Standardization of pressure calibration (7-70 MPa) using digital pressure calibrator

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Received 25 May 2009; revised 25 November 2009; accepted 26 November 2009

Proficiency testing of pressure measurement was carried out among 21 laboratories using digital pressure calibrator. Total 178 pressure measurements (pressure range, 7-70 MPa) were recorded at 10 equally spaced pressure points. Normalized error ($E_n$) values of 15 laboratories were within acceptable limits as per guidelines given by National Measurement Institute, Govt of India. $E_n$ values of 3 laboratories were found beyond the acceptable limit.

Keywords: Interlaboratory comparison, Pressure calibration, Pressure metrology, Proficiency testing

Introduction
As trade barriers are lifted, manufacturers must be prepared to comply with international standards, including assessment of competency in measurements. In order to establish international / national compatibility, uniformity and affirmation of measurement results, considerable efforts are being made globally so that measurements made in one location in the world are equivalent / compatible in other locations on the same or related products. Such tasks are achieved by organizing international comparisons and proficiency testing by inter-laboratory comparisons of measurement results carried out on the same pressure gauge. National Measurement Institutes (NMIs) provide guidelines to industries and other researchers in terms of pressure calibration. This study presents standardization of pressure calibration (range, 7-70 MPa) using digital pressure calibrator.

Methodology
Present proficiency testing programme was designed as per guidelines stipulated in ISO/IEC1 and NABL2. A high precision digital pressure calibrator (DPC), Serial No.- H540/101, make-DH-Budenberg, UK was used for pressure calibration. Selection of measurement points is an important aspect of proficiency testing programme. Entire pressure range (7-70 MPa) was divided into 10 equally spaced measurement points (7, 14, 21, 28, 35, 42, 49, 56, 63 and 70 MPa).

Characterization of Digital Pressure Calibrator (DPC)
Characterization of DPC was performed by direct comparison method3,4 against national hydraulic secondary pressure standard. Traceability of DPC is established by calibrating DPC against a national secondary pressure standard (NPL200MPN) through an unbroken chain5,6. Measurement uncertainty of NPL200MPN is estimated as 68 x 10^-6 x p at a coverage factor $k = 1$.

Before calibration, both instruments (NPL200MPN and DPC) were leveled using leveling screws and spirit level. Necessary weights were placed on the carrier of NPL200MPN and adjusted as per the values of pressure indication on DPC. This is repeated several times so that error due to adjustment of weights can be minimized. Sufficient time, 10 min, was provided between two successive observations so that both systems were in complete equilibrium. At this position, there was no pressure drop in connecting line and consequently no movement of fluid. This procedure was repeated for all 10 pressure points (7, 14, 21, 28, 35, 42, 49, 56, 63 and 70 MPa) and observations were repeated six times (thrice in increasing order and thrice in decreasing order) for each pressure point and values of measured pressure, their repeatability and expanded uncertainty were computed.
Fig. 1—Applied pressure $p$ for three successive calibrations of artifact versus: a) Calibration factor ($C_f$) and its average values; b) Relative deviations (% of reading) of measured pressures $p_1$, $p_2$, and $p_3$; and c) Relative deviations (% of full scale pressure) of measured pressures $p_1$, $p_2$, and $p_3$. 

[Graphs showing the pressure, calibration factor, and relative deviations for different months (June 2006, March 2007, April 2008).]
Arithmetic mean of pressure values \( (p_1, p_2, \text{ and } p_3) \) are reference values of pressure measured \( (p) \) for individual measurement point throughout the entire pressure scale. In order to study stability behaviour of DPC, calibration factor \( (C_f) \) is determined as

\[
C_f = \frac{p_s}{p_3} \quad \ldots (1)
\]

where, \( p_3 \) is reading of DPC and \( p_s \) is corresponding pressure measured by standard during calibration. Artifact behaviour was found identical in all three calibrations. During all three calibrations, DPC behaved almost in a similar fashion (Fig. 1a). Relative deviations of measured pressures \( (p_1, p_2, \text{ and } p_3) \) from reference values, \( p \) were found well within \( \pm 0.033 \% \) of the reading (Fig. 1b) and \( \pm 0.01 \% \) of full scale (Fig. 1c). Deviations were well within manufacturer specifications of 0.05 \% of span (full scale), maximum deviation of 0.033 \% of reading was taken in to consideration to estimate expanded uncertainty \( U(p) = (0.011 + 0.00015 \times p) \) MPa. This concludes that artifact remained stable during whole PT programme within its estimated measurement uncertainty. Stability of a similar type of DPC is also reported\(^7\) in higher pressure range. Measurement uncertainty in pressure calibration estimated at 70 MPa is shown in Table 1.

**Calibration Procedure**

Out of a total of 21 laboratories, each participating laboratory was assigned a random code number while reference laboratory (NPLI) was assigned ‘1.’ All laboratories were advised to install experimental set-up (Fig. 2) and to use clean mineral oil as pressure transmitting fluid. Each laboratory was requested to pressurize both standard and DPC up to 70 MPa at least three times to ensure that there is no leak in the system. In this way, compressibility of transmitting oil, packing of valves, pump plunger and O-ring seals were stabilized to reach an optimum level.

Under default settings and connection scheme of the instrument (Fig. 3), there are two gauge connecting heads at the top of DPC. One of the head, marked as ‘G1’ is closed by dummy plug. Laboratories were asked not to open this gauge connecting head and connect laboratory’s standard with another gauge connecting head, marked as ‘G2’, which is opened. There are also two needle
valves, marked as A and B on top of the artifact, which were closed. Laboratories were instructed to close both valves during measurements. It was also instructed not to use hydraulic screw pump of artifact to generate pressure in the system. Laboratories were requested to use their own pressure generating system in their routine calibration services being rendered to clients.

Participants were not allowed to make any connection with OUTPUTS (three ports). Laboratories were strictly instructed not to pressurize DPC (> 720 bar); over pressure may damage sensor. It was advised to connect DPC to a power supply at least 12 h before starting measurement. It was strongly recommended not to cut power supply before completing measurements. Laboratories were requested not to try to make 'zero adjustment' with DPC and note down initial reading without pressure and make appropriate corrections (+ or -) in subsequent observations.

Calibration of artifact starts with leak testing and selection of a reference or datum level. For leak testing, standard and DPC were pressurized up to 700 bar using hydraulic screw pump and needle valves and wait for at least 10 min. Thereafter, release pressure slowly to zero. This process was repeated at least three times to ensure
Fig. 5—Normalized error value ($E_n$) as a function of measured pressure ($p$) for laboratory with: a) Code No. 2-8; b) Code No. 9-15; and c) Code No. 16-22 (Gap between two horizontal dotted lines shows acceptable limit of normalized error value).
no leaks in the system. In this way, compressibility of transmitting oil, packing of valves, pump plunger and O-ring seals were stabilized to reach an optimum level. Selection of appropriate and precise reference or datum plane is very important for applying hydrostatic head correction. A precise reference or datum plane was then established for standard and DPC for hydrostatic head correction.

After performing all tasks, laboratories were requested to vent the system to atmosphere and wait for at least 1 h before starting observations. An atmospheric pressure was applied to the system and first observation of ‘0’ pressure was recorded after 10 min (Fig. 4). DPC was then pressurized to next measurement point (70 bar) and corresponding value of pressure measured by standard was recorded after applying all corrections (temperature, hydrostatic head and unit conversion). Subsequently, DPC was pressurized to subsequent pressure points and pressure measured by standard was recorded. This process was repeated till full-scale pressure (700 bar) was achieved. Sufficient time (10 min) is given between two successive observations to allow system to reach a state of thermal equilibrium. After reaching full-scale pressure, observations were repeated in decreasing order of pressure till pressure reaches to zero.

All participants were advised to apply temperature and head corrections carefully before submitting results and requested to correct values of measured pressure for 23°C using thermal expansion coefficient of piston - cylinder assembly (if dead weight tester used as standard) or elastic element (if pressure dial gauge or digital calibrator used as standard) using standard method. Participants were also requested to evaluate uncertainty associated with pressure measurements as per ISO / NABL guidelines. Each participant was requested to prepare an uncertainty budget at maximum pressure, considering all uncertainty components as reported.

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**Data Analysis**

Measurement performance of participants had been assessed on the basis of error normalized ($E_n$) calculated for each measurement point. $E_n$ values were estimated at each pressure as

$$E_n = \frac{p - p'}{\sqrt{U(p')^2 + U(p)^2}} \quad \ldots(2)$$

where $p, p'$ participant’s measured pressure value; $p$, calculated reference value; $U(p'),$ participant’s claimed expanded uncertainty at a coverage factor $k = 2$ and $U(p),$ expanded measurement uncertainty of reference value at a coverage factor $k = 2.$ An $E_n$ value ($< 1$) indicates agreement within combined uncertainties for results to be internationally acceptable. An $E_n$ number between $-1$ and $+1$ indicates an acceptable degree of compatibility with reference value when quoted uncertainties are taken into account. $E_n$ number ($> \pm 1$) is unacceptable and requires immediate investigation and corrective action by the laboratory concerned.

**Results and Discussion**

Relative deviations of measured pressure, $p'$, of each participant from reference value, $p$, are shown for laboratories with Code No. 2-8 (Fig. 5a), Code No. 9-15 (Fig. 5b) and Code No. 16-22 (Fig. 5c). Measurement results of 15 laboratories (Code No. 2, 5, 7-9, 12 and 14-22), out of total 21 laboratories, were well within acceptable limits of normalized error over entire pressure range (7-70 MPa). However, measurement results of another 3 laboratories (Code No. 4, 10 and 11) were quite good having $E_n$ values $> \pm 1$ only at one pressure point each. $E_n$ values of remaining 3 laboratories (Code No.-3, 6 and 13) were $> \pm 1$ for 2 or more than 2 pressure points. An $E_n$ value greater than unity means that there is a significant bias in the laboratory’s results and that quoted value of its associated uncertainty does not adequately accommodate that bias, therefore further investigations are needed by the laboratory.

**Conclusions**

Interlaboratory comparison programme (proficiency testing) was carried out in pressure range 7-70 MPa using pressure digital pressure calibrator. Total number of 21 laboratories participated in this programme. The comparison was performed at 10 equally spaced pressure points selected throughout entire pressure range. Total 178 measurement results reported were in agreement with reference laboratory. $E_n$ values of 15 laboratories were within acceptable limits through out the entire pressure scale. However, $E_n$ values of 3 other laboratories were also quite acceptable except only at one pressure point each. $E_n$ values of the remaining 3 laboratories were found beyond acceptable limit for 2 or more than 2 pressure points.
Acknowledgements
Authors thank Dr Vikram Kumar, Director, National Physical Laboratory, New Delhi and Dr Hari Gopal, Director, National Accreditation Board for Testing & Calibration Laboratories, New Delhi for their support and encouragement. Authors also thank Dr P C Kothari, Dr K K Jain, Mr A K Saxena, Dr Naveen Garg, Mr B V Kumaraswamy and Mr Om Prakash, all from NPL, Delhi, for their co-operation. Thanks to 21 accredited laboratories for completing PT on time. Authors also thank secretariat of NABL for administrative help.

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