MPa, 63 MPa and 70 MPa throughout the entire pressure range. Six measurements were performed at each eleven pressure points, three each in increasing and decreasing orders of pressures, respectively. Laboratories were advised to report the values of various parameters related to the artifact including the temperatures at which the measurements were made. As it was a digital gauge, all participating laboratories were requested to perform the measurement at a temperature sufficiently close to 23° C so that the standard uncertainty of the various coefficient of the artifact do not contribute to the overall measurement uncertainty. Data are analyzed as per ISO/IEC GUM document. The normalized error (E_n) values are estimated in the entire pressure range. These results are quite encouraging for the manufacturers and calibration laboratories that are facing a new environment after the WTO agreement.

Keywords: pressure, calibration, laboratory accreditation

1. INTRODUCTION

The primary objective of organizing this PT is to assess the laboratory's technical competence to perform measurements. It also supplements the laboratory's own internal quality control procedures and provides objective evidences that a laboratory is competent enough and can achieve the level of uncertainty for which accreditation is granted. As per ILAC/ALLAC, all accredited laboratories are expected to participate in the proficiency testing, which will help them to improve accuracy, reliability and reproducibility of calibration results and to have the measurement traceability to the national metrology institute i.e. NPLI, New Delhi, India. It is hoped that this exercise gives them the confidence of their technical competence of routine calibration/services rendered to clients. Document NABL-162 (2001) describes the administrative procedures and operation of proficiency testing to be followed by NABL as well as all participating laboratories [1].

2. METHODOLOGY

The PT programme is designed as per guidelines stipulated in NABL-162, ISO/IEC Guide 43 [2] and ISO/IEC 17025 [3]. The main steps involved in organizing the program are sending invitation letters to the participating laboratories, selection and procurement of the proper artifact, preparation and circulation of the 'Technical Protocol', selection of pressure

points for comparison, finalization of circulation programme of the artifact, coordination of the movement of the artifact at different participating institutes, characterization of the artifact at the beginning and end of the programme at NPLI for establishing the stability of the calibration data, compilation of measurement results and data analysis.

2.1. Invitation to laboratories

This is the second set of PT conducted by NPLI in the pressure range 7 MPa to 70 MPa. As has been discussed, there were 28 laboratories accredited by NABL from Document 502 [4] and out of which 10 laboratories qualified for participation in the PT with high precision dead weight testers which have the best measurement capabilities better than 0.05 % of the full scale pressure. A second category of laboratories of which the measurement capabilities are better than 0.25 % and coarse than 0.05 % of the full scale pressure, has been invited. Initially, invitation letters were sent to 12 laboratories and 10 laboratories responded to our invitation and finally, eight laboratories participated in this PT. However, M/s VARLAB Instrumentation Services (VIS), Kochi had performed the measurements but did not submit their results in spite of several reminders.

| Specifications of the artifact | | | | | | | | |
|--------------------------------|-------------------------------------|--------------------------|--|--|--|--|--|--|
| Make: | Sl. No H540/101 | Full Scale Range: | | | | | | |
| DH-Budenberg, U.K. | Electronic Module Sl. No.: -MC808 | 0 to 70 Mpa | | | | | | |
| Measurement Range in this | Resolution: 0.1 bar for the present | Measurement Uncertainty: | | | | | | |
| Comparison: 0 to 700 bar | comparison using default settings | 0.05 % of the full scale | | | | | | |
| Power Supply: AC Mains | Weight : 17 kg | Type: Strain Gauge Type | | | | | | |
| Operated 220 V / 50 Hz. | | Pressure Sensor | | | | | | |

Table 1. Detailed manufacturer's specifications of the artifact.

2.2. Selection and procurement of the artifact

Based on the replies of different questionnaires sent to the participating laboratories and with suitable in depth analysis of the performance criteria of these laboratories, it was decided that a high precision digital pressure calibrator is the best option to be used as an artifact. The artifact used for the measurements is a high precision Digital Pressure Calibrator, Serial No. H540/101, make-DH-Budenberg, UK. Detailed manufacturer's specifications of the artifact are given in Table 1.

2.3. Preparation of the 'Technical Protocol'

A copy of the 'Technical Protocol' containing the details of the artifact, handling, calibration procedure, environmental conditions to be maintained, calculation of the results, reporting of the results etc. was provided to all the participating laboratories before arrival of the artifact in their organization. Laboratories were advised to ensure that the various instructions in the 'Technical Protocol' were followed carefully and completely and implemented as instructed. The details of final circulation programme, calibration schedule and reporting of results are shown in Table 2.

| Name of the Laboratory | Contact Person (s) | Artifact Arrival Date | Date(s) of Calibration | Artifact Dispatch Date | Remarks, if any / Date(s) of Reporting Results |
|------------------------------|------------------------|-----------------------------|---------------------------|------------------------------|---|
| National Physical Laboratory | Dr. Sanjay Yadav / | 01-05-2003 | 29-05-2003 to | 31-07-2003 | - |
| (NPLI), | Dr. A.K. Bandyopadhyay | | 03-06-2003 | | |

Table 2. Details of circulation scheme and calibration schedule.

| New Delhi | | | | | |
|------------------------------------|-----------------------------|-----------------|---------------|------------|------------|
| Neel Engineering Solutions (NES), | Mr. O. P. Verma | 01-08-2003 | 09-11/08-2003 | 31-08-2003 | 18-06-2004 |
| Faridabad | | | | | |
| C & I Systems (C & I S), Kota | Mr. Ashok Patni | 01-09-2003 | 13-09-2003 | 17-09-2003 | 05-01-2004 |
| Bharat Heavy Electricals Limited | Mr. R. K. Vapta | 18-09-2003 | 28-09-2003 | 07-10-2003 | 17-10-2003 |
| (BHEL), Bhopal | | | | | |
| Yenkay Instruments & Controls Pvt. | Mr. K. S. Yenpure | 07-10-2003 | 30-10-2003 | 03-11-2003 | 06-03-2004 |
| Ltd. (YICPL), Pune | | | | | |
| Nishitronics Instrumentation (NI), | Mr. Vijay Hingmire | 03-11-2003 | 16-11-2003 | 25-11-2003 | 03-01-2004 |
| Pune | | | | | |
| WAAREE Instruments | Mr. N. Srinivas | 25-11-2003 | 05-12-2003 | 07-12-2003 | 08-01-2004 |
| Ltd.(WAAREE), Dadra | | | | | |
| VARLAB Instrumentation Services | Mr. K.R.V. Verma | 12-12-2003 | * | 09-01-2004 | * |
| (VARLAB), Kochi | | | | | |
| National Physical Laboratory | Dr. Sanjay Yadav / | 12-01-2004 | - | 13-01-2004 | - |
| (NPLI), New Delhi | Dr. A.K. Bandyopadhyay | | | | |
| National Council for Cement and | Mr. Sarpal Singh | 14-01-2004 | 04-03-2004 | 09-03-2004 | 26-04-2004 |
| Building Materials (NCCBM), | | | | | |
| Ballabhgarh | | | | | |
| National Physical Laboratory | Dr. Sanjay Yadav / | 09-03-2004 | 6-8/03-2004 | - | - |
| (NPLI), New Delhi | Dr. A.K. Bandyopadhyay | | | | |
| | * Results are not submitted | d by this labor | atory. | | |

2.4. Circulation and movement of the artifact

Participants were advised to complete the measurements within two weeks and then dispatch the artifact to next participant within next one week. It is very satisfying to mention that the functioning of the programme was almost smooth, except a little delay at one or two participants, and all the participants performed their measurements well in time. There was no technical problem, fault, snag or difficulty reported by any of the participants. A schematic diagram of the movement of the artifact is depicted in Fig. 1.

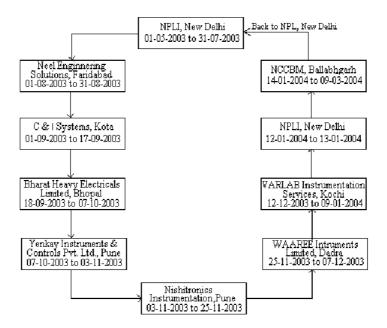
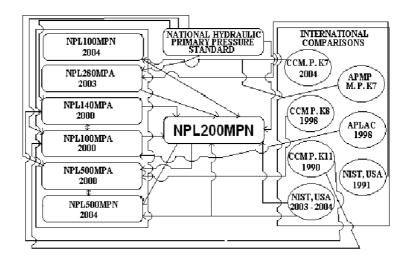


Fig. 1. Circulation and movement of the artifact during comparison.

2.5. Measurand

The measurand in the present proficiency testing is 'pressure' measured by the participating laboratory's standard. All the participants were advised to perform six

observations at each pressure point (10 in the present case), three each in increasing and decreasing orders of pressures and report their measurement results after applying temperature corrections at 23°C. It must be mentioned here since the parameter "pressure" is also connected to the local acceleration of gravity " g_{local} ", therefore appropriate corrections are made during the processing of the data, so that the contribution of " g_{local} " is minimized.



2.6. Characterization of the artifact and assigning of reference values

Fig. 2. Traceability tree for the NPL200MPN, the secondary hydraulic pressure standard used for the characterization of the artifact.

The characterization of the artifact was performed against the national hydraulic secondary pressure standard, designated as NPL200MPN, first at the start of the programme during July, 2003 and finally at the end of programme during March, 2004 using the wellestablished method of cross-floating of pressure balances [5-8]. The traceability of the NPL200MPN is established by cross-floating it against national primary pressure standard [9,10], designated as NPL1-H1. NPL200MPN has also participated in the recently concluded bilateral comparison with NIST, USA. Our results agree well within 1.0 x 10⁻⁵ with NIST, USA and are also well within our claimed measurement standard uncertainty of 40 x 10⁻⁶. A complete traceability tree of the NPL200MPN is depicted in Fig. 2 [11, 12, 13]. The characterization of the artifact was performed at 10 pressure points i.e. (7, 14, 21, 28, 35, 42, 49, 56, 63 and 70) MPa, selected for the present comparison and observations were repeated six times, three times in increasing order and three times in decreasing order, for each pressure point and the values of pressure generated, their repeatability and expanded uncertainty were computed using computer softwares developed for this purpose [14,15]. The details of the measured pressures p_1 , and p_2 , and their measurement uncertainties are shown in Table 3 for both the successive calibrations performed in July 2003 and March 2004. The reference values of pressure measured, p, are the arithmetic mean of the data obtained during these two calibrations. The detailed uncertainty budget thus prepared for the measurements performed on the artifact is shown in Table 4.

Table 3. Details of metrological characteristics of the artifact and assignment of reference values. (All the values reported are at $g_{\text{NPL}} = 9.7912393 \text{ m/s}^2$ and temperature of $T_r = 23^{\circ}\text{C}$).

| | | reported are at SNPL | | 9.1912595 III/5 u | | | | |
|---|----------|----------------------|----------|--------------------|------------------|-----------------|-----------------|------------------|
| | Nominal | Pressure | Pressure | Average | Standard | Deviations from | Deviations from | Uncertainty |
| | Pressure | (MPa) | (MPa) | Pressure | Deviations of | Average Values | Average Values | Evaluated |
| | MPa | p_1 | P_2 | (MPa) | Average Pressure | July 2003 | March 2004 | Through |
| | | July | March | p | (MPa) | (MPa) | (MPa) | Stability of the |
| | | 2003 | 2004 | Ref. Values | | | | Artifact (MPa) |
| [| 7 | 7.002 | 7.004 | 7.003 | 0.001 | -0.001 | 0.001 | 0.014 |

| 14 | 14.005 | 14.010 | 14.008 | 0.003 | -0.002 | 0.002 |
|----|--------|--------|--------|-------|--------|--------|
| 21 | 21.006 | 21.012 | 21.009 | 0.004 | -0.003 | 0.003 |
| 28 | 28.008 | 28.013 | 28.010 | 0.003 | -0.002 | 0.002 |
| 35 | 35.013 | 35.020 | 35.016 | 0.005 | -0.004 | 0.004 |
| 42 | 42.011 | 42.013 | 42.012 | 0.002 | -0.001 | 0.001 |
| 49 | 49.017 | 49.019 | 49.018 | 0.002 | -0.001 | 0.001 |
| 56 | 56.019 | 56.019 | 56.019 | 0.000 | 0.000 | 0.000 |
| 63 | 63.023 | 63.021 | 63.022 | 0.001 | 0.001 | -0.001 |
| 70 | 70.027 | 70.027 | 70.027 | 0.000 | 0.000 | 0.000 |

Table 4. Uncertainty budget of the artifact at 70 MPa and $T_r = 23^{\circ}$ C.

| SOURCE OF UNCERTAINTY (Xi) | ESTIMATES (x _i) (MPa) | LIMITS ±∆x _i (MPa) | PROBABILITY DISTRIBUTION – TYPE A or TYPE B FACTOR | STANDARD UNCERTAINTY u(xi) (MPa) | SENSITIVITY COEFFICIENT | UNCERTAINTY CONTRIBUTIO N u _i (y) (MPa) | DEGREE OF FREEDOM (uf) |
|--|--------------------------------------|-------------------------------------|--|--|----------------------------|--|---------------------------|
| Uncertainty of the Standard u_{B1} | 70 | 0.0047 | Normal – Type B | 0.0047 | 1 | 0.0047 | × |
| Repeatability in the First Calibration (Maximum) u_{A1} | 0.010 | 0.011 | Normal – Type A / \sqrt{n} | 0.0043 | 1 | 0.0043 | 5 |
| Repeatability in the Second Calibration (Maximum) u_{A2} | 0.0059 | 0.0059 | Normal – Type A / √n | 0.0024 | 1 | 0.0024 | 5 |
| Standard Deviation of Two Calibrations u_{A3} | 0.0057 | 0.0057 | Normal – Type A | 0.004 | 1 | 0.004 | x |
| Uncertainty due to Stability (Maximum Deviation from the Reference Value) u_{A4} | 0.02 | 0.02 | Normal – Type B | 0.014 | 1 | 0.014 | 8 |
| $u_c(P)$ | | | k = 1 | | | 0.016 | 874 |
| EXPANDED UNCERTAINTY | | | <i>k</i> = 2.14 | | | 0.032 | |
| The expanded uncertainty ass The relative expanded uncerta | | | | | 10-4 | | |

In order to study the behaviour and stability of the artifact, we have defined a calibration factor (C_f) as follows:

$$C_f = \frac{p_g}{p_s},\tag{1}$$

where, p_g is the gauge pressure shown by the artifact and p_s is corresponding pressure measured by the standard during calibration. We have already mentioned that we have performed experiment in July, 2003 and repeated the same measurement in March, 2004. If we define p_{s1} and p_{s2} are the reference values with the same gauge pressure p_g ,

$$p_{ref} = \frac{p_{S1} + p_{S2}}{2},\tag{2}$$

$$\frac{\delta p}{p} = \frac{p_{S1} + p_{S2}}{p_{ref}}.$$
(3)

In Fig. 3 we have plotted C_f as a function of measured pressure for July 2003 and March 2004 measurements. Similarly, the relative deviations of the measured pressures $\delta p/p$ as a function p is shown in Fig. 3b. It is clearly evident from Fig. 3a that the artifact behaved almost in a similar fashion during both calibrations. The relative deviations of the measured pressures p_{S1} and p_{S2} from the reference values, p_{ref} are found well below + 0.025 % [Fig.

3(b)] which is well within the manufacturer specifications of + 0.05 % and our estimated expanded uncertainty of + 0.05 %. This concludes that the artifact remained stable during the whole PT programme.

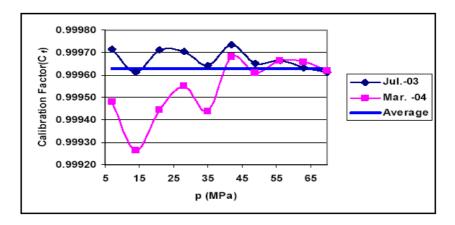


Fig. 3a. The Calibration Factor (C_f) and its average values plotted as a function of applied pressure p for all the three successive calibrations.

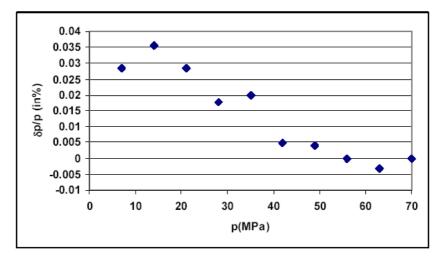


Fig. 3b. Relative deviations of the measured pressures p_{S1} and p_{S2} from the reference values of the successive calibration.

2.7. Participants

Nine laboratories participated in the program. In order to maintain confidentiality in the results, each participating laboratory was assigned a random code number and only these code numbers are used herein thereafter in this PT report. The details of these code numbers are not divulged herein. However, these code numbers have been reported to NABL, separately. The code number assigned to the reference laboratory, NPLI is '1' (Ref.).

2.8. Experimental setup and calibration procedure

All the laboratories were advised to perform the experiment as shown in Fig. 4. The stepby-step calibration procedure was described in the 'Technical Protocol', which was already circulated.

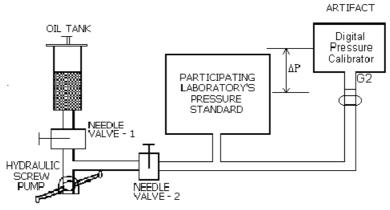


Fig. 4. Experimental setup for measurement.

The calibration of the artifact starts with leak testing and the selection of a reference or datum level. For leak testing, they were requested to pressurize both the standard and the artifact up to 70 MPa with the help of a hydraulic screw pump and needle valves and wait for at least 10 minutes. Thereafter, release the pressure slowly to zero. This process is repeated at least three times to ensure that there are no leaks in the system. After performing all the necessary steps, laboratories were requested to vent the system to atmosphere and wait for at least one hour before starting the observations. Thereafter, it was advised to apply the atmospheric pressure to the system and wait for 10 minutes and record the first observation of '0' pressure in the data sheet. The artifact is then pressurized to a second measurement point i.e. 7 MPa and the corresponding value of the pressure measured by the standard is recorded after applying all corrections i.e. temperature correction, hydrostatic head correction and unit conversion. Subsequently, fix the reading of the artifact (as per nominal value) to the next pressure point and record the pressure measured by the standard. Repeat this process till the full-scale pressure of 70 MPa is achieved. Sufficient time of approximately 10 minutes is given between two successive observations to allow the system to reach a state of thermal equilibrium. They were strictly instructed to keep the relative difference between the nominal pressure (artifact reading) and the measured / applied pressure by the standard better than 0.025 % of the reading.

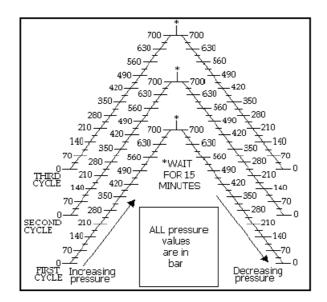


Fig. 5. Sequence of measurements taken.

After reaching full-scale pressure, they were advised to wait for at least 15 minutes before repeating the observations in the decreasing order of pressure till the pressure reaches zero. Thus they were asked to record the total 22 observations, 11 each in the order of increasing and decreasing pressures, to perform one pressure cycle. The measurements are then repeated for at least 3 pressure cycles to make the total number of 66 observations. A layout diagram shown in Fig. 5 presents the sequence of measurements taken. They were also requested to evaluate the uncertainty associated with pressure measurements as per ISO Guide to the Expression of Uncertainty in Measurement / NABL Document 141 [16, 17] and prepare their uncertainty budget at maximum pressure, considering all Type A and Type B uncertainty components.

3. REPORTING OF RESULTS AND DATA ANALYSIS

The laboratories were advised to submit their measurement results on specially designed proformas given in the 'Technical Protocol'. They were also asked to submit copies of the calibration certificates for the reference instruments used in measurements, a calculation sheet for determining the uncertainty in measurements and a calibration certificate issued to the customer for such measurements. The values of measured pressure, acceleration of local gravity and reference temperature reported by participants and the measurement uncertainty estimated at maximum pressure are shown in Table 5. Before compiling and comparing the results the following corrections are applied.

| Nominal | LABORATORY CODE | | | | | | | | | |
|--------------------------|-----------------|-------------|-------------|-----------|-------------|----------|----------|-------------|--|--|
| Pressure bar | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | | |
| 70 | 70.034 | 69.9 | 69.961 | 68.4166 | 69.87 | 69.935 | 70.0307 | 69.5024 | | |
| 140 | 140.079 | 139.957 | 140.035 | 136.9666 | 139.94 | 140.181 | 140.0986 | 139.604 | | |
| 210 | 210.089 | 209.888 | 210.063 | 206.0833 | 209.81 | 210.377 | 210.1715 | 209.647 | | |
| 280 | 280.105 | 279.907 | 280.122 | 275.0666 | 279.88 | 280.59 | 280.257 | 279.806 | | |
| 350 | 350.161 | 349.83 | 350.188 | 345.2333 | 349.95 | 350.852 | 350.3412 | 349.886 | | |
| 420 | 420.123 | - | 420.151 | - | 419.82 | 421.049 | 420.3213 | 419.916 | | |
| 490 | 490.181 | - | 490.275 | - | 490.08 | 491.278 | 490.4494 | 490.063 | | |
| 560 | 560.188 | - | 560.329 | - | 559.96 | 561.54 | 560.5565 | 560.223 | | |
| 630 | 630.224 | - | 630.358 | - | 630.02 | 631.737 | - | - | | |
| 700 | 700.269 | - | 700.441 | - | 700.09 | 701.999 | - | - | | |
| $g(m/s^2)$ | 9.791239 | | | | 9.787011 | | | | | |
| | 3 | 9.78905 | 9.787117 | 9.78147 | 2 | 9.783847 | 9.79096 | 9.79096 | | |
| Ref. Temp.°C | 23 | 22.4-22.6 | 20 | 19.5-20.5 | 20 | 22 | 23 | 23 | | |
| $u(P_{rep}) \ge 10^{-6}$ | 500 or | 462 or | 820 or | 2642 or | 180 or | 143 or | 340 or | 861 or | | |
| (at k = 2) | 0.05 % | 0.046 % | 0.082 % | 0.264 % | 0.018 % | 0.014 % | 0.034 % | 0.086 % | | |
| Traceability | NPLI-H1 | Measure | Measure | FCRI, | Measure | IDEMI, | NPLI, | NCCBM, | | |
| | NIST, | Techniques, | Techniques, | Palghat | Techniques, | Mumbai | New | Ballabhgarh | | |
| | USA | Chennai | Chennai | | Chennai | | Delhi | | | |
| Best Measurement | - | 0.1 bar of | 0.2 % | 0.2 % | 0.08 % | 0.12 % | 0.055 % | 0.6 % | | |
| Capabilities as per | | full scale | | | | | | | | |
| NABL Doc. 502, | | | | | | | | | | |
| 2002 | | 1 | | | | | | 1 | | |

Table 5. Details of the reference values measured pressure (p_{rep}) reported by the participants and other metrological characteristics of the laboratories standards.

3.1. Gravity correction

The measured pressure values reported by the laboratories are corrected for $g_{\text{NPLI}} = 9.7912393$ m/s² (acceleration of gravity at NPL, New Delhi, India) using the following relationship;

$$p' = p_{rep} * (g_{\text{NPLI}} / g_{\text{Local}}), \tag{4}$$

where p' and p_{rep} are the values of corrected and reported pressure, respectively and g_{Local} is the value of acceleration of gravity reported by the laboratory. The relative change of $(\delta g/g_{NPLI})$ with g_{NPLI} is shown in Fig. 6 for different locations of the measurements. After applying gravity correction, the relative change of reported pressure (p_{rep}) with corrected pressure (p') is also plotted as a function of measured pressure for all the laboratories and is shown in Fig. 7. It is evident from Fig. 7 that the relative change of reported pressure (p_{rep}) with corrected pressure (p') is consistent with Fig. 6. For example, the laboratories with Code No. 7 and 8, which are located in a nearby town of reference laboratory i.e., NPLI, the relative change is minimal (< 0.004 %). However, this change is maximum in case of laboratory with Code No. 4 (< 0.1%).

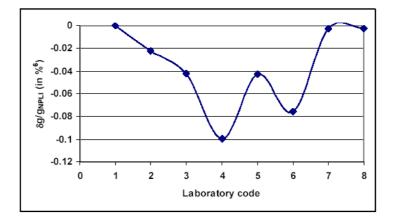


Fig. 6. Variation of $\delta g/g_{\text{NPLI}}$ value at different locations with reference to g_{NPLI} .

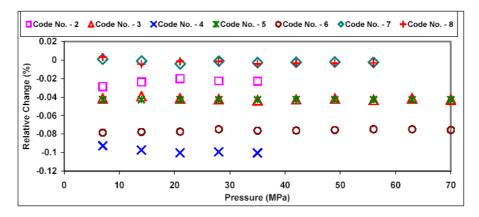


Fig. 7. Relative change (%) in reported pressure (p_{rep}) with corrected pressure (p').

3.2. Estimation of measurement uncertainty

The expanded uncertainty reported by the laboratory (at k = 2) is converted into relative uncertainty and then the final uncertainty is computed using the following formula;

$$U(p') = [\{U(p_{rep}) / p_{rep}\} / 10^{-6}] x p',$$
(5)

where U(p') is the expanded uncertainty of the corrected measured pressure at a coverage factor k = 2, $U(p_{rep})$ is the expanded uncertainty at a coverage factor k = 2 reported by the laboratory at maximum pressure, assuming it as the maximum measurement uncertainty.

3.3. Estimation of normalized error (E_n)

In accordance with international practice, measurement performance has been assessed on the basis of Error Normalized (E_n) number of each measurement. It is suffice to say that E_n values are estimated for each participant as per guidelines in the literature [5, 6, 18];

$$E_n Value = \frac{p_{LAB} - p_{Ref}}{\sqrt{\{U(p_m)\}^2 + \{U(p_{Ref})\}^2}},$$
(6)

where $p_{LAB} = p'$ is the participant's measured pressure value, $p_{Ref} = p$ is the calculated reference value, $U(p_m)$ is the participant's claimed expanded uncertainty at a coverage factor k = 2 and $U(p_{Ref})$ is the expanded measurement uncertainty of the reference value at k = 2.

4. RESULTS AND DISCUSSIONS

Details of the corrected pressure (p') for gravity (g_{NPLI}) with relative deviations of measured pressure (p') of each participant from reference value (p) are shown in Table 6.

| Nominal | LABORATORY CODE | | | | | | | | | | | |
|--------------------------|-----------------|------------------|------------------|------------------|------------------|------------------|------------------|-----------|--|--|--|--|
| Pressure | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | | | | |
| MPa | p (MPa) | <i>p</i> ' (MPa) | <i>p</i> ' (MPa) | <i>p</i> ' (MPa) | <i>p</i> ' (MPa) | <i>p</i> ' (MPa) | <i>p</i> ' (MPa) | p'(MPa) | | | | |
| 7 | 7.003 | 6.992 | 6.999 | 6.848 | 6.990 | 6.999 | 7.003 | 6.950 | | | | |
| 14 | 14.008 | 13.999 | 14.009 | 13.710 | 14.000 | 14.029 | 14.010 | 13.961 | | | | |
| 21 | 21.009 | 20.993 | 21.015 | 20.629 | 20.990 | 21.054 | 21.018 | 20.965 | | | | |
| 28 | 28.011 | 27.997 | 28.024 | 27.534 | 28.000 | 28.080 | 28.026 | 27.981 | | | | |
| 35 | 35.016 | 34.991 | 35.034 | 34.558 | 35.010 | 35.112 | 35.035 | 34.990 | | | | |
| 42 | 42.012 | - | 42.033 | - | 42.000 | 42.137 | 42.033 | 41.993 | | | | |
| 49 | 49.018 | - | 49.048 | - | 49.029 | 49.165 | 49.046 | 49.008 | | | | |
| 56 | 56.019 | - | 56.057 | - | 56.020 | 56.196 | 56.057 | 56.024 | | | | |
| 63 | 63.022 | - | 63.062 | - | 63.029 | 63.221 | - | - | | | | |
| 70 | 70.027 | - | 70.074 | - | 70.039 | 70.253 | - | - | | | | |
| |] | Relative devia | ations of mea | sured pressur | e from referen | nce values (% | 5) | | | | | |
| 7 | - | -0.17 | -0.06 | -2.21 | -0.19 | -0.07 | 0.00 | -0.76 | | | | |
| 14 | - | -0.06 | 0.01 | -2.12 | -0.06 | 0.15 | 0.02 | -0.34 | | | | |
| 21 | - | -0.07 | 0.03 | -1.81 | -0.09 | 0.21 | 0.04 | -0.21 | | | | |
| 28 | - | -0.05 | 0.05 | -1.70 | -0.04 | 0.25 | 0.06 | -0.10 | | | | |
| 35 | - | -0.07 | 0.05 | -1.31 | -0.02 | 0.27 | 0.05 | -0.08 | | | | |
| 42 | - | - | 0.05 | - | -0.03 | 0.30 | 0.05 | -0.05 | | | | |
| 49 | - | 0.06 | - | 0.02 | 0.30 | 0.06 | -0.02 | | | | | |
| 56 | - | - | 0.07 | - | 0.00 | 0.32 | 0.07 | 0.01 | | | | |
| 63 | - | 0.06 | - | 0.01 | 0.32 | - | - | | | | | |
| 70 | - | - | 0.07 | - | 0.02 | 0.32 | - | - | | | | |
| $u(p_{rep}) \ge 10^{-6}$ | 2500 | 1567 | 1543 | 366 | 2851 | 4061 | 1500 | 2291 | | | | |
| (at k = 2) | or 0.25 % | or 0.16 % | or 0.16 % | or 0.04 % | or 0.29 % | or 0.41 % | or 0.15 % | or 0.23 % | | | | |

Table 6. Details of the corrected pressure (p') deviations from reference values (p).

The measured values, the associated uncertainties and calculated E_n values at individual pressure points are evaluated but not shown in this paper. The normalized error (E_n) values

are summarized in Table 8 for the entire pressure scale of 7 - 70 MPa. In general, the performance of the laboratory is considered satisfactory if the normalized error E_n is < 1. The tabulated data reveal that there are total 56 measurement results. Measurement results of only one laboratory with Code No. 3 out of total seven laboratories are well within acceptable limits of normalized error over the entire pressure range of 7 - 70 MPa. However, the measurement results of the laboratory with Code No. 7 are also quite good having E_n values > 1 only at one pressure point.

The E_n value of 30 measurement results out of total 56, is < 1, which is 53.6 % success rate and are within the acceptable limit. E_n values of the laboratory referred as Code No. 4 and 6 are beyond the acceptable limit throughout the entire pressure scale.

| | | | <i>n</i> | | | 1 | | | | |
|---|---------|-------------------|----------|-----------------|---------|---------|---------|---------|--|--|
| Nominal Pressure | | LABORATORY CODE | | | | | | | | |
| Мра | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | | |
| 7 | - | -2.48 | -0.65 | -8.41 | -3.60 | -1.27 | -0.03 | -7.64 | | |
| 14 | - | -0.95 | 0.11 | -8.07 | -1.06 | 2.85 | 0.28 | -3.39 | | |
| 21 | - | -1.08 | 0.31 | -6.85 | -1.69 | 4.09 | 0.70 | -2.09 | | |
| 28 | - | -0.71 | 0.50 | -6.43 | -0.70 | 4.78 | 0.94 | -1.04 | | |
| 35 | - | -1.06 | 0.52 | -4.93 | -0.32 | 5.25 | 0.90 | -0.76 | | |
| 42 | - | - | 0.51 | - | -0.54 | 5.69 | 0.83 | -0.47 | | |
| 49 | - | - | 0.64 | - | 0.43 | 5.76 | 0.95 | -0.21 | | |
| 56 | - | - | 0.70 | - | 0.05 | 6.10 | 1.13 | 0.09 | | |
| 63 | - | - | 0.66 | - | 0.20 | 6.07 | - | - | | |
| 70 | - | - | 0.69 | - | 0.33 | 6.21 | - | - | | |
| | | | Unce | ertainty Detail | s | | | | | |
| $u(p_{rep}) \ge 10^{-6}$ | 2500 or | 1567 or | 1543 or | 366 or 0.04 | 2851 or | 4061 or | 1500 or | 2291 or | | |
| (at k = 2) | 0.25 % | 0.16 % | 0.16 % | % | 0.29 % | 0.41 % | 0.15 % | 0.23 % | | |
| Maximum Relative | 2500 or | 3000 or | 2900 or | 2500 or | 3800 or | 4800 or | 2900 or | 3400 or | | |
| Combined | 0.25 % | 0.3 % | 0.29 % | 0.25 % | 0.38 % | 0.48 % | 0.29 % | 0.34 % | | |
| Uncertainty at $k = 2$ $U(p') \ge 10^{-6}$ | | | | | | | | | | |
| Best Measurement Capabilities as per NABL Doc. 502, 2002 | - | 1.0 % to 0.5 % | 1.0 % | 0.25 % | 0.3 % | 0.3 % | 0.6 % | 0.6 % | | |

Table 7. Summary of the E_n values and the estimated combined expanded uncertainty.

The larger the absolute value of the E_n number, the bigger the problem as in case of laboratories with Code No. 4 and 6. An E_n value greater than unity means that there is a significant bias in the laboratory's results and that the quoted value of its associated uncertainty does not adequately accommodate that bias and needs further investigations by the laboratory.

The graphical representation of the normalized error values (E_n) as a function of measured pressure is shown in Fig. 8 and their relative deviations from the reference pressure values are depicted in Fig. 9. It is clear from Fig. 9 that relative deviations of 30 measurement points out of the total 56, i.e. 53.6 % are well within their best measurement capabilities reported in NABL Document 502 [4] which also agree with their calculated E_n values. Total 21 measurement results i.e. 37.5 % are found within the uncertainty band of reference value, while 31 measurements results i.e. 55.4 % fall within their combined uncertainty band.

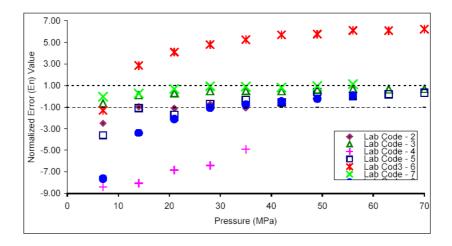


Fig. 8. The E_n value as a function of measured pressure (p'). The gap between two horizontal dotted lines shows the acceptable limit of the normalized error value.

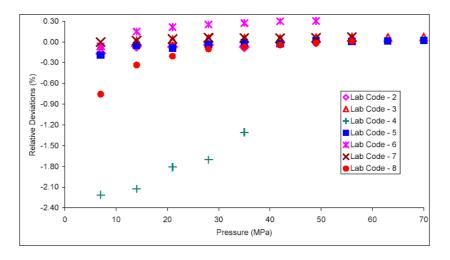


Fig. 9. Relative deviations of measured pressure (p') from the reference value (p).

5. CORRECTIVE ACTION AND SUGGESTION TO THE LABORATORY

As mentioned in section 5, E_n numbers greater than unity require investigations and corrective action by the participating laboratory. The laboratory's management needs to ensure that the problem is rectified and procedures are put in place to prevent a recurrence. Laboratories with Code Nos. 4 and 6 were asked to review the results and take appropriate corrective actions. Laboratories with Code Nos. 2, 5 and 8 were also advised to review their results carefully and take appropriate corrective actions. These laboratories were requested to improve their calibration facilities and to modify the measurement method and to estimate the measurement uncertainties properly.

6. CONCLUSION

The proficiency testing concludes that out of the total **56** measurement results reported here in this paper, **30 (53.6 %)** are in agreement with the reference laboratory. The E_n values of only one laboratory are within acceptable limits throughout the entire pressure scale. However, the E_n values of one other laboratory with Code No. 7 are also quite acceptable except one pressure point of 56 MPa. The E_n values of two laboratories with Code No. 4 and 6 are found beyond the acceptable limit throughout the entire pressure scale except one pressure point. The deviations between laboratories values and reference values at 21 measurement points (37.5 %) are almost well within the uncertainty bands of the reference values. Total 31 measurements results, i.e. 55.4 %, fall within their combined uncertainty band. However, 53.6 % measurement results are found well within their best measurement capabilities reported in NABL Document 502. Overall, the results are considered to be fairly good, being the first proficiency testing for all the participating laboratories.

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PROGRAM TESTOWANIA KOMPETENCJI LABORATORIÓW AKREDYTOWANYCH PRZEZ *NABL* W ZAKRESIE POMIARÓW CIŚNIENIA 7-70 MPA Z UŻYCIEM CYFROWEGO KALIBRATORA CIŚNIENIA

Streszczenie

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