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National and Global Status of the High Pressure Measurement and Calibration Facilities

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Rapid and increasing use of high-pressure technology in science and engineering over the last few decades has prompted researchers to look for better instruments and technologies with lower measurement uncertainties in diversified applications wherein pressure is used as one of the most crucial process parameters. The present paper discusses the succinct assessment of high-pressure metrology (HPM). A graphical summary of the various key & supplementary comparisons (K&SC) carried out in pressure metrology along with Calibration and Measurement Capabilities (CMCs) of the various National Metrology Institutes (NMIs) is also included. Also discussed are the overview of the various high-pressure measurement facilities established at CSIR-NPL, India and a summary of the various Proficiency Testing (PT) Programs conducted as reference laboratory by CSIR-NPL for 140 testing and calibration laboratories, accredited by the National Accreditation Board for the Testing and Calibration of Laboratories (NABL), India. The paper also briefly describes the impact of redefined SI unit of mass on the realization of the pressure scale. It is always difficult to find a concise report on all the aspects of a specific measuring parameter, high pressure in the present case, in a single window, therefore, authors believe that the present paper would be highly useful for the students, engineers, researchers, metrologists, industries, accredited laboratories, and other users.

Keywords: Metrology; Pressure; Accuracy; Precision; SI Units; CMCs; KCDB data

1 Introduction

In many disciplines of science and industry, particularly in technological and industrial manufacturing processes, the physical quantity "pressure" is an essential measurand. Researchers and technologists have been working hard to develop simple, cost-effective, and precise methods of measuring pressure to cater the advanced industrial demands for calibrations and measurement standards[1-5]. Pressure measurements are classified as static and dynamic measurements. When there is a molecular motion, it is referred to as static pressure. Often, the motion of the fluid changes with the force exerted to its surroundings and such measurements are called dynamic pressure measurements. Pressure measurement was primarily employed during the first industrial revolution to convert water to steam, but it is now widely used in modern society, with applications in areas as diverse as electricity, gas, optics, aerospace, defense, meteorology, automotive, medical, and safety. The majority of the pressure measurements are made for commercial purposes, as part of R&D and production engineering to assure product quality, dependability, and safety. Pressure measurements are extensively employed in life support apparatus, anesthetic delivery, and sphygmomanometers, *i.e.* blood pressure measuring instruments, in the medical field. This is the reason that the primary pressure standards starting from barometric pressure to the high pressure measuring range are always in focus and need to be established and maintained for providing traceability to pressure measuring instruments [6-10].

A wide range of applications of pressure metrology exists starting from atmospheric pressure, i.e. 100 kPa to a few GPa. Normally, a high-pressure term is employed for pressures more than 100 kPa. However, the majority of the high-pressure applications are limited to few a gigapascals (GPa) [11-13]. Fig. 1 depicts some of such applications of pressure measurements.

The present paper is an attempt to present a concise report on the Primary pressure measurement techniques; Effect of redefined SI unit of mass on the realization of pressure scale; Role, status and capabilities in high pressure measurement; National hydraulic high-pressure standards established at CSIR-NPL and a synopsis of the various proficiency

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testing programs conducted in the high-pressure range.

2 Primary pressure measurement techniques

The accurate and precise measurement of pressure is carried out using different types of primary, secondary and working standards. Through a process of metrological characterization, primary standards define the practical pressure scale to fundamental units of mass, length, temperature, and time, among other things, without requiring direct comparison or calibration. For this purpose, the primary instruments based on liquid column manometer (LCM) and pressure balances (PBs) are very common in use. In the case of LCM, the height of a liquid column of known density is measured under known gravity conditions while in the case of PB, the force acting on a piston of known cross-sectional area, rotated into a cylinder of matched dimension under known gravity conditions. The pneumatic and hydraulic types of PBs fall under these categories. The use of LCM is limited around atmospheric pressure with the highest accuracy. However, in the case of PB, the range is extended to several GPa with reasonably good accuracy[14-16]. The graphical view of a simple U-tube type manometer and PB is shown in Fig. 2.

Furthermore, due to mercury's status as neurotoxic and hazardous to the environment, the acquisition of mercury products is prohibited. As a result, government laboratories, national laboratories, and enterprises are compelled to retire their mercury manometers and re-establish their pressure-measurement capabilities. Several of the National Metrology Institutes (NMIs) including CSIR-NPL, across the globe, still continue to use its mercury-based manometers as the national pressure standard but have started focused efforts towards alternative arrangements[17-18]. As mentioned earlier, pressure is a mass-related quantity, as illustrated in Fig. 2, and its manifestation is directly linked to the traceability of mass, which is no longer now artifact-based. Quantum physics is now used to realize the unit mass. As a result, the researchers have turned their attention to the realization of Pascal's quantum[19-24]. Lasers and Fabry-Pérot optical cavities are used to explore the fundamental physics behind light-matter interaction. The dielectric susceptibility of a matter is stronger than the dielectric susceptibility of vacuum. As a result, light in a gas moves slower and has a shorter wavelength than light in a vacuum. An optical interferometer manometer (OIM), wherein the refractive index of the gas molecules is monitored as a function of applied pressure, is one of the interesting areas for the realization of quantum Pascal (Pa) or new pressure scale. Although, the application of the OIM technique is limited to a pressure range from fraction of a Pa to 150 kPa but its implications in establishing traceability in higher pressure ranges are achievable and far reaching in the high-pressure range, extended intermediate pressure range devices are utilized to provide traceability. As a result, PBs based on piston cylinders may be traced using OIM, which can also be used to trace higher pressure ranges in the measurement chain. Thus, the PBs becomes the choice of transfer standard for providing traceability from low pressure range using OIM to high-pressure range.

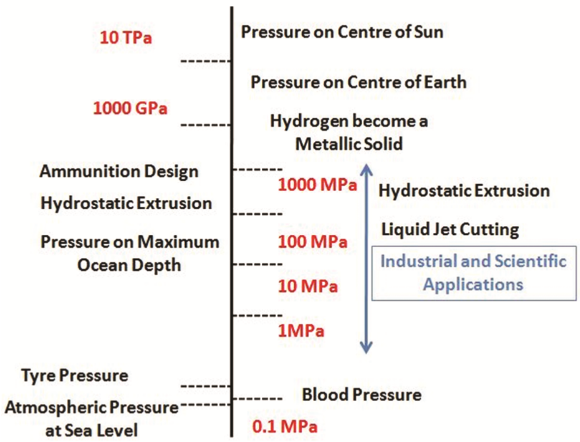

Fig. 1 — Range and applications of the pressure measurement.

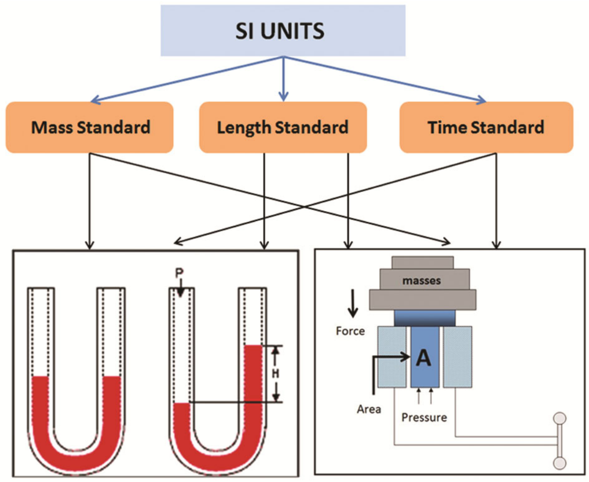

Fig. 2 — Graphical view of primary instruments for pressure measurement i.e. U-tube manometer and Pressure balance connected with SI units.

3 Effect of redefined kilogram on the pressure realization

Pressure is a derived unit which is traced back to one of the seven SI base units i.e. Mass. In high-pressure metrology and several other domains, mass is one of the most essential units. The issues related to mass come under the purview of BIPM's Consultative Committee on Mass Related Quantities (CCM), and the SI unit system has been amended and mass is now defined in terms of fundamental constant. The resolution towards the modification of the SI units was unanimously accepted by 60 BIPM member states in General Conference on Weights and Measures (CGPM). The CGPM approved the outcome of this extensive procedure at its 26th meeting in November 2018, and it is now in use from May 20, 2019. The SI is now based on a set of seven constants having fixed numerical values [25-30]. The dependency of the unit of mass on other units is shown in Fig. 3.

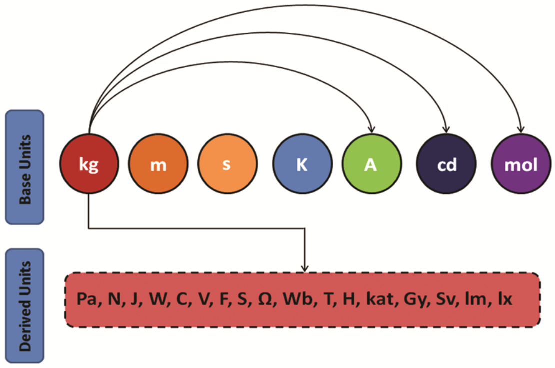

Fig. 3 — Effect of the unit of mass on various other units.

This shift in the fundamental base SI units has also encouraged the development of future pressure realization systems. Because pressure is a mass-related quantity, it is intrinsically linked to mass traceability, which is no longer based on the artifacts. It is now defined as a quantity proportional to the Planck constant h at a given numerical value. As previously stated, the PB requires mass traceability in order to realize pressure. As a result, the pressure accuracy is reliant on the dependent SI units.

For the achievement of mass standards in each country under the new SI units system, all NMIs with traceability through the International Prototype of Kilogram (IPK) are advised to assess and amend their measurement uncertainty budget accordingly[31]. Though, the mass values provided in the BIPM certificates will be the same; very recently the measurement uncertainty of 20 µg is added in place of the existing measurement uncertainty of 10 µg reported by BIPM earlier. This condition will be maintained until the ongoing key comparison is completed and some consensus arrives. Fig. 4 depicts a schematic diagram of the realization of mass and its traceability through two feasible routes. The experiments on the Kibble balance and XRCD approach, on the other hand, would be continued in order to improve measurement uncertainty.

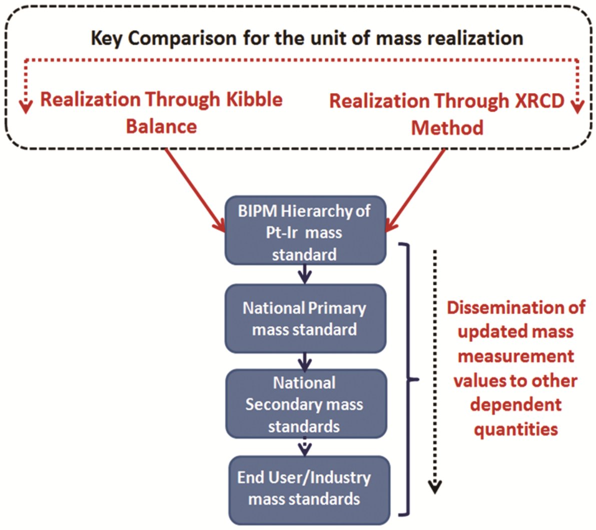

Fig. 4 — Traceability of the mass realization with current consensus value.

Customers, on the other hand, will not see the immediate impact of this change in the marketplace. Manufacturers of scientific instruments are likely to face the most significant shift, since some may need to adjust their products in the future to fit the new SI approach for better measurements. Another significant advantage of the revised SI is greater measurement scalability. In the previous system mass accuracy falls considerably smaller or larger than the mass standard. For example, drug manufacturers may need to test substances in quantities a million times smaller than a typical kilogram in order to follow the research on new medications.

4 Role, Status and Capabilities in High Pressure Measurement

Several scientific reviews and books go into extensive details about the high-pressure measurements. Different NMIs use primary standards based on PBs which are normally manufactured by leading companies such as DH-Budenberg (Now merged to WIKA Group), GE, Ruska (Now merged to Fluke), DH Instruments (Now merged to Fluke), and Harwood Engineering Company Inc, etc. It is herein clarified that in no way authors have the intention to promote a particular manufacturer but reference is made only for the sake of knowledge for readers. Several reports on the capabilities of various NMIs and also state of the art models of the leading manufacturing companies are summarized in the literature [32-33].

The NMIs demonstrate the international equivalence of their measuring standards and the global acceptance of their calibration and measurement certificates issued through CIPM Mutual Recognition Arrangement (CIPM MRA). As an outcome of the stringent process which includes successful participation in international comparisons and implementation of Quality System in the laboratory as per ISO/IEC 17025(2017) standards [34], are the CMCs. The international peer-reviewed and approved CMCs and associated technical data are publicly available in the Key Comparison database (KCDB) of BIPM. Because of the continued development, rapid growth, and demands from users and industries for dependable and repeatable parts, NMIs are constantly improving their measurement capabilities and skills for the overall growth of their respective countries.

The international status of the existing CMCs and participation of NMIs in different K&SC exercises in pressure parameters are shown in Fig. 5 & Fig. 6, respectively. The analysis of the CMC data revealed that 26 NMIs took part in various K&SC exercises but did not register their CMCs in the KCDB. It is not the intention of the authors to present the technological advancement or status of any country depicting the number of CMCs and K&SCs but to convince the readers that the sustained participation in key comparisons and registering CMCs are extremely important for the economic growth of the country's [9-10]. The pressure ranges and associated measurement uncertainties are included keeping in view of the larger interest and reach of the readers in a single-window information base. The maximum high-pressure range in CMCs of different NMIs with associated uncertainty is shown in Fig. 7 & Fig. 8. Only 21 NMIs have the CMCs in the pressures range ≥500 MPa and up to 1.6 GPa. Other NMIs may also have high-pressure measurement capabilities but their CMCs are not registered in the KCDB. Furthermore, due to obvious reasons for being outside the scope of the paper, an investigation of the status of the vacuum range is excluded. The detailed data can be found in the literature[13, 35].

5 Status of HPM facilities at CSIR-NPL

The CSIR-National Physical Laboratory (CSIR-NPL) is the NMI of India, which is also popularly known as NPLI in the metrological world. As per Parliament Act of India, it is responsible for establishing, maintaining, upgrading, and disseminating the National Standards of Measurement. All the NMIs are responsible for establishing a continuous chain of measurement traceability using national and international standards. The demand for dependable, repeatable, and long-lasting measurement tools is rapidly increasing. Therefore, the role of NMIs becomes more demanding and crucial for strengthening the industrial growth. Over the years, the CSIR-NPL has excelled in HPM, establishing various primary and secondary pressure and vacuum standards, as well as providing apex level calibration and testing services to industries in the pressure range of 3 μPa to 1.0 GPa. The following references[5,36-44] provide detailed information on the status of these facilities.

Most of the metrological standards established at CSIR-NPL are made globally compatible by participating in various CCM and APMP linked key comparisons. The Group has published 17 CMCs and participated in more than 20 key comparison exercises in the past. Fig. 9 depicts various hydraulic pressure standards established at CSIR-NPL. Using various pneumatic and hydraulic pressure standards established, the group has participated in different Proficiency Testing (PT) Programs in the high-pressure range. A detailed summary of the PT programs is described in the subsequent section.

6 High Pressure Proficiency Testing Programs by CSIR-NPL

As part of the evaluation process, the national accreditation body (NCB) conducts the PT programs to assess the technical competence of its accredited laboratories to perform the specified and approved tasks for which accreditation has been requested / granted. This examination is in addition to the on-site laboratory evaluation by the technical experts. The NCBs also requires laboratories to participate in the PT exercises for all types of analyses, tests and calibrations performed in their laboratories whenever

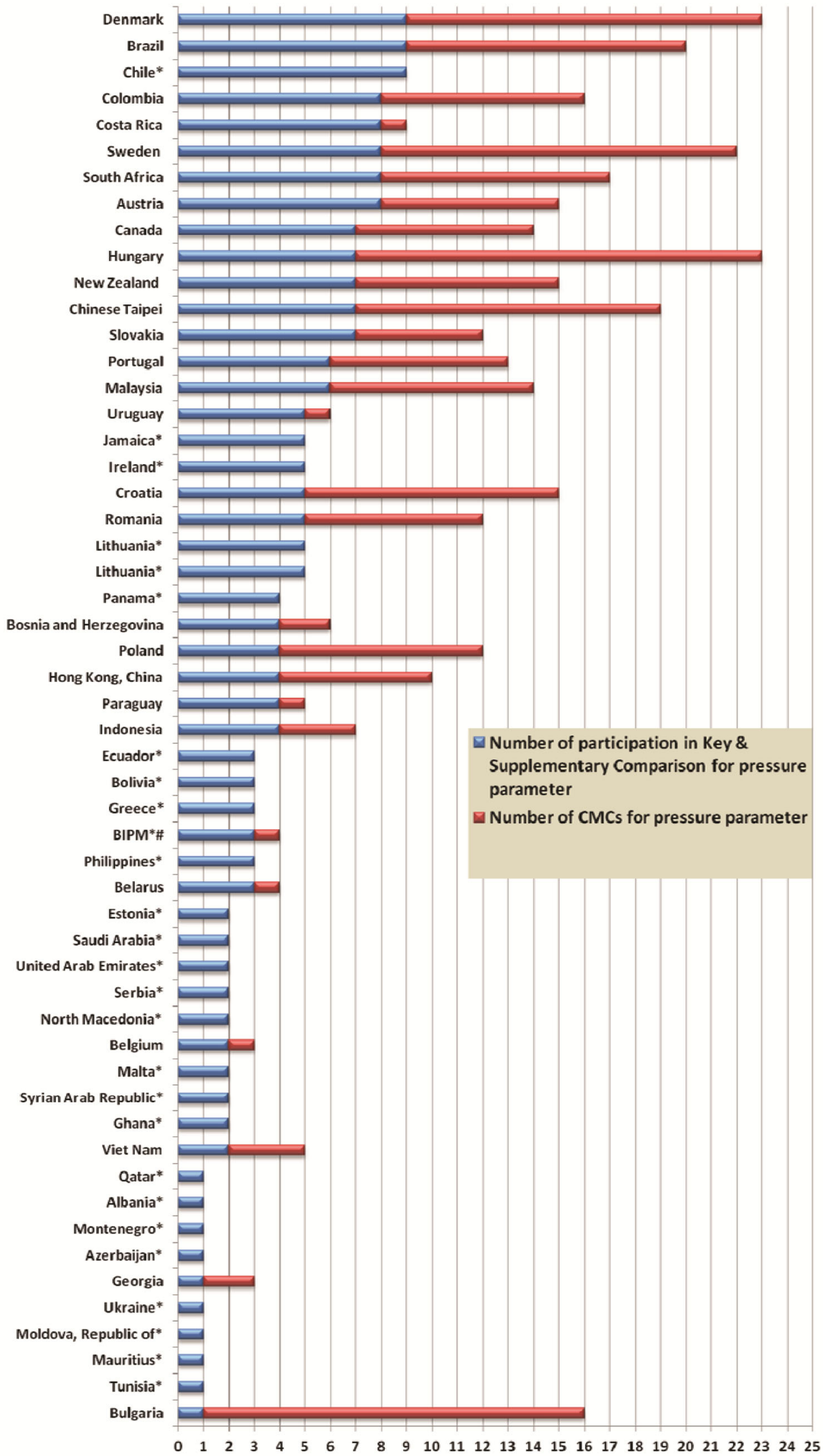

Fig. 5 — Participation of NMI in Key & Supplementary comparison exercise and their associated CMCs ($number$ <10) in pressure; * no CMC registered in pressure *# organization.

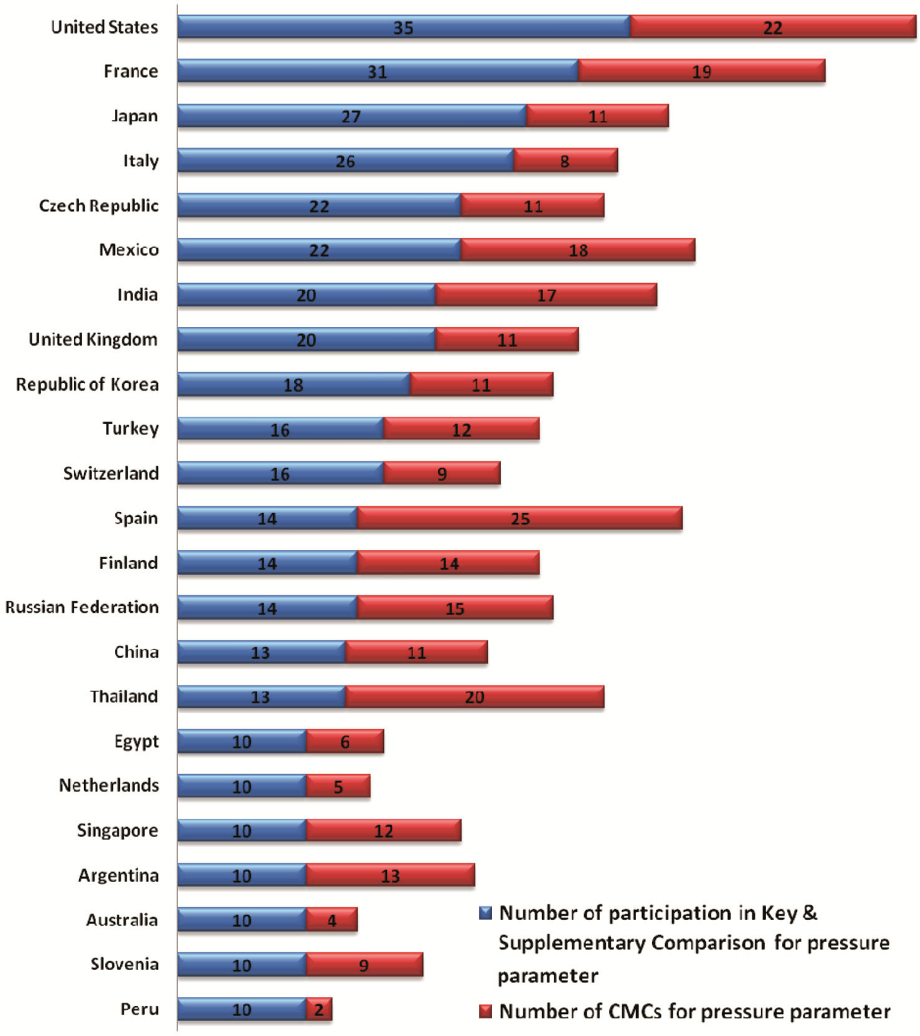

Fig. 6 — Participation of NMI in Key & Supplementary comparison exercises and their approved CMCs ($number \geq 10$) in pressure.

relevant PT exercises are available. The exercise allows accredited calibration laboratories to demonstrate their technical competence in providing the routine calibration services to their clients, as well as assess their laboratories.

The ISO/IEC 17025 (2017) standard [34] and APLAC MRA001 [45] also stipulate the requirement of successful participation of calibration / test laboratories in PT programs. Also as per ISO/IEC[46] Guide 43, all the NCBs are required to run the PT programs for their approved laboratories. In conjunction with the CSIR-NPL, the PT programs for NABL accredited laboratories were developed and successfully carried out several PTs. This procedure adds a second layer of quality control to laboratory testing results. In addition to strengthening the laboratory's own quality control systems with additional external audits, it provides objective evidences that a laboratory is competent and capable of achieving the level of uncertainty for which certification is issued.

The reference laboratory, in this case, CSIR-NPL, also undertakes self-evaluation and maintains its competencies. As a result, CSIR-NPL has coordinated 10 NABL-sponsored PT programs in various pressure ranges over the years[47-57]. A total number of 140 NABL accredited laboratories were accommodated using a choice of artifacts such as mechanical, analog, and digital types according to their scope and technical ability in high-pressure measurement.

The measurement performance of participating laboratory in the inter-laboratory comparison is

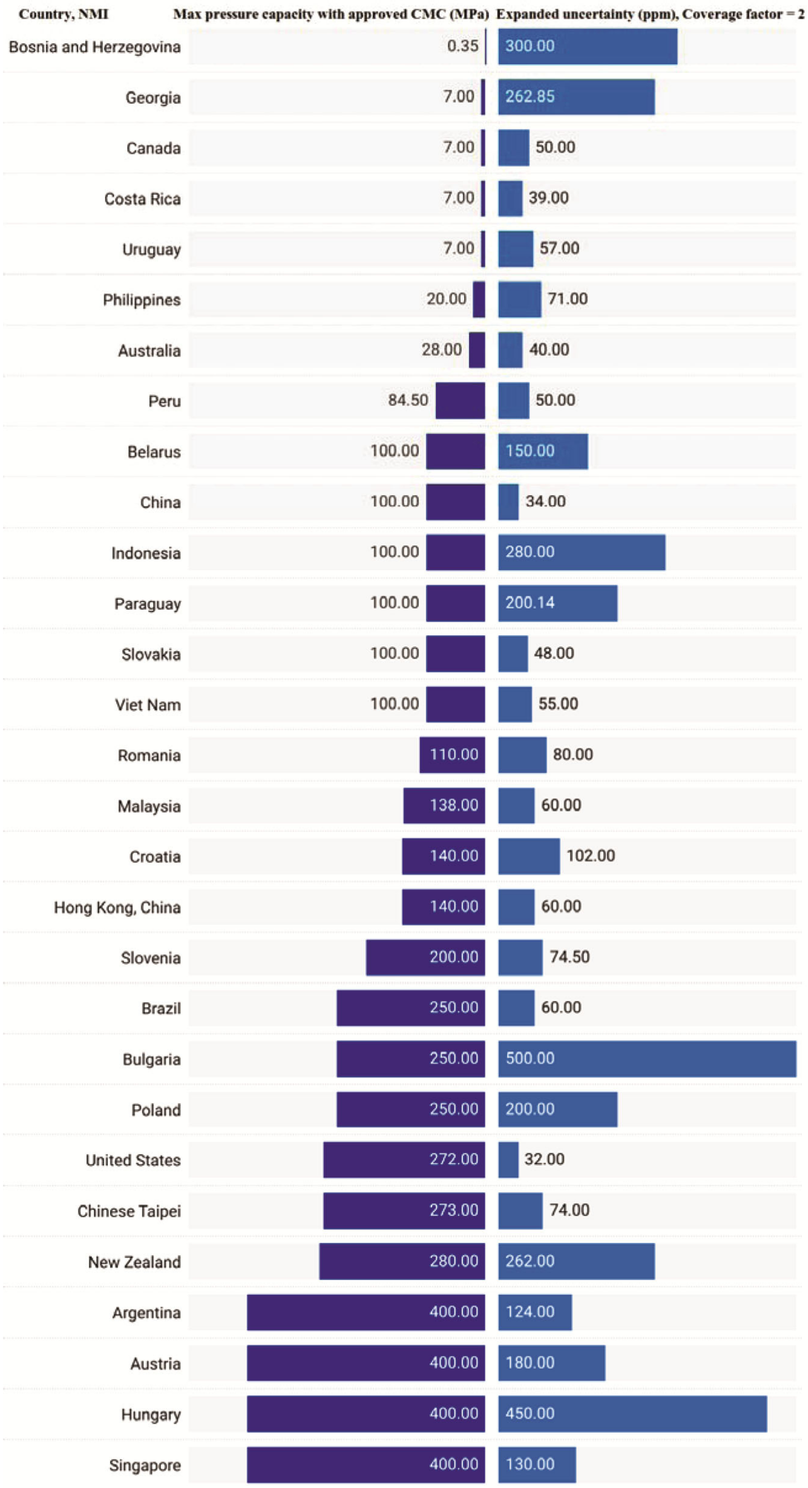

Fig. 7 — Maximum pressure capacity with approved CMCs of NMIs in pressure <500 MPa.

assessed on the basis of the Error Normalized (E_n) number of each measurement. It suffices to say that En values are estimated for each participant at each pressure using the equation;

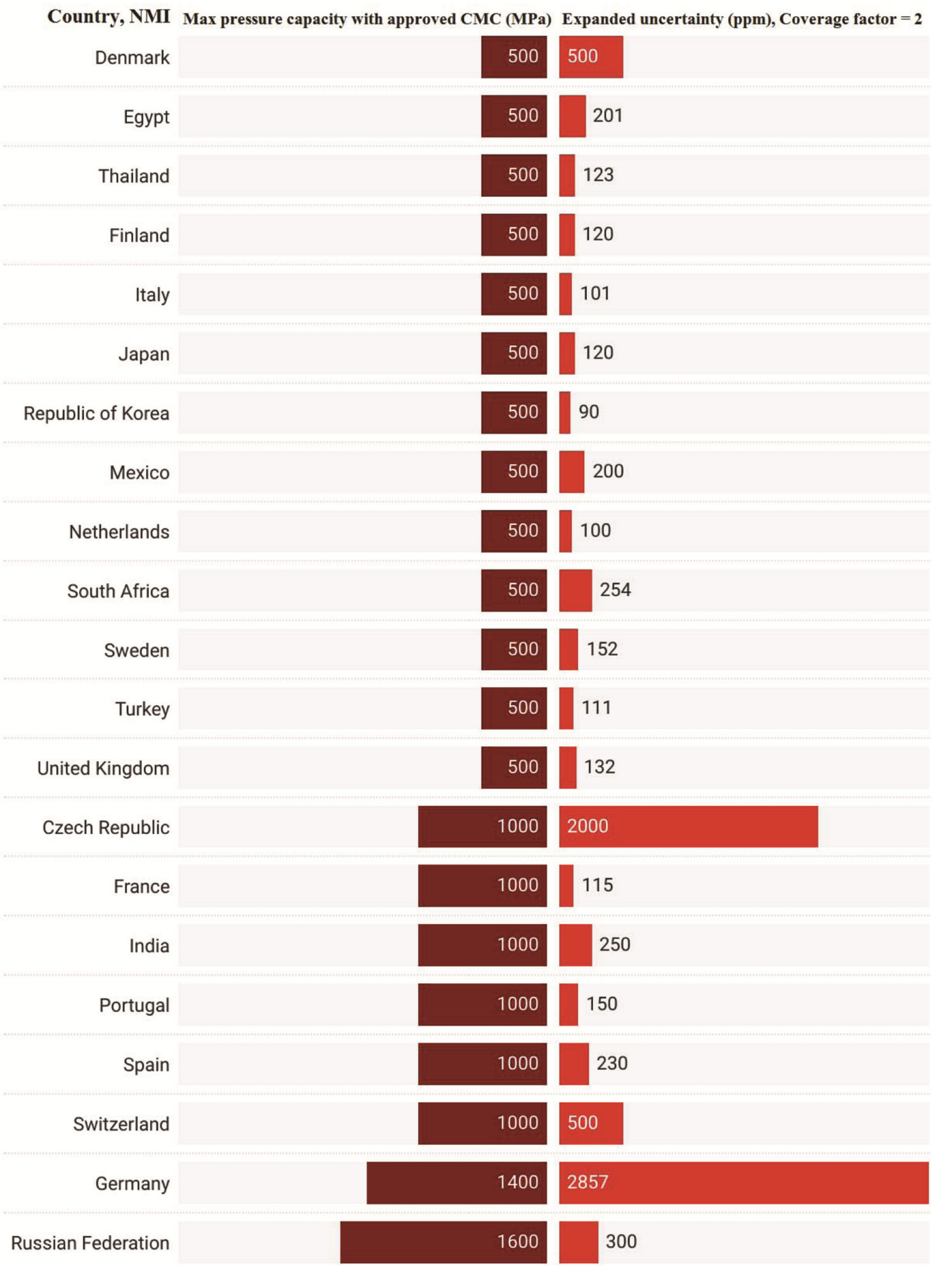

Fig. 8 — Maximum pressure capacity with approved CMCs of NMIs in pressure ≥500 MPa.

$$E_n Value = \frac{P_{LAB} - P_{\mathrm{Re}\,f}}{\sqrt{\{U(P_m)\}^2 + \{U(P_{\mathrm{Re}\,f})\}^2}} \quad \ldots (1)$$

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

An E_{n} value within ±1.0 is acceptable. The deviation from the ±1.0 indicates that the laboratory's results have a bias, and that the cited values of the associated uncertainty does not fully account for that bias, necessitating further route cause analysis by the laboratory.

Due to obvious reasons, it is not possible to report the findings of all the PTs in this study. As an example, Fig. 10 depicts the outcome of one of the PT exercises (NABL-Pressure -PT007). Throughout the whole pressure range of (6–60)

MPa, the comparison was carried out at ten arbitrarily chosen pressure points: 6, 12, 24, 30, 36, 42, 48, 54, and 60 MPa. The results show that throughout the full pressure scale, the *E*n values of 7 laboratories are within acceptable limits. The E_n values of two other laboratories are similarly fairly satisfactory except for one pressure point. For more than 3 pressure points, the *E*n values of the remaining 5 laboratories are found to be beyond the permitted range.

Further, Table 1 shows the participant's summary of different conducted PT programs in the hydraulic pressure range.

The results obtained in PTs were extremely encouraging but this program is not rigorously

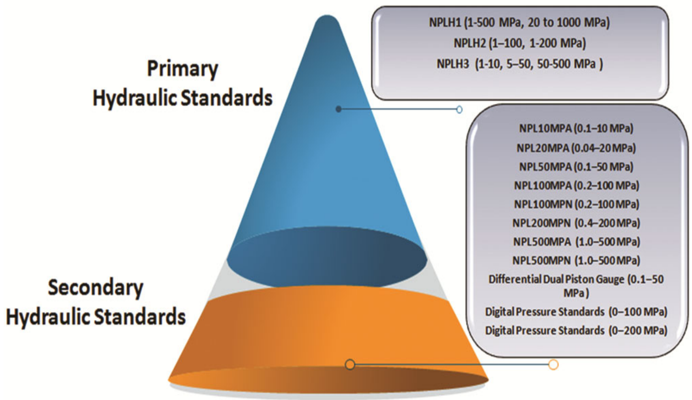

Fig. 9 — Primary and secondary hydraulic pressure standards established at CSIR-NPL.

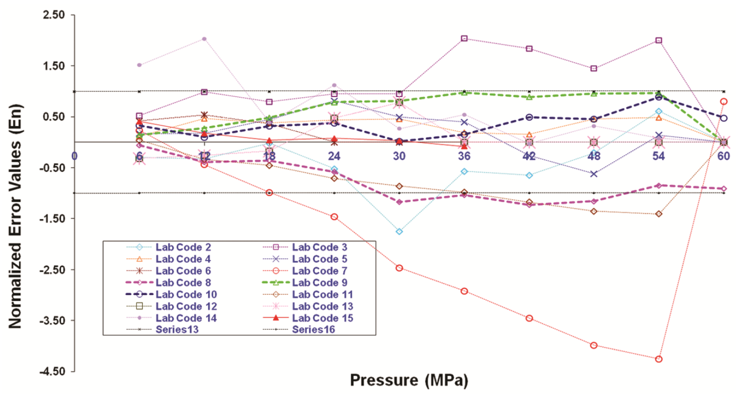

Fig. 10 — The normalized error value (E_n) as a function of measured pressure for each laboratory.

Table 1 — Summery of the proficiency testing programs in the hydraulic pressure measurement

Program Serial Code	Range and Instrument	Duration of the program	Number participated of Laboratories/ organization	Accepted E_n values of Laboratories/organization for entire pressure range	Results
NABL-Pressure - PT001	Inter-laboratory comparison in the pressure range 7–70 MPa using dead weight tester as an artifact	November 2002 to May 2004	7	6	Out of the total 69 measurement results reported, 61 (88.4%) measurement results are found in good agreement with the results of the reference laboratory
NABL-Pressure - PT002	Inter-laboratory comparison in the pressure range 5–70 MPa using digital pressure calibrator as an artifact	July 2003 to May 2004	8	1	Out of the total 145 measurement results reported, 86 (59.3%) measurement results are found in good agreement with the results of the reference laboratory

(*contd.*)

Table 1 — Summery of the proficiency testing programs in the hydraulic pressure measurement (*contd.*)

Program Serial Code	Range and Instrument	Duration of the program	Number participated of Laboratories/ organization	Accepted E_n values of Laboratories/organization for entire pressure range	Results
NABL-Pressure - PT003	Inter-laboratory comparison in the pressure range 5–70 MPa using pressure dial gauge as an artifact	February 2003 to March 2004	11	2	Out of the total 145 measurement results reported, 86 (59.31%) measurement results are found in good agreement with the results of the reference laboratory
NABL-Pressure - PT004	Inter-laboratory comparison in the pressure range 7–70 MPa using dead weight tester as an artifact	March 2008 to April 2010	9	5	Out of the total 87 measurement results reported, 68 (78.2%) results are found in good agreement with the results of the reference laboratory
NABL-Pressure - PT005	Inter-laboratory comparison in the pressure range 7–70 MPa using digital pressure calibrator as an artifact	May 2006 to May 2008	21	9	Out of the total 178 measurement results reported, 165 (92.7 %) measurement results are found in good agreement with the results of the reference laboratory
NABL-Pressure - PT006	Inter-laboratory comparison in the pressure range 10–70 MPa using pressure dial gauge as an artifact	May 2006 to October 2007	17	9	Out of the total 159 measurement results reported, 135 (84.91 %) measurement results are found in good agreement with the results of the reference laboratory
NABL-Pressure - PT007	Inter-laboratory comparison in the pressure range 6–60 MPa using pressure dial gauge as an artifact	May 2006 to October 2007	15	7	Out of the total 117 measurement results reported, 95 (81.2%) measurement results are found in good agreement with the results of the reference laboratory

followed up and is almost inactive now. It is expected that NABL would take lead in this direction to make sure that such programs are continued for the self-evaluation of the accreditation laboratories.

7 Conclusion

A pithy study of the national and global status of high pressure measurement facilities; primary measurement methods; effect of the redefined SI unit of mass on HPM: existing CMCs of various NMIs and their successful participation in K&SCs; and role of the PT program and detailed analysis of the previous excises, is presented and discussed. A concise report on the analysis with a graphical summary including most of the aspects of high pressure metrology in a single window manner would be highly useful to readers specially students, researchers, metrologists, regulators and calibration laboratories as a reference source and explore new problems in HPM for the growth of the field.

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