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Proficiency testing through interlaboratory comparison in the pressure range up to 70 MPa using pressure dial gauge as an artifact

Sanjay Yadav*, V K Gupta, Om Prakash and A K Bandyopadhyay

National Physical Laboratory, Dr K S Krishnan Road, New Delhi 110 012

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The proficiency testing (PT), a quality control, provides an additional means to ensure quality of laboratory testing results. The primary objective of organizing PT is to assess laboratory's technical competence to perform measurements. This paper describes PT of 11 laboratories, nine accredited by National Accreditation Board for Testing and Calibration of Laboratories (NABL), having best measurement capabilities (< 0.25 % of full-scale pressure) by means of interlaboratory comparison using pressure dial gauge (5-70 MPa) as an artifact. This programme was identified by code number NABL-Pressure-PT003. National Physical Laboratory (NPLI), New Delhi coordinated this programme (February 2003–March 2004) and acted as a reference laboratory. The comparison was carried out at 14 arbitrarily chosen pressure points (5, 10, 15, 20, 25, 30, 35, 40, 45, 50, 55, 60, 65 and 70 MPa) throughout the entire pressure range (5–70 MPa). Out of the total 145 measurement results, 86 (59.31%) results were found in good agreement with the results of reference laboratory, NPLI, New Delhi. The relative deviations of the laboratories values with reference values were found almost well within the uncertainty band of the reference values (44.14 % pressure points), within their combined uncertainty band (59.31% pressure points) and within their best measurement capabilities (80.34 % pressure points). Since most of the laboratories have underestimated their measurement uncertainties, 40.69 % measurement results were found out of the combined uncertainty band during this comparison. Overall, results were reasonably good being the first PT for all the participating laboratories. This exercise gives an opportunity to calibration laboratory to demonstrate its technical competence of routine calibration services rendered to clients and to have the measurement traceability to the national metrology institute, NPLI, New Delhi.

Keywords: Proficiency testing, Pressure, Pressure dial gauge, Artifact, Quality control

IPC Code: G01D3/028

Introduction

A wide variety of industrial applications in India are around atmospheric pressure (10^5 Pa - 100 MPa). These diversified industrial applications are in nuclear, thermal and hydro power plants; refineries and petro-chemical companies; drugs and pharmaceutical industries; manufacturing of gases, fertilizers, pesticides and chemicals; synthesis of super hard materials like diamond, optimization of domestic appliances like pressure cooker and filling of cooking gas cylinders, assessment of health like blood pressure monitors, optical, aerospace, defense, meteorological, automotive, semi-conductor, environmental, ventilation, filtration and process control in general[1-4].

In order to establish international/national compatibility, uniformity and affirmation of measurement results, considerable efforts are being made globally so that the 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 comparison of the measurement results carried out on the same artifact. This is also a requirement under Mutual Recognition Arrangement (MRA) to participate in the proficiency testing (PT) and establish the technical competence. Thus, National Accreditation Board for Testing & Calibration Laboratories (NABL) conducts PT among the NABL accredited calibration laboratories in India through the National Metrology Institute (NMI) of India i.e., National Physical Laboratory (NPLI), New Delhi which has acted as a Reference laboratory.

PT program, designated as NABL-Pressure-PT003, is the first inter-laboratory comparison in India in the hydraulic pressure region (5-70 MPa or 50-700 bar) using pressure dial gauge as an artifact. Total 11 laboratories, nine NABL accredited and two others, pressure calibration laboratories, having measurement

*Author for correspondence
Tel: 91-11-25746270; Fax: 91-11-25752678
E-mail: syadav@mail.nplindia.ernet.in

capabilities coarse (< 0.25 % of full-scale pressure), were covered in this PT. The primary objective of organizing PT is to assess the laboratory's technical competence to perform measurements. It supplements laboratory's own quality control procedures by providing additional external audit and also provides objective evidences that a laboratory is competent enough and can achieve the level of uncertainty for which accreditation is granted. External quality control provides important comparisons to determine the uncertainty of participating laboratory testing procedures. Document NABL-162 (2001)[5], describes the administrative procedures and operation of PT to be followed by NABL as well as all participating laboratories.

Methodology

The PT programme, designed as per guidelines stipulated in NABL-162[5], ISO/IEC Guide 43[6] and ISO/IEC 17025[7], includes selection and procurement of the proper artifact, preparation and circulation of the technical protocol (TP), selection of pressure points for comparison, finalization of circulation programme of the artifact, coordination of movement of the artifact at different participating institutes, characterization of the artifact at the beginning and end of the programme at NPL, New Delhi for establishing the stability of the calibration data, compilation of measurement results and data analysis. Nine accredited laboratories, participated in this PT were selected from the directory of NABL accredited laboratories[8].

Selection and Procurement of the Artifact

From the questionnaire survey of responses received from participants, it was decided that a high precision pressure dial gauge is the best option to be used as an artifact. The artifact used for the measurements is a high precision Pressure Dial Gauge, Serial No.- CM42041, make-HEISE, USA.

Preparation of Technical Protocol (TP)

The detailed TP was prepared highlighting all necessary requirements, calibration procedure and guidelines for the circulation of the artifact. TP and circulation programme are integral part of this paper. A copy of the 'TP' was provided to all participating laboratories before arrival of artifact in their organization. Laboratories were asked to ensure that the various instructions in the TP were followed carefully, completely and implemented as instructed.

Selection of Measurement Points

Selection of the measurement points is an important aspect of PT programme. The entire measurement pressure range (5 - 70 MPa) was divided into 14 measurement points (5, 10, 15, 20, 25, 30, 35, 40, 45, 50, 55, 60, 65 and 70 MPa).

Circulation and Movement of the Artifact

All the participants were advised to complete the measurements in two weeks and dispatch the artifact to next participant within next two weeks. The whole circulation programme was completed in two loops (Fig. 1). There was no major technical problem, fault, snag or difficulty reported by any of the participant.

Characterization of the Artifact and Assigning Reference Values

Characterization of the artifact was performed by direct comparison method[1-4] against the national hydraulic secondary pressure standard, designated as NPL200MPA, first at start of the programme during February, 2003, second in middle during June, 2003 and finally at the end of programme during March, 2004. The traceability of NPL200MPA is established by cross-floating it against national primary pressure standard[9-10], designated as NPL1-H1 and its measurement uncertainty is estimated as 61 x 10^{-6} x P at a coverage factor k = 1. NPL200MPA has also participated in the recently concluded bilateral comparison with NIST, USA[11]. NPLI results agree well within 1.0 x 10^{-5} with NIST, USA and are also well within claimed measurement standard uncertainty of 40 x 10^{-6}. Fig. 2 depicts the complete traceability tree of the NPL200MPA[11-16].

Before calibration, both instruments (NPL100MPN and artifact) were leveled using leveling screws and sprit level. The necessary weights were placed on the carrier of the NPL200MPA and adjusted as per the values of pressure generated by the artifact. This is repeated several times so that the error due to this adjustment of the weights is minimized. Sufficient time (10 min) was provided between two successive observations so that both systems are in complete equilibrium. At this position, there was no pressure drop in the connecting line and consequently no movement of fluid. This procedure was repeated for 14 pressure points (5, 10, 15, 20, 25, 30, 35, 40, 45, 50, 55, 60, 65 and 70 MPa), and observations were repeated six times, (3 times, increasing order; 3 times, decreasing order), for each pressure point and the values of pressure generated, their repeatability and expanded uncertainty were computed using computer

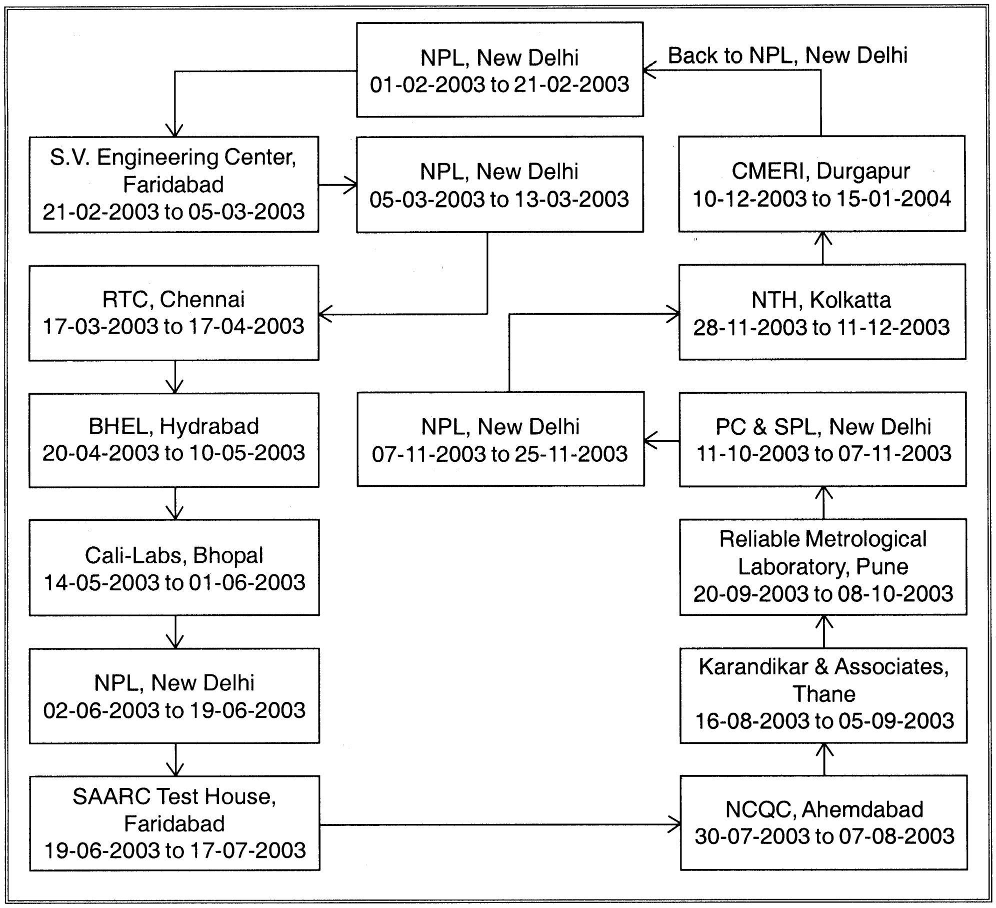

Fig. 1— Circulation and movement of the artifact during comparison. Period shown herein is the actual period for which the artifact remained with the participating laboratory

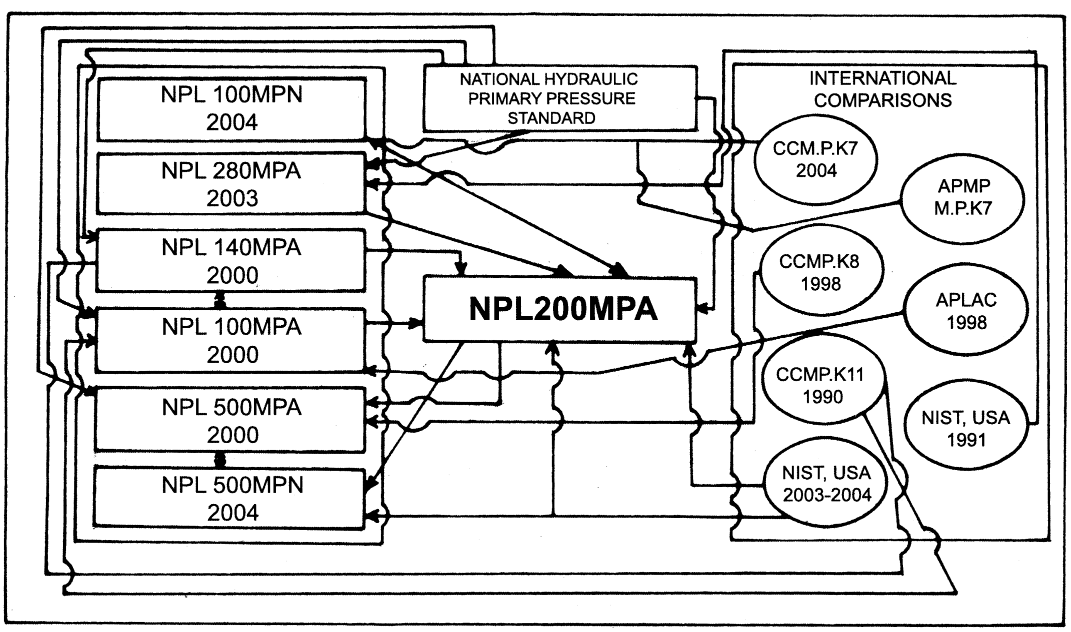

Fig. 2 — Traceability tree for the NPL200MPA, the secondary hydraulic pressure standard used for the chracterisation of the artifact

Table 1 — Details of metrological characteristics of the artifact and assignment of reference values
(All the values reported here are at g_{NPL} = 9.7912393 m/s^2 and reference temperature of T_r = 23 °C)

Nominal pressure MPa	Pressure MPa p_1 Feb. 2003	Pressure MPa P_2 June 2003	Pressure MPa P_3 March 2004	Average pressure MPa p Reference values	Standard deviations of average pressure MPa	Deviations from average values Feb. 2003 MPa	Deviations from average values June 2003 MPa	Deviations from average values March 2003 MPa	Uncertainty evaluated through Type A method MPa	Uncertainty evaluated through stability of the artifact MPa
5	4.949426	4.996193	5.012876	4.98617	0.0329	-0.0367	0.0100	0.0267	0.0190	0.0333
10	9.921606	9.975033	9.982282	9.95964	0.0331	-0.0380	0.0154	0.0226	0.0191	
15	14.91535	14.97045	14.99425	14.96002	0.0405	-0.0447	0.0104	0.0342	0.0234	
20	19.9224	19.96418	20.00185	19.96281	0.0397	-0.0404	0.0014	0.0390	0.0229	
25	24.92773	24.96452	24.98875	24.96033	0.0307	-0.0326	0.0042	0.0284	0.0177	
30	29.91639	29.95484	29.9653	29.94551	0.0258	-0.0291	0.0093	0.0198	0.0149	
35	34.93328	34.97005	34.98374	34.96236	0.0261	-0.0291	0.0077	0.0214	0.0151	
40	39.92852	39.96195	39.96821	39.95289	0.0213	-0.0244	0.0091	0.0153	0.0123	
45	44.91039	44.9365	44.95494	44.93394	0.0224	-0.0236	0.0026	0.0210	0.0129	
50	49.89391	49.92566	49.93705	49.91887	0.0224	-0.0250	0.0068	0.0182	0.0129	
55	54.88569	54.90901	54.90865	54.90112	0.0134	-0.0154	0.0079	0.0075	0.0077	
60	59.87578	59.9025	59.92523	59.90117	0.0248	-0.0254	0.0013	0.0241	0.0143	
65	64.85582	64.8975	64.90313	64.88548	0.0258	-0.0297	0.0120	0.0176	0.0149	
70	69.84251	69.91412	69.94127	69.89930	0.0510	-0.0568	0.0148	0.0420	0.0295	

softwares developed for this purpose[17-18]. The pressure measured by NPL200MPA was calculated using the following equation:

$$p = \frac{\Sigma_i m_i . g_{NPL}(1 - \rho_{air}/\rho_{mi}) + \gamma C}{A_0(1+\lambda s P_n)\,[1+(\alpha_c+\alpha_p)\,(T-T_r)]} \pm \Delta p \qquad \ldots(1)$$

where, m_i, mass of the standard weight; g_{NPL}, local acceleration of gravity; ρ_{air}, density of the air at temperature, barometric pressure and humidity prevailing in the laboratory; ρ_{mi}, density of the i^{th} weight of the standard; γ, surface tension of the pressure transmitting fluid; C, circumference of the standard piston where it emerges from the fluid; A_0, effective area of the standard piston-cylinder assembly at zero pressure; α_c & α_p, thermal expansion coefficients of standard cylinder and piston material; T, temperature of the standard piston-cylinder assembly; T_r, temperature at which A_0 is referred; λ_s, pressure distortion coefficient of the effective area for the standard; and Δp, is the head correction in terms of pressure. The head correction term $\Delta p = [(\rho_f - \rho_{air}) . g_{NPL} . H]$, where H is the difference in height between the reference levels of the standard and the artifact and (ρ_f) is the density of the transmitting fluid.

From the details of the pressure measured (p) and their measurement uncertainties (Table 1) for all the three successive calibrations performed (February 2003, June 2003, March 2004), reference values of pressure were measured as the arithmetic mean of data obtained during these calibrations. The detailed uncertainty budget was prepared for measurements performed on the artifact (Table 2).

The values of measured pressures (p_1, p_2, p_3), were determined using Eq. (1) for three successive calibrations. The reference values, p, are the arithmetic mean of all the three values of measured

Table 2 — Uncertainty budget of the artifact at maximum pressure of 70 MPa and at $T_r = 23^{o}$ C

Source of uncertainty X_i	Estimates X_i Mpa	Limits $\pm \Delta X_i$ Mpa	Probability distribution – Type A or Type B factor	Standard uncertainty $U(X_i)$ Mpa	Sensitivity coefficient	Uncertainty contribution $U_i(Y)$ Mpa	Degree of freedom (υ_F)
Uncertainty of the Standard u_{B1}	70	0.0047	Normal – Type B	0.0047	1	0.0047	∞
Uncertainty due to Resolution of the Artifact u_{B2}	0.1	0.05	Rectangular – Type B/√3	0.029	1	0.03	∞
Repeatability in the First Calibration (Maximum) u_{A1}	0.023	0.023	Normal – Type A/√n	0.014	1	0.02	5
Repeatability in the Second Calibration (Maximum) u_{A2}	0.026	0.026	Normal – Type A/√n	0.015	1	0.02	5
Repeatability in the Third Calibration (Maximum) u_{A3}	0.0932	0.0932	Normal – Type A/√n	0.04168	1	0.042	5
Standard Deviation of Three Calibrations u_{A4}	0.05102	0.05102	Normal – Type A/√n	0.02984	1	0.03	2
Uncertainty due to Stability (Maximum Deviation from the Reference Value) u_{A5}	0.057	0.057	Normal – Type B/√3	0.0333	1	0.04	2
$u_c(P)$			$k = 1$			0.08	19
EXPANDED UNCERTAINTY			$k = 2.14$			0.17	

The expanded uncertainty associated with pressure measurements is 0.17 MPa.
The relative expanded uncertainty associated with pressure measurements is 2.5×10^{-3}.

pressures p_1, p_2 and p_3 for individual measurement point throughout the entire pressure scale. In order to study the behavior and stability of the artifact, calibration factor (C_f) of the artifact was plotted as a function of measured pressure [Fig. 3(a)] and the relative deviations of the measured pressures p_1, p_2 and p_3 from the reference values, p were plotted as a function of p [Fig. 3(b)]. The calibration factor (C_f) was determined as follows:

$$C_f = \frac{p_g}{p_S} \qquad \ldots(2)$$

where, p_g is the reading of the artifact and p_S is corresponding pressure measured by the standard during calibration. The artifact behaved almost in similar fashion during all the three calibrations except slightly lower pressure point of 10 MPa, which is obvious below 10 percent of the full scale pressure of the artifact [Fig. 3(a)]. The relative deviations (full scale) of the measured pressures p_1, p_2 and p_3 from the reference values, p were found well below 0.08 percent [Fig. 3(b)] which is well within the manufacturer specifications of 0.1 percent and NPLI estimated expanded uncertainty (0.25 %). This concludes that the artifact remained stable during the whole PT programme.

Participants

Finally, 12 laboratories participated in the PT including reference laboratory, NPLI, New Delhi. To maintain confidentiality in results, each participating laboratory was assigned a random code number, which to the reference laboratory, NPLI, New Delhi in the present case, is '1'.

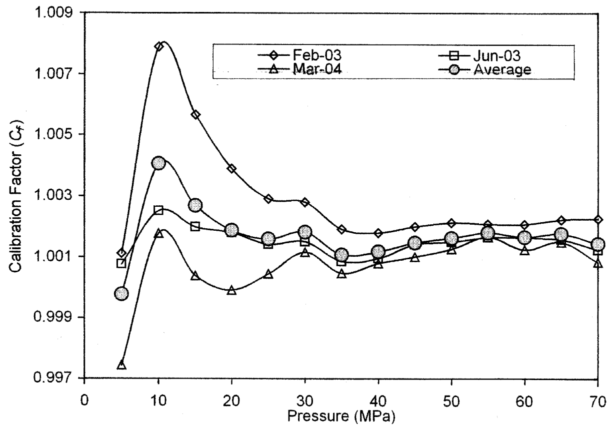

Fig. 3(a) — The Calibration Factor (C_f) and its average values plotted as a function of applied pressure p for all the three successive calibrations

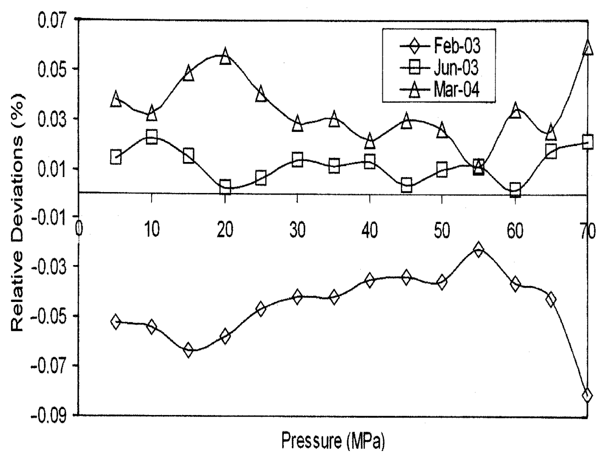

Fig. 3(b) — Relative deviations (full scale) of the measured pressures p_1, p_2 and p_3 from the reference values p for all the three successive calibrations

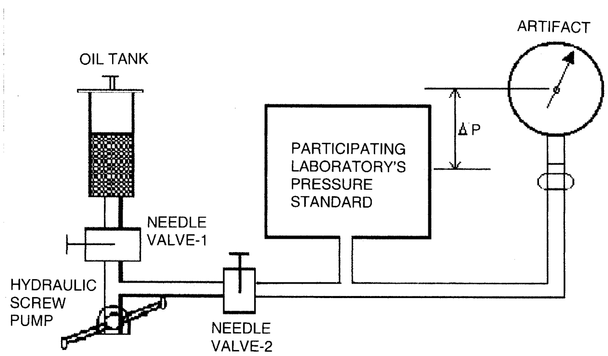

Fig. 4 — Experimental setup for the measurement using pressure dial gauge as an artifact

Experimental Setup and Calibration Procedure

All the laboratories were advised to install the experimental set-up (Fig. 4) and asked to place their laboratory standard and artifact on strong rigid table in calibration room, preferably the stainless steel sheet (thickness, 15 mm or more) as the top of the working table to isolate the vibrations.

Laboratories were requested to clean standard and artifact with soft cloth or tissue paper or cotton. Check the free rotation of screw hydraulic pump handle and valves. Pour a clean mineral oil supplied with the artifact in oil reservoir. Open valve-1 and close valve-2 and turn screw pump handle anticlockwise fully for sucking transmitting fluid from the oil reservoir. Close valve-1 and open valve-2. Turn the screw pump handle clockwise to create and transmit generated pressure in to the standard gauge and the artifact. Level both the standard and the artifact with the help of sprit leveler. After leveling the instruments, experimental setup thus would be ready for calibration of the artifact.

The calibration of the artifact starts with leak testing, zero adjustment and the selection of a reference or datum level. For leak testing, laboratories were requested to pressurize standard and the artifact up to 700 bar with the help of hydraulic screw pump and needle valves and wait for at least 10 min and then release pressure slowly to zero. Laboratories were asked to repeat this process at least three times to ensure that there are no leaks in the system. In this way, compressibility of transmitting oil, packing of valves, pump plunger and O-ring seals are stabilized to reach an optimum level.

Zero adjustment of the artifact is then performed using the zero adjustment knob of the artifact. Participating laboratories were also requested to ensure zero adjustment of their standard (in case of digital pressure instrument or pressure dial gauge). In case zero adjustment knobs are not provided with standards, laboratories were asked to record the initial bias in the measurements and apply necessary correction at the appropriate level.

Selection of appropriate and precise reference or datum plane is very important for applying hydrostatic head correction. Usually, reference or datum plane is marked on the standard or noted in the operation manuals. If such information is not available, centre point of elastic element is considered the reference or datum plane. Needle setting of the artifact is also one of the important points during measurements. The normal practice is to check the reflection of the needle from the mirror. In order to minimize the parallax error, the best position for

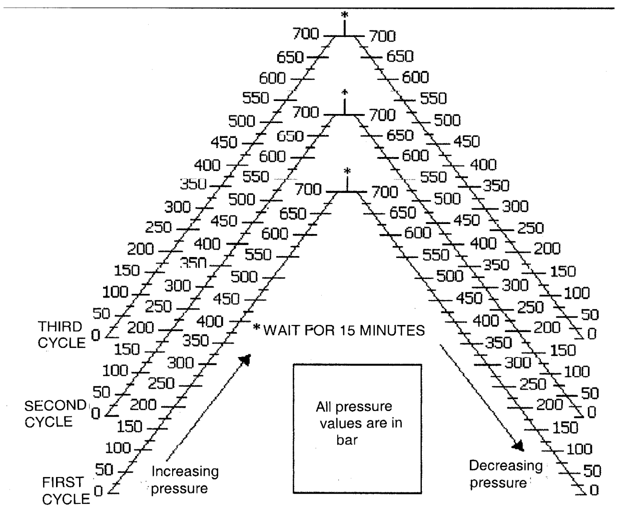

Fig. 5 — Sequence of measurements taken

measurement would be when the reflected image coincides with real object i.e., needle in the present case. Laboratories were advised to follow the same eye estimation uniformly for all the pressure points.

In this way, the system was ready to perform calibration (Fig. 5). The full-scale pressure (measurement range in the present case) of 700 bar was then divided into 14 equally spaced pressure points (50, 100, 150, 200, 250, 300, 350, 400, 450, 500, 550, 600, 650 and 700 bar). The needle of the artifact was then brought to a first measurement point by pressurizing the system and the corresponding value of the pressure measured by the standard was recorded after applying all corrections (temperature correction, hydrostatic head correction and unit conversion). Laboratories were advised to record the corrected pressure measured by the standard only in bar or MPa. Subsequently, needle of the artifact was fixed to next pressure point and the pressure measured by the standard was recorded. This process was repeated till full-scale pressure of 700 bar is achieved. Laboratories were asked to maintain sufficient time (10 min) between two successive observations to allow system to reach a state of thermal equilibrium. It was also suggested to wait for at least 15 min after reaching full-scale pressure before the observations are repeated in the decreasing order of pressure till pressure reaches to zero. Laboratories were requested to record at least 28 observations, 14 each in the order of increasing and decreasing pressures, to perform one pressure cycle and then to repeat the measurements for at least 3 pressure cycles to make the total number of 84 observations.

All the participants were advised to apply the temperature and head corrections carefully before submitting the results. They were requested to correct the values of the measured pressure for 23 °C using thermal expansion coefficient of the piston – cylinder assembly (if dead weight tester is used as standard) or elastic element (if pressure dial gauge or digital calibrator is used as standard) using standard equations. The head correction term $\Delta p=[(\rho_f-\rho_{air}).g_L H]$, is a very important correction term and contributes significantly below 100 MPa, where H is the difference in height between the reference levels of the standard and the artifact, g_L is the local acceleration of gravity (m/sec^2) and ρ_f is the density (kg/m^3) of the pressure transmitting fluid used in the measurements.

Laboratories 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 on uncertainty following Type A and Type B methods of evaluation[19-20]. Each participating laboratory was requested to prepare an uncertainty budget at maximum pressure, considering all Type A and Type B uncertainty components.

Results and Data Analysis

All the laboratories were advised to submit their measurement results on specially designed proformas. The values included measured pressure, acceleration of local gravity, reference temperature, and measurement uncertainty estimated at maximum pressure (Table 3). Before, compiling and comparing the results, it is necessary to apply certain corrections in the values reported by the laboratories to make them comparable. The following corrections were applied:

Gravity Correction

The measured pressure values reported by the laboratories are corrected for g_{NPL} = 9.7912393 m/s^2 (acceleration of gravity at NPL, New Delhi, India) using the following relationship:

$$P' = p_{\mathrm{rep}}*(g_{\mathrm{NPL}}/g_{\mathrm{LAB}}) \qquad \ldots(3)$$

where p' and p_{rep} are the values of corrected and reported pressure, respectively and g_{LAB} is the value of acceleration of gravity reported by the laboratory.

Estimation of Measurement Uncertainty

The expanded uncertainty reported by the laboratory is converted into relative uncertainty and

Table 3 — Details of the reference values, measured pressure (p_{rep}) and other metrological characteristics of the laboratories standards

Nominal pressure bar	Laboratory code 1	2	3	4	5	6	7	8	9	10	11	12
50	49.862	51.07001	49.979	49.7652	49.84688	49.736	49.8	50.16	50.292	49.8172	50.2457	50.30922
100	99.596	101.07226	100.4398	99.4997	99.43297	99.63	99.1665	99.6	101.208	99.9965	100.313	100.35193
150	149.6	150.11421	150.5488	149.5277	149.61123	149.267	148.1335	149.17	150.708	150.0124	150.7543	150.56278
200	199.628	200.11686	200.446	199.4252	199.4876	198.841	198.3335	199.45	200.917	200.0937	201.0794	200.74485
250	249.603	250.29653	250.5467	249.5184	250.04277	248.123	249.0335	248.86	250.875	250.2403	251.0355	250.73248
300	299.455	299.41081	300.4117	299.3834	299.90664	297.861	299.1	298.63	300.792	300.3215	301.0524	300.73227
350	349.624	349.35522	350.3753	349.4113	350.25814	348.192	350.4335	349.36	350.292	350.3374	351.054	350.79599
400	399.529	400.33618	400.5457	399.3415	400.36049	398.366	399.5	399.24	400.875	400.386	401.0254	400.89183
450	449.339	449.35896	450.2712	449.0433	450.11093	447.86	449.7	448.14	450.542	450.6143	451.1079	450.75248
500	499.189	499.12706	499.9159	499.0387	500.1169	498.145	500.0335	498.67	500.833	500.7446	500.7356	500.93377
550	549.011	549.20036	549.8757	548.871	550.17627	547.967	549.867	547.92	550.792	551.4332	550.5604	550.8276
600	599.012	587.48096	599.7508	598.6707	600.30361	598.083	599.3665		601.458		600.7793	600.85489
650	648.855		649.6874	648.6009	650.30592	647.622	649.8		650.708		650.6446	651.01499
700	698.993			698.531	700.46988	697.721	699.533		702.042		700.7677	701.17761
g (m/s^2)	9.7912393	9.7909591	9.787177	9.78383	9.7828872	9.7909591	9.7881843	9.78643	9.78380	9.7914503	9.787917	9.78869
Ref. Temp. (°C)	23	23	23	21.8-24.2	23	23.4	23	23	23	23	23	22.97
$u(P_{rep})$ x 10^{-6} (at k = 2)	2500 or 0.25 %	1567 or 0.16 %	1543 or 0.16 %	366 or 0.04 %	2851 or 0.29 %	4061 or 0.41 %	1500 or 0.15 %	2291 or 0.23 %	1864 or 0.19 %	2000 or 0.20 %	215 or 0.03 %	1689 or 0.17 %
Traceability	NPLI-H1 NIST, USA	NCCBM, Ballabhgarh	NPLI, New Delhi	Measure Techniques, Chennai	Nagman, Chennai	NCCBM, Ballabhgarh	IDEMI, Mumbai	ERTL, Mumbai	IDEMI, Mumbai	NCCBM, Ballabhgarh	*	*
Best Measurement Capabilities as per NABL Doc. 502, 2002	-	1.0 % to 0.5 %	1.0 %	0.25 %	0.3 %	0.02 to 1.6 kg/cm^2 or 0.3 %	0.6 %	0.6 %	0.4 % to 0.15 %	3.1 % to 0.4 %	#	#

* These laboratories do not provide traceability.

\# These laboratories are preparing for the accreditation.

then final uncertainty is computed using the following formula:

$$U(p') = [\{U(p_{\text{rep}})/p_{\text{rep}}\}/10^{-6}]\times p' \quad \ldots(4)$$

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, assuming it as the maximum measurement uncertainty.

Estimation of Normalized Error (E_n)

In accordance with international practice, measurement performance was assessed on the basis of Normalized Error Value (E_n) for each measurement. E_n values are estimated for each participant at each pressure as per reported guidelines[5-6,21]:

$$E_n Value = \frac{p_{LAB} - p_{\mathrm{Re}\,f}}{\sqrt{\{U(p_m)\}^2 + \{U(p_{\mathrm{Re}\,f})\}^2}} \quad \ldots(5)$$

where $p_{LAB} = p'$ is the participant's measured pressure value, $p_{Ref} = p$ is the calculated reference value, $U(p')$ 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 a coverage factor $k = 2$.

Corrected pressure (p') for gravity (g_{NPL}) with relative deviations of measured pressure (p') of each participant from reference value (p) was recorded (Table 4). Graphs were plotted for results (Figs 6-21). Calculated E_n values at individual pressure points were summarized (Table 5).

Discussions

An E_n value (<1) indicates agreement within the combined uncertainties for the results to be internationally acceptable. An E_n number between –1 and +1 indicates an acceptable degree of compatibility between the laboratory's result and the reference value when the quoted uncertainties are taken into account. E_n number outside –1 and +1 range is unacceptable and requires immediate investigation and corrective action by laboratory concerned. In general, performance of the laboratory is considered satisfactory if absolute value of normalized error E_n is $\leq$ 1. The data (Tables 4 & 5) reveals that there are total 145 measurement results. Measurement results of only 2 laboratories (Code No. 4 & 5) are well within acceptable limits of normalized error over the entire pressure range (5-70 MPa). However, measurement results of the laboratories with Code No 6, 8 and 10 are also quite good having E_n values > 1 only at one or two pressure points. E_n value of 86 measurement results out of total 145, is $\leq$ 1, (59.31 %). These results are acceptable. E_n values of the laboratory referred as Code No 9, 11 and 12 are beyond the acceptable limit throughout the entire pressure scale except one pressure point of 35 MPa for the laboratory with Code No 9. The larger the absolute value of E_n number, bigger the problem. 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 need further investigations at the part of the laboratory.

The graphical representations (Figs 6-19) give the agreement between participating laboratories and the reference laboratory. The deviations lying within the uncertainty band of the reference laboratory is an indication of satisfactory results without any bias in the measurements. The deviations between laboratories values and reference values at 64 measurement points out of the total 145 are almost well within the uncertainty band of the reference values (44.14 %).

Further, only 86 measurements results (59.31%) fall within their combined uncertainty band. This clearly shows the under estimation of measurement uncertainty by most of the laboratories. The main reasons for the deviations in the results are due to errors in laboratory's measuring instrument or estimation/measurement of local acceleration of gravity, the error in applying the temperature and head corrections and the under estimation of measurement uncertainty. Three laboratories with Code Nos – 4, 6 and 12 reported their measurement results at the reference temperature of <> 23 °C. However, they were clearly instructed to correct their values at 23 °C. Two laboratories with Code Nos 2 and 12 have also reported their values at 0 pressure point. This implies that they might have not adjusted the zero values at the artifact using 'Zero Adjustment Knob' as suggested in the TP. Laboratories would be able to rectify the problems by a review of their uncertainty calculations and other systematic affects as mentioned above.

It is clear from the data (Tables 4 & 5) and graphs (Figs 20 & 21) that the relative deviations of 94 measurement points out of the total 117 (80.34 %) are

Table 4 — Details of the corrected pressure (p') for g_{NPL} and relative deviations of the measured pressure from reference values (p)

Nominal pressure MPa	Laboratory Code											
	1	2	3	4	5	6	7	8	9	10	11	12
	p (MPa)	p'(MPa)	p'(MPa)	p'(MPa)	p'(MPa)	p'(MPa)	p'(MPa)	p'(MPa)	p'(MPa)	p'(MPa)	p'(MPa)	p'(MPa)
7	4.9862	5.1071	5.0000	4.9803	4.9889	4.9737	4.9816	5.0185	5.0330	4.9816	5.0263	5.0322
10	9.9596	10.1075	10.0481	9.9575	9.9518	9.9633	9.9197	9.9649	10.1285	9.9994	10.0347	10.0378
15	14.9600	15.0119	15.0611	14.9641	14.9739	14.9271	14.8180	14.9243	15.0823	15.0009	15.0805	15.0602
20	19.9628	20.0123	20.0529	19.9576	19.9658	19.8847	19.8395	19.9548	20.1070	20.0089	20.1148	20.0797
25	24.9603	25.0304	25.0651	24.9707	25.0256	24.8130	24.9111	24.8982	25.1066	25.0235	25.1121	25.0798
30	29.9455	29.9419	30.0536	29.9610	30.0163	29.7870	29.9193	29.8777	30.1021	30.0315	30.1155	30.0811
35	34.9624	34.9365	35.0521	34.9676	35.0557	34.8202	35.0543	34.9532	35.0558	35.0330	35.1173	35.0887
40	39.9529	40.0348	40.0712	39.9644	40.0702	39.8377	39.9625	39.9436	40.1180	40.0377	40.1162	40.0996
45	44.9339	44.9372	45.0458	44.9383	45.0495	44.7873	44.9840	44.8360	45.0885	45.0605	45.1261	45.0870
50	49.9189	49.9141	50.0123	49.9417	50.0544	49.8159	50.0190	49.8915	50.1214	50.0734	50.0906	50.1064
55	54.9011	54.9216	55.0104	54.9287	55.0646	54.7983	55.0039	54.8189	55.1211	55.1421	55.0747	55.0971
60	59.9012	58.7498	60.0000	59.9124	60.0816	59.8100	59.9554	-	60.1915	-	60.0983	60.1011
65	64.8855	-	64.9957	64.9092	65.0861	64.7641	65.0003	-	65.1203	-	65.0865	65.1185
70	69.8993	-	-	69.9060	70.1068	69.7741	69.9751	-	70.2576	-	70.1006	70.1360

—*Contd...*

Table 4 — Details of the corrected pressure (p') for g_{NPL} and relative deviations of the measured pressure from reference values (p) —(*Contd*)

Nominal pressure MPa	Laboratory Code											
	1	2	3	4	5	6	7	8	9	10	11	12
	p (MPa)	p'(MPa)	p'(MPa)	p'(MPa)	p'(MPa)	p'(MPa)	p'(MPa)	p'(MPa)	p'(MPa)	p'(MPa)	p'(MPa)	p'(MPa)
	Relative deviations of measured pressure from reference values (%)											
7	-	2.43	0.28	-0.12	0.06	-0.25	-0.09	0.65	0.94	-0.09	0.80	0.92
10	-	1.49	0.89	-0.02	-0.08	0.04	-0.40	0.05	1.70	0.40	0.75	0.79
15	-	0.35	0.68	0.03	0.09	-0.22	-0.95	-0.24	0.82	0.27	0.81	0.67
20	-	0.25	0.45	-0.03	0.01	-0.39	-0.62	-0.04	0.72	0.23	0.76	0.59
25	-	0.28	0.42	0.04	0.26	-0.59	-0.20	-0.25	0.59	0.25	0.61	0.48
30	-	-0.01	0.36	0.05	0.24	-0.53	-0.09	-0.23	0.52	0.29	0.57	0.45
35		-0.07	0.26	0.01	0.27	-0.41	0.26	-0.03	0.27	0.20	0.44	0.36
40	-	0.20	0.30	0.03	0.29	-0.29	0.02	-0.02	0.41	0.21	0.41	0.37
45		0.01	0.25	0.01	0.26	-0.33	0.11	-0.22	0.34	0.28	0.43	0.34
50	-	-0.01	0.19	0.05	0.27	-0.21	0.20	-0.05	0.41	0.31	0.34	0.38
55		0.04	0.20	0.05	0.30	-0.19	0.19	-0.15	0.40	0.44	0.32	0.36
60	-	-1.92	0.16	0.02	0.30	-0.15	0.09	-	0.48	-	0.33	0.33
65		-	0.17	0.04	0.31	-0.19	0.18	-	0.36	-	0.31	0.36
70	-	-	-	0.01	0.30	-0.18	0.11	-	0.51	-	0.29	0.34
$u(p_{rep}) \times 10^{-6}$	2500	1567	1543	366	2851	4061	1500	2291	1864	2000	215	1689
(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 %	or 0.19 %	or 0.20 %	or 0.03 %	or 0.17 %

Table 5 — Summary of the normalized error (E_n) of each participant and combined expanded uncertainty estimated during comparison

Nominal pressure MPa	Laboratory code 1	2	3	4	5	6	7	8	9	10	11	12
7	-	8.16	0.94	-0.47	0.15	-0.52	-0.32	1.90	3.00	-0.29	3.20	3.05
10	-	5.01	3.02	-0.08	-0.21	0.08	-1.37	0.16	5.41	1.25	3.01	2.60
15	-	1.17	2.30	0.11	0.24	-0.46	-3.26	-0.70	2.61	0.85	3.21	2.22
20	-	0.84	1.53	-0.10	0.04	-0.82	-2.12	-0.12	2.31	0.72	3.03	1.94
25	-	0.95	1.43	0.17	0.69	-1.24	-0.68	-0.73	1.88	0.79	2.42	1.58
30	-	-0.04	1.23	0.21	0.62	-1.11	-0.30	-0.67	1.67	0.90	2.26	1.50
35	-	-0.25	0.87	0.06	0.70	-0.86	0.90	-0.08	0.86	0.63	1.77	1.20
40	-	0.69	1.01	0.11	0.77	-0.61	0.08	-0.07	1.32	0.66	1.63	1.22
45	-	0.02	0.85	0.04	0.68	-0.69	0.38	-0.64	1.10	0.88	1.70	1.13
50	-	-0.03	0.64	0.18	0.71	-0.43	0.69	-0.16	1.30	0.97	1.37	1.24
55	-	0.13	0.68	0.20	0.78	-0.39	0.64	-0.44	1.28	1.37	1.26	1.18
60	-	-6.55	0.56	0.07	0.79	-0.32	0.31	-	1.55	-	1.31	1.11
65	-	-	0.58	0.14	0.81	-0.39	0.61	-	1.16	-	1.23	1.19
70	-	-	-	0.04	0.78	-0.38	0.37	-	1.64	-	1.15	1.12
Uncertainty Details												
$u(p_{rep}) \times 10^{-6}$ (at k = 2)	2500 or 0.25 %	1567 or 0.16 %	1543 or 0.16 %	366 or 0.04 %	2851 or 0.29 %	4061 or 0.41 %	1500 or 0.15 %	2291 or 0.23 %	1864 or 0.19 %	2000 or 0.20 %	215 or 0.03 %	1689 or 0.17 %
Maximum Relative Combined Uncertainty at k = 2 $U(p') \times 10^{-6}$	2500 or 0.25 %	3000 or 0.3 %	2900 or 0.29 %	2500 or 0.25 %	3800 or 0.38 %	4800 or 0.48 %	2900 or 0.29 %	3400 or 0.34 %	3100 or 0.31 %	3200 or 0.32 %	2500 or 0.25 %	3000 or 0.3 %
Best Measurement Capabilities as per NABL Doc. 502, 2002	-	1.0 % to 0.5 %	1.0 %	0.25 %	0.3 %	0.02 to 1.6 kg/cm^2 or 0.3 %	0.6 %	0.6 %	0.4 % to 0.15 %	3.1 % to 0.4 %	#	#

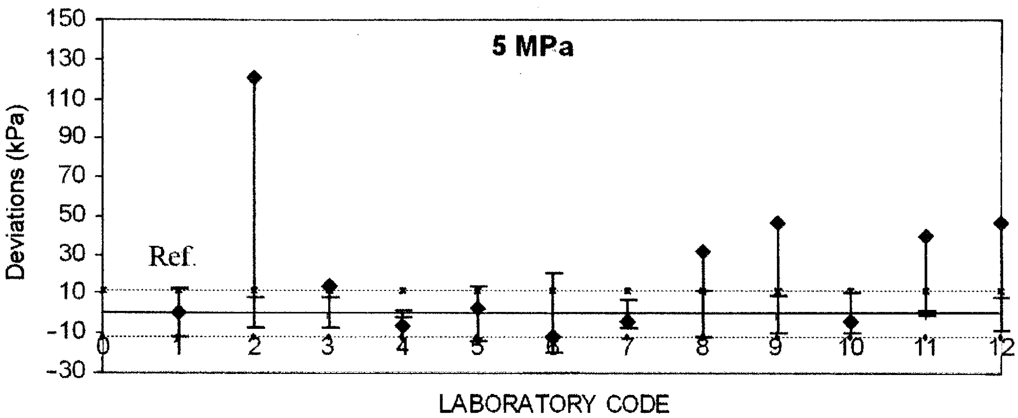

Fig. 6 — Black points indicate the deviation of the measured pressure (p') by the laboratory from the reference value (p) at 5 MPa and error bars shows the estimated reported expanded measurement uncertainty at $k = 2$. The gap between two horizontal dotted lines shows the expanded uncertainty band of the reference value.

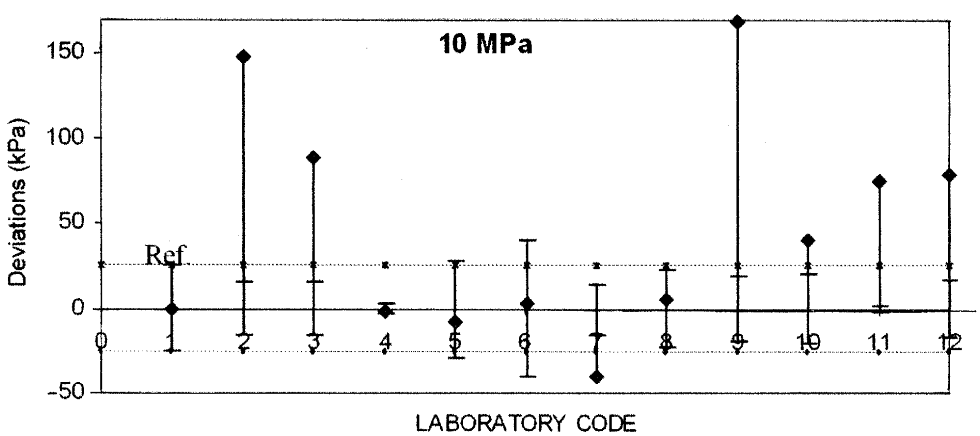

Fig. 7 — Black points indicate the deviation of the measured pressure (p') by the laboratory from the reference value (p) at 10 MPa and error bars shows the estimated reported expanded measurement uncertainty at $k = 2$. The gap between two horizontal dotted lines shows the expanded uncertainty band of the reference value.

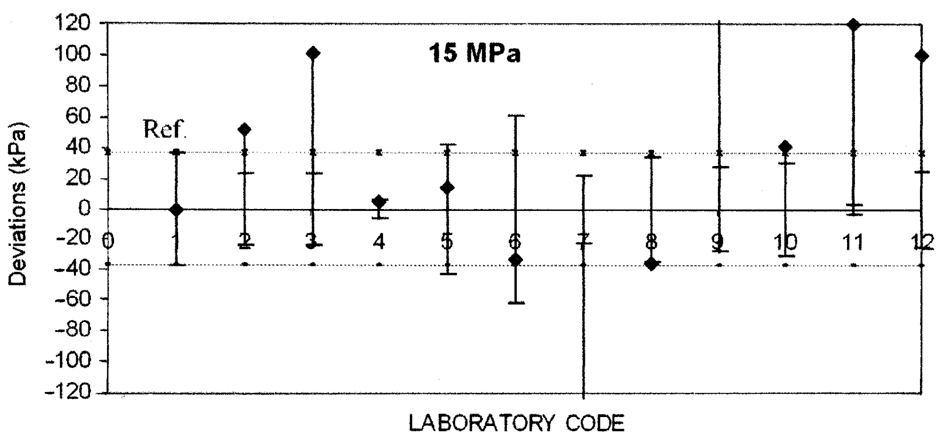

Fig. 8 — Black points indicate the deviation of the measured pressure (p') by the laboratory from the reference value (p) at 15 MPa and error bars shows the estimated reported expanded measurement uncertainty at $k = 2$. The gap between two horizontal dotted lines shows the expanded uncertainty band of the reference value.

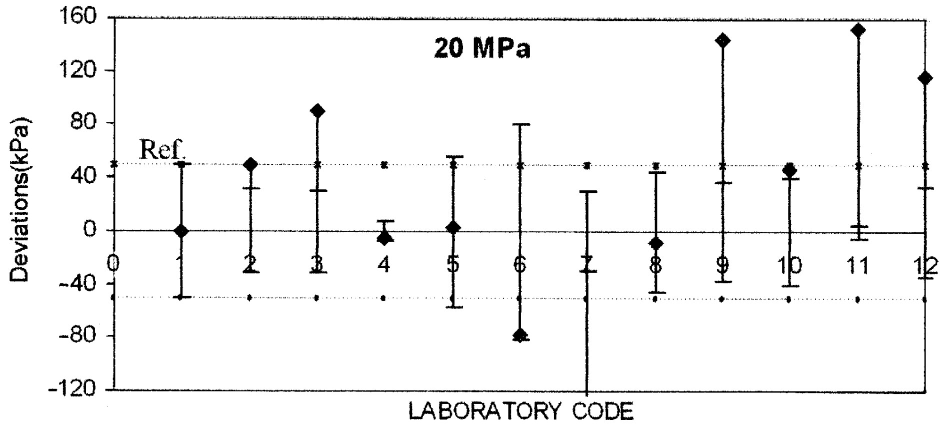

Fig. 9 — Black points indicate the deviation of the measured pressure (p') by the laboratory from the reference value (p) at 20 MPa and error bars shows the estimated reported expanded measurement uncertainty at $k = 2$. The gap between two horizontal dotted lines shows the expanded uncertainty band of the reference value.

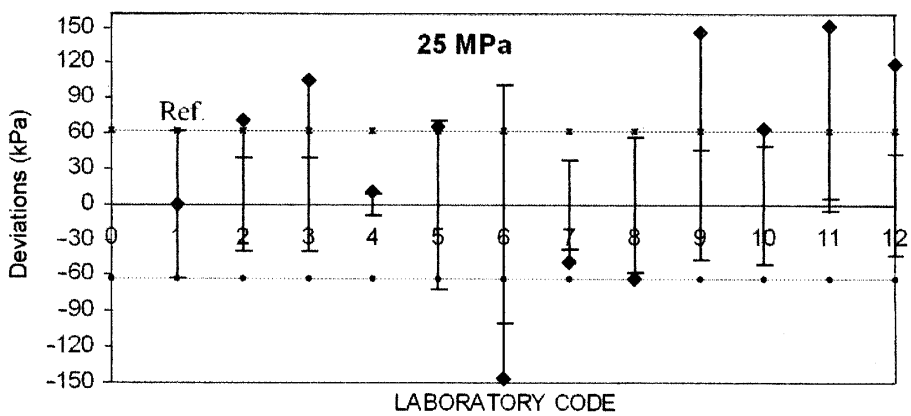

Fig. 10 — Black points indicate the deviation of the measured pressure (p') by the laboratory from the reference value (p) at 25 MPa and error bars shows the estimated reported expanded measurement uncertainty at $k = 2$. The gap between two horizontal dotted lines shows the expanded uncertainty band of the reference value.

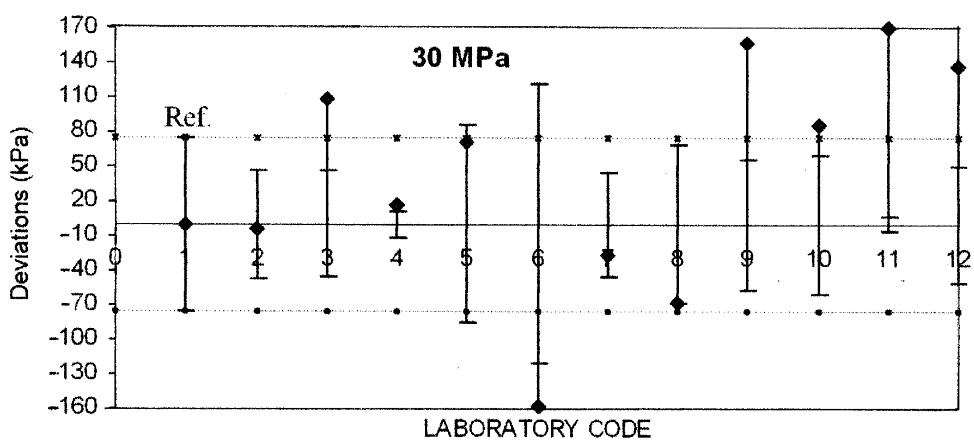

Fig. 11 — Black points indicate the deviation of the measured pressure (p') by the laboratory from the reference value (p) at 30 MPa and error bars shows the estimated reported expanded measurement uncertainty at $k = 2$. The gap between two horizontal dotted lines shows the expanded uncertainty band of the reference value.

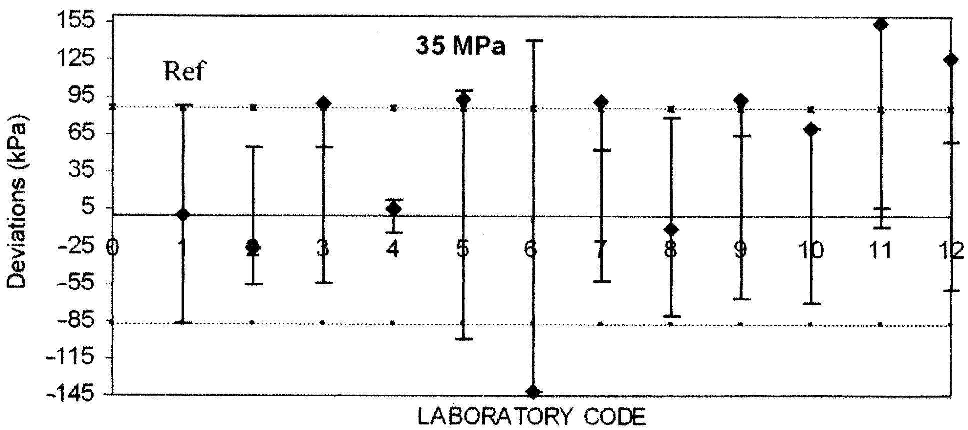

Fig. 12 — Black points indicate the deviation of the measured pressure (p') by the laboratory from the reference value (p) at 35 MPa and error bars shows the estimated reported expanded measurement uncertainty at $k = 2$. The gap between two horizontal dotted lines shows the expanded uncertainty band of the reference value.

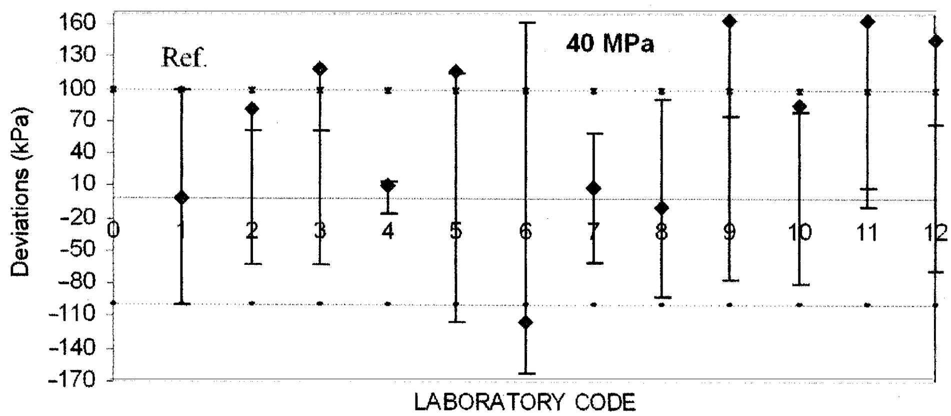

Fig. 13 — Black points indicate the deviation of the measured pressure (p') by the laboratory from the reference value (p) at 40 MPa and error bars shows the estimated reported expanded measurement uncertainty at $k = 2$. The gap between two horizontal dotted lines shows the expanded uncertainty band of the reference value.

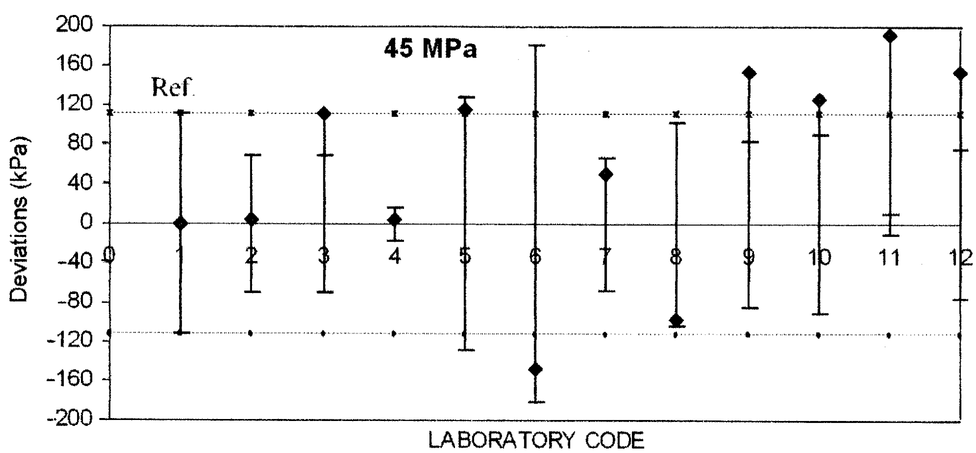

Fig. 14 — Black points indicate the deviation of the measured pressure (p') by the laboratory from the reference value (p) at 45 MPa and error bars shows the estimated reported expanded measurement uncertainty at $k = 2$. The gap between two horizontal dotted lines shows the expanded uncertainty band of the reference value.

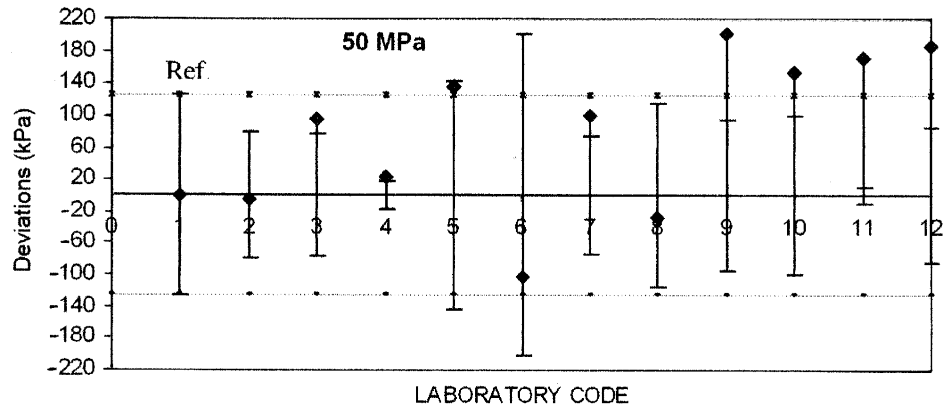

Fig. 15 — Black points indicate the deviation of the measured pressure (p') by the laboratory from the reference value (p) at 50 MPa and error bars shows the estimated reported expanded measurement uncertainty at $k = 2$. The gap between two horizontal dotted lines shows the expanded uncertainty band of the reference value.

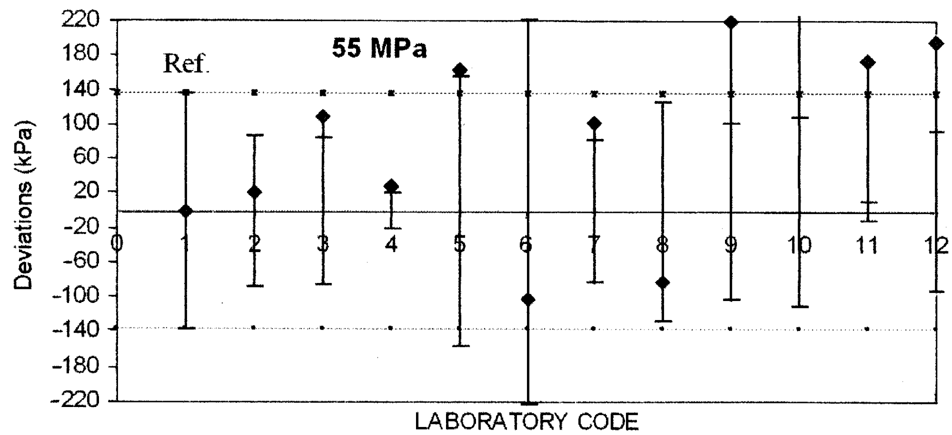

Fig. 16 — Black points indicate the deviation of the measured pressure (p') by the laboratory from the reference value (p) at 55 MPa and error bars shows the estimated reported expanded measurement uncertainty at $k = 2$. The gap between two horizontal dotted lines shows the expanded uncertainty band of the reference value.

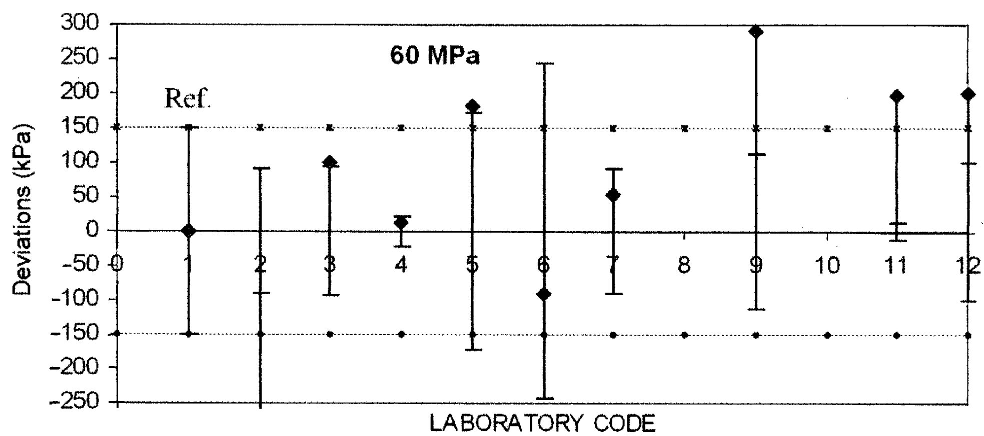

Fig. 17 — Black points indicate the deviation of the measured pressure (p') by the laboratory from the reference value (p) at 60 MPa and error bars shows the estimated reported expanded measurement uncertainty at $k = 2$. The gap between two horizontal dotted lines shows the expanded uncertainty band of the reference value.

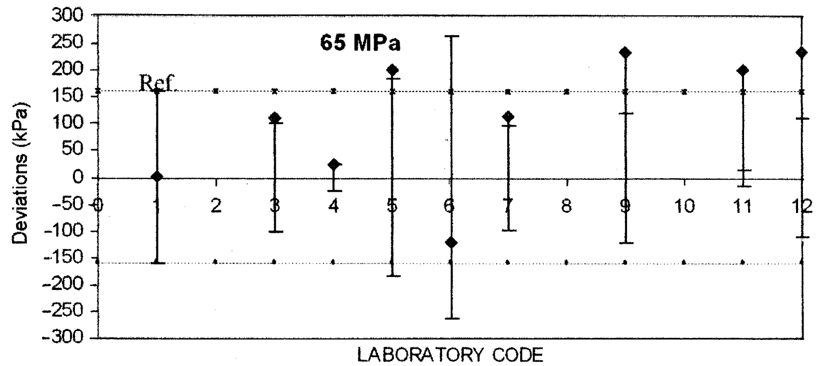

Fig. 18 — Black points indicate the deviation of the measured pressure (p') by the laboratory from the reference value (p) at 65 MPa and error bars shows the estimated reported expanded measurement uncertainty at $k = 2$. The gap between two horizontal dotted lines shows the expanded uncertainty band of the reference value.

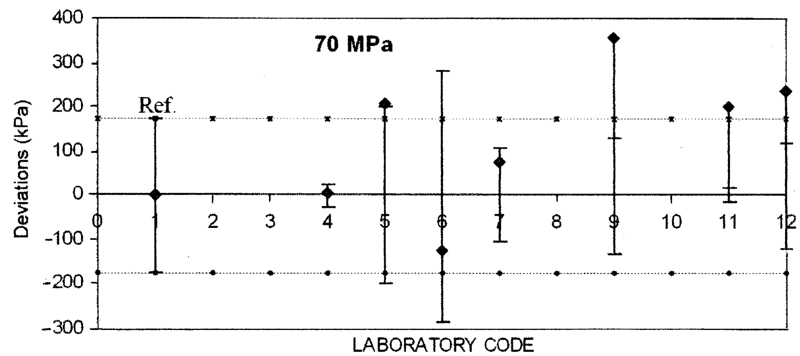

Fig. 19 — Black points indicate the deviation of the measured pressure (p') by the laboratory from the reference value (p) at 70 MPa and error bars shows the estimated reported expanded measurement uncertainty at $k = 2$. The gap between two horizontal dotted lines shows the expanded uncertainty band of the reference value.

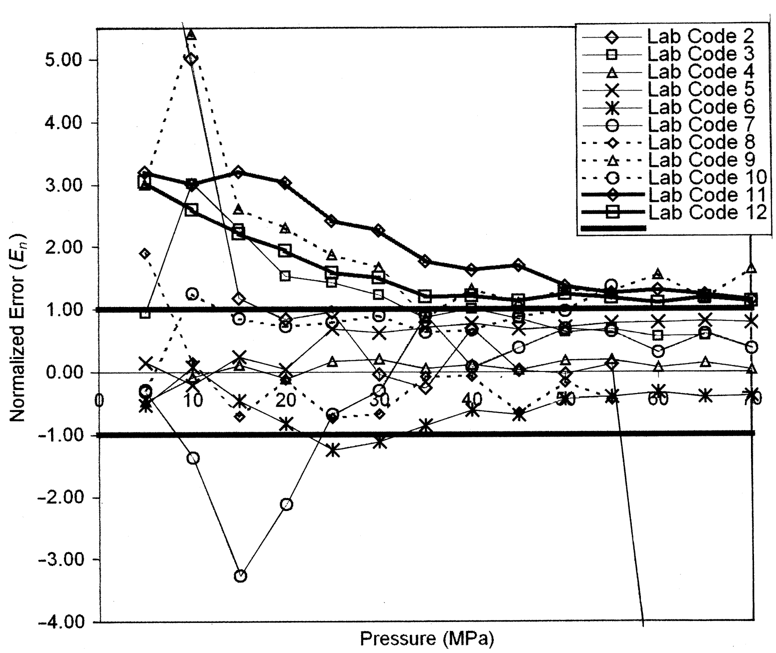

Fig. 20 — The normalized error value (E_n) as a function of measured pressure (p') for each laboratory. The gap between two horizontal dotted lines shows the acceptable limit of the normalized error value.

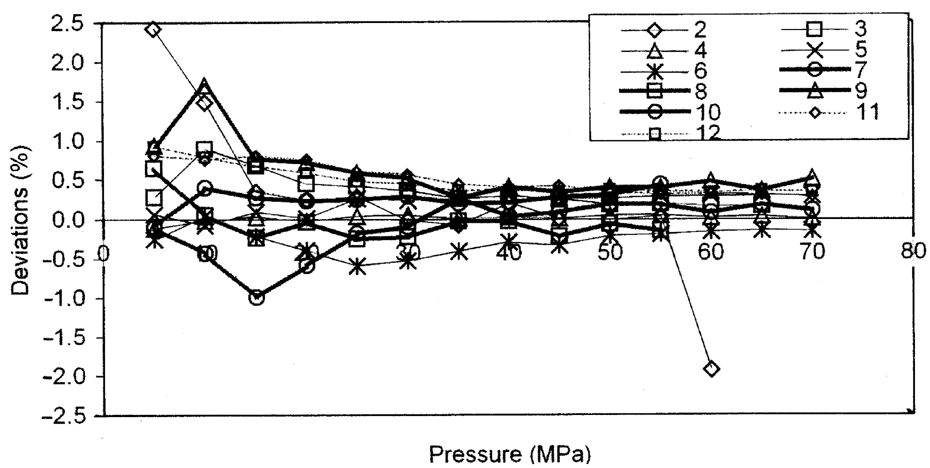

Fig. 21 — Relative deviations of the measured pressure (p') by each laboratory from the reference value (p)

well within their best measurement capabilities as reported in NABL Document 502. However, the relative deviations of two laboratories (Code No – 11 and 12), which are not the part of NABL Document 502, are found to be 0.8 to 0.3 percent and 0.9 to 0.3 percent, respectively.

Conclusions

This interlaboratory comparison programme (PT) was carried out in the pressure range 5 – 70 MPa using pressure dial gauge as an artifact for 11 laboratories, 9 NABL accredited and two others. The comparison was performed at 14 pressure points selected arbitrarily throughout the entire pressure range. PT concludes that out of the total 145 measurement results reported here in this report, 86 (59.31 %) are in agreement with the reference laboratory. The E_n values of only two laboratories are within acceptable limits throughout the entire pressure scale. However, E_n values of three other laboratories are also quite acceptable except one or two pressure points. The E_n values of three laboratories are found beyond acceptable limit throughout the entire pressure scale except one pressure point. The deviations between laboratories values and reference values at 64 measurement points (44.14 %) are almost well within the uncertainty bands of reference values. Total 86 measurements results (59.31 %), fall within their combined uncertainty band. However, 80.34 percent measurement results are found well within their best measurement capabilities reported in NABL Document 502. Since most of the laboratories have under estimated their measurement uncertainties, 40.69 percent measurement results are found out of the combined uncertainty band during this comparison. Overall, results are considered to be reasonably good being the first proficiency testing for all the participating laboratories.

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