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Pressure measurements in gas (absolute mode)

KEY COMPARISON

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Final Report on the Key Comparison APMP.M.P-K4 in Absolute Pressure from 1 Pa to 10 kPa

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ABSTRACT

The report summarizes the Asia-Pacific Metrology Programme (APMP) key comparison APMP.M.P-K4 for absolute pressure spanning the range of 1 Pa to 10 kPa which was carried out at six National Metrology Institutes (NMIs: NMIJ, CMS/ITRI, NPLI, NIMT, NIM and KRISS) during the period of January 2018 to July 2019. The comparison was made via a calibrated transfer standard measured at each of the NMIs facilities using their laboratory standards. The transfer package constructed for this comparison performed as designed and provided a stable artifact to compare laboratory standards. Overall, the participants were found to be statistically equivalent to the key comparison reference value.

Contents

1. INTRODUCTION

The first CCM key comparison (CCM.P-K4) was piloted by the National Institute of Standards and Technology (NIST, USA) using high-resolution pressure transducers as transfer standards in the absolute pressure range of 1 Pa to 10 kPa and finished well in 2002. The second CCM key comparison (CCM.P-K4.2012)[1] was also piloted by the NIST in the absolute pressure range of 1 Pa to 10 kPa and ended in 2012. Two NMIs (Korea Research Institute of Standards and Science (KRISS) and National Physical Laboratory of India (NPLI)) in APMP region participated in CCM.P-K4, and one NMI (National Metrology Institute of Japan (NMIJ) participated in CCM.P-K4.2012.

In 2014, the TCM in Asia-Pacific Metrology Programme (APMP) approved APMP key comparison (APMP.M.P-K4) piloted by KRISS.

This report summarizes the key comparison APMP.M.P-K4 for absolute pressure spanning the range of 1 Pa to 10 kPa which was carried out at six National Metrology Institutes (NMIs: NMIJ, Center for Measurement Standards/Industrial Technology Research Institute (CMS/ITRI), NPLI, National Institute of Metrology Thailand (NIMT), National Institute of Metrology (NIM) and KRISS) during the period of January 2018 to July 2019. The comparison was made via a transfer standard measured at each of the NMIs facilities using their laboratory standards. The transfer package constructed for this comparison was performed as designed and provided a stable artifact to compare laboratory standards. NMIJ served as a link lab for CCM KC (CCM.P-K4), which was the only NMI in APMP region to participate in CCM KC. Overall, the participants were found to be statistically equivalent to the key comparison reference value.

The following sections provide brief descriptions of the laboratory standards, the design and construction of the transfer standard packages, the organization and chronology of the comparison, and the general calibration procedure required by the protocol. Methods for the analysis of the calibration data were chosen to provide, as much as possible, uniform treatment of the results from individual laboratories, whether they were the pilot or another participant laboratory.

2. LABORATORY STANDARDS

The following laboratories participated in this comparison, listed along with coordinators for contacts.

Table 1. List of Participating Laboratories.

Laboratory, Contact person	Address	Phone number	E-mail
KRISS, Han Wook Song	Rm 305, Bldg 209, KRISS, 267, Gajeong-ro, Yuseong-gu, Daejeon, South Korea	Tel: +82-42-868-5821	hanugi16@kriss.re.kr
NMIJ, Kenta ARAI/ Momoko KOJIMA	National Metrology Institute of Japan (NMIJ), National Institute of Advanced Industrial Science	Tel: +81-80-2161-6836	kenta-arai@aist.go.jp/ m.kojima@aist.go.jp

	and Technology (AIST), AIST Tsukuba Central3, 1-1-1 Umezono, Tsukuba 305-8563, JAPAN		
CMS/ITRI, Sheng-Jui Chen	Center for Measurement Standards, Industrial Technology Research Institute, 321, Sec. 2, Kuang Fu Rd., Hsinchu, 30011, Taiwan, R.O.C.	TEL: +886 3 573 2912 FAX: +886 3 572 4952	SJ.Chen@itri.org.tw
NPLI, Ashok Kumar	NPLI, Room No: 114, TEC Building, National Physical Laboratory, Dr. K. S. Krishnan Marg, New Delhi, 110012, India	Tel: + 91-11-4560-8533 Fax: + 91-11-4560-8690	ashok553@nplindia.org; ashok553@gmail.com
NIMT, Suwat Phanakulwijit/ Jeerasak Pitakarnnop	National Institute of Metrology (Thailand), 3/4 -5 Moo 3, Klong 5, Klong Luang, Pathumthani 12120, Thailand	Tel: +66-2577- 5100 ext: 2106, 2239 Fax: +66-2577-3659	suwat@nimt.or.th / jeerasak@nimt.or.th
NIM, Yuanchao Yang/ Hongyan Yu	Pressure and Vacuum Lab, Division of Thermophysics Metrology, National Institute of Metrology, No.18 Bei San Huan Dong Lu, Chaoyang Dist., Beijing 100029, China	Tel: +86-10-6452-5115 Fax: +86-10-6421-8637	yangyc@nim.ac.cn

The principal measurement methods used in this comparison involved 4 types of laboratory standards: static expansion systems (SES), force-balanced piston gauges (FPG), ultrasonic interferometer manometer (UIM), and other calibrated transducers (such as capacitance diaphragm gauge). KRISS used the FPG as their laboratory standard while NMIJ used two types of standards according to the pressure range (SES from 1 Pa to 100 Pa and RSG from 300 Pa to 10000 Pa). NIM used two types of standards according to the pressure range (SES from 1 Pa to 30 Pa and FPG from 100 Pa to 10000 Pa) while NPLI used two types of standards for whole pressure range (UIM and FPG). Two participants (CMS/ITRI and NIMT) used the calibrated transducers as standards.

2.1. KRISS

KRISS used a Force balanced Piston Gauge(FPG)), manufactured by Fluke/DH-Instruments, USA. The FPG effective area was calibrated in both gauge and absolute modes by a 35 mm piston gauge, which is maintained as the KRISS primary pressure standard. The characteristics of the FPG were as follows:

Range: 1 Pa to 10 kPa   Uncertainty (k = 2): U = 0.015 Pa + 5×10^{-5}p

where p is the pressure in Pa.

2.2. NMIJ

NMIJ used two standards, the SES (< 100 Pa) and the differential pressure gauges calibrated by a double pressure balance as standards (>= 100 Pa) as same system used in CCM.P-K4 2012 [1]. The relative uncertainties of the static expansion system are:

Range: 1 Pa to 150 Pa   Uncertainty (k = 2): U = 1.8×10^{-3}p

For the range of 300 Pa to 10 kPa, NMIJ used two differential pressure gauges calibrated by a double pressure balance as standards. A 1 kPa resonant silicon gauge (RSG) was used for 300 Pa to 1 kPa and a 10 kPa RSG was used for 1 kPa to 10 kPa. Each of the gauges was calibrated under the line pressure of 100 kPa (absolute mode) in the double-pressure-balance system. Then the low pressure port was evacuated and used as an absolute pressure gauge (with a line pressure of 0 kPa). The indication of the gauge shows some dependence on the line pressure, so the dependency was evaluated and applied as a correction. The uncertainty for this measurement system using differential pressure gauges is as follows:

Range: 300 Pa to 1 kPa   Uncertainty (k = 2): U = 0.12 Pa + 2 × 10^{-5}p

Range: 1 kPa to 10 kPa   Uncertainty (k = 2): U = 0.15 Pa + 5.5 × 10^{-5}p ,

where p is the pressure in Pa.

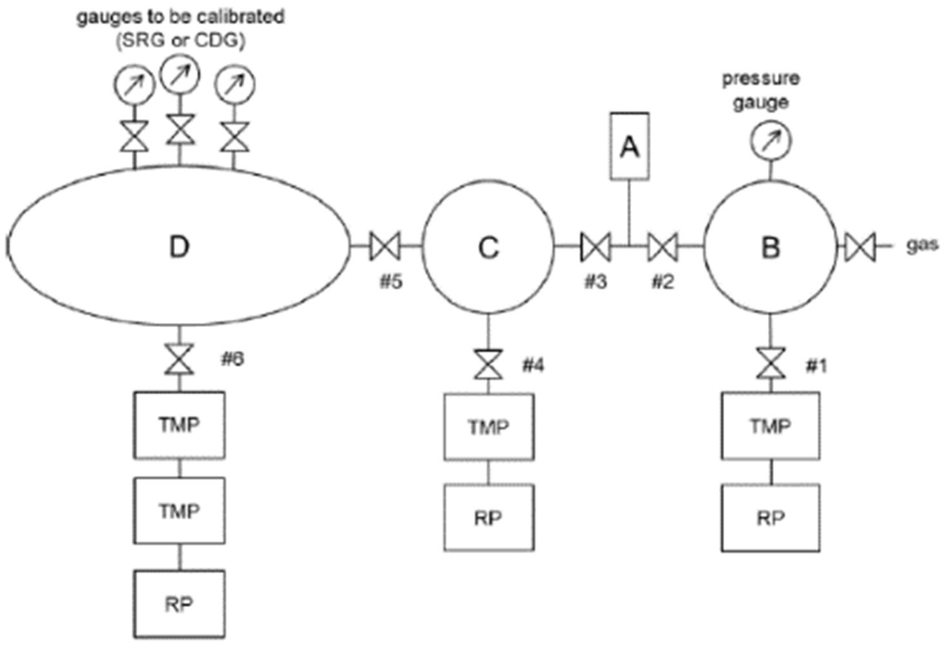


Figure 1 Schematic diagram of the NMIJ Static Expansion System

2.3. CMS/ITRI

A direct comparison system with our internal service identifier L01 was used for this comparison, in which two Capacitance Diaphragm Gauges, with full measurement range of 1.33 kPa and 133 kPa respectively and both traceable to PTB's static expansion system, serve as reference standards in this system. The measurement range for the L01 system when the above mentioned two CDGs serving as reference standards is 1 Pa to 100 kPa with an uncertainty of $U = 1.8\times10^{-2}p$ ($k = 2$).

2.4. NPLI

NPLI has used two primary pressure standards; (i) Ultrasonic interferometer manometer (UIM) (ii) Forced balanced Piston Gauge (FPG) to obtain the data for KC in the range of 1 Pa to 10 kPa; however, as per instruction from the pilot lab, only UIM data were utilized for KC and En value calculation. The UIM at NPLI was established in 1982 and is situated at the top of the traceability chain and is capable of working in absolute and gauge modes. The performance of UIM has been regularly checked, and the experimental uncertainty is found to be $\mathrm{U} = \sqrt{(0.0092\ \mathrm{Pa})^2 + (7.2\times10^{-6}p)^2}$. Sometimes the uncertainty in the range of 1Pa to 1kPa was observed to be higher than the theoretically calculated resolution value 0.0092 (k = 2) depending on the environmental conditions, mercury column stability, fringe counting, zero instability, column tilt, reference pressures and other parameters affecting the resolution. The UIM is very robust and stable in the pressure measurement range 1 kPa to 130 kPa, and most of the time, the uncertainty is well within the claimed expanded uncertainty. The pressure values were calculated using highly precise column length measurement using the phase measurement technique and an exact fractional algorithm. A window-based software has been used to accurately calculate pressure using the measurement of time of flight, speed of sound in mercury, and 44 different frequencies covering two full circles of fractional fringes using phase measurement. Although UIM is forty years old equipment, the uncertainties calculated theoretically and experimentally are well within the claimed uncertainties.

2.5. NIMT

The laboratory standards at NIMT used in this key comparison are two Capacitance Diaphragm Gauges (CDGs) with full-scale range of 133 Pa and 1333 Pa, and a Quartz Reference Pressure Transducer (Q-RPT) with full-scale range of 110 kPa. Both CDGs are traceable to Physikalisch-Technische Bundesanstalt (PTB) and Q-PRT is traceable to NIMT's primary pressure standard by dimensional measurement method.

The uncertainties for the NIMT CDGs and Q-PRT are given below:

1 Torr CDG (133 Pa)	Range: < 133 Pa	Uncertainty (k = 2): $U = 1.6\times10^{-3}p$ to $3.4\times10^{-3}p$
10 Torr CDG (1333 Pa)	Range: 133 Pa to 1.3 kPa	Uncertainty (k = 2): $U = 1.6\times10^{-3}p$ to $2.7\times10^{-3}p$
Q-PRT	Range: 1.3 kPa to 110 kPa	Uncertainty (k = 2): U = 0.00073 kPa to 0.0022 kPa

2.6. NIM

The standard used was a static expansion system (SES) for the pressure range from 1 Pa to 30 Pa. It has participated in the international comparison CCM.P-K15.1 with METAS, Switzerland in the pressure range from 0.1 mPa to 1 Pa. The characteristics of the SES were as follows.

Range: 1 Pa to 30 Pa Uncertainty (k = 2): U = 1.6 × $10^{-3}p$

The force balanced piston gauge (FPG), manufactured by Fluke/DH-Instruments, USA, was used for the pressure range from 100 Pa to 10 kPa. The FPG effective area was calibrated in both gauge and absolute modes by a 20 cm^2 piston gauge, which is maintained as NIM primary pressure standard. The characteristics of the FPG were as follows.

Range: 100 Pa to 10 kPa Uncertainty (k = 2): U = 0.04 Pa + 3×$10^{-5}p$

3. TRANSFER STANDARDS

The transfer standard consists of two parts: (1) a pressure transducer package (PTP) and (2) a support electronics package (SEP). The PTP (see Fig. 2) includes two different transducers housed in a temperature-controlled enclosure with a thermometer, a monitoring gauge for monitoring and necessary plumbing. The internal thermal stabilization of CDGs was provided by temperature-controlled enclosure.

The PTP has the following general characteristics:

Dimensions(case1): 70 cm length x 53 cm width x 91 cm height

Weight: ~48 kg

Vacuum connections: 1/4-inch female VCR

Internal volume: ~ 50 cm^3 for pipe lines, ~ 2.5 cm^3 for each CDG, ~ 20 cm^3 for monitoring gauge and ~ 2 cm^3 for each valve

The characteristics of the two transducers are summarized in Table 2. The SEP includes a signal conditioning electronics for the CDGs, display electronics for the reference vacuum gauge, temperature control circuitry for the transducer enclosure, and a notebook computer. The SEP has the following electrical requirements:

Dimensions(case2): 70 cm length x 53 cm width x 73 cm height

Weight: ~50 kg

Voltage: 220 Volts AC

Frequency: 50/60 Hz

Power Consumption: <0.5 kW

Table 2. Characteristics of the Transfer Standard Transducers.

Transducer	Sensor type	Range	Resolution	Maximum

		(Pa)	(Pa)	Overpressure (Pa)
CDG1	Capacitance Diaphragm Gauge	133	0.001 % of FS	200
CDG2	Capacitance Diaphragm Gauge	13 300	0.001 % of FS	20 000

Case 1, shown in Figure 3, is of the clam shell type to ease the unpacking and repacking of the PTP. With Case 1 sitting upright, as indicated by the arrows on the sides of the case, the fasteners should be unlatched and the front and rear covers should be removed. The PTP should be inspected to ensure that no obvious damage has occurred during shipment.

Case 2, shown in Figure 4, contains the SEP for the PTP. The front and rear covers of the case also should be removed. The SEP is shock mounted into Case 2.

The PTP should be installed to the pressure standard of the participating laboratory so that its baseplate is approximately horizontal. The baseplate should be mounted on a stable horizontal surface. The leveling screws on the baseplate should be adjusted so that the bubble in the level-meter is centered to within the circle. (The level-meter is located inside the thermostatic box as shown in Fig.3.)

Case 2, containing the SEP, can be located in any convenient location within 1.5 meters from the PTP. The cabling from the PTP should be connected to the support electronics in the SEP before electrical power is applied. A schematic of the proper connections between the SEP and the PTP are shown in Figure 5. Color-coded tape is used on the cables and on or near the instrument connectors to facilitate the identification of matching connectors.

The PTP is shipped with its internal plumbing under vacuum with isolation valves V_1, V_3, V_4 and V_5 closed, and isolation valve V_2 open. The purpose is to protect the gauges against overpressure in case of a leak during shipment. Connect the pressure standard of each NMI to isolation valve V_1 of the PTP using suitable vacuum plumbing (internal diameter should be at least as large as that for the 1/4-inch VCR fittings). And evacuate the plumbing between the pressure standard and V_1 to a pressure of ~10^{-3} Pa before opening V_1. (We reached the 10^{-3} Pa within 1 h at KRISS. The lowest pressure value was 3 X 10^{-4} Pa.) Connect the proper vacuum pump using valve V_3 for evacuating the PTP. Each valve is operated manually.

For interlaboratory shipment, the PTP and SEP were mounted in specially designed commercial containers that conform to ATA (Air Transport Association) and pertinent military standard specifications for vibration and shock isolation.

4. ORGANIZATION OF THE KEY COMPARISON

4.1. CHRONOLOGY OF THE MEASUREMENTS

The transfer standard packages were developed for the comparisons. The comparison schedule, shown in Table 3, has been developed based on desired timing from the individual laboratories and to minimize the time required for the comparison. Participant laboratories are required to notify the pilot laboratory if they did not receive the artifact on or before the target date of arrival. Participant laboratories are required to notify the pilot laboratory via email (one week in advance) if they are unable to ship the transfer standard on the target date of completion. All efforts should be made to adhere to the schedule in order to minimize scheduling problems and to enhance the possibility of a timely completion of the comparison.

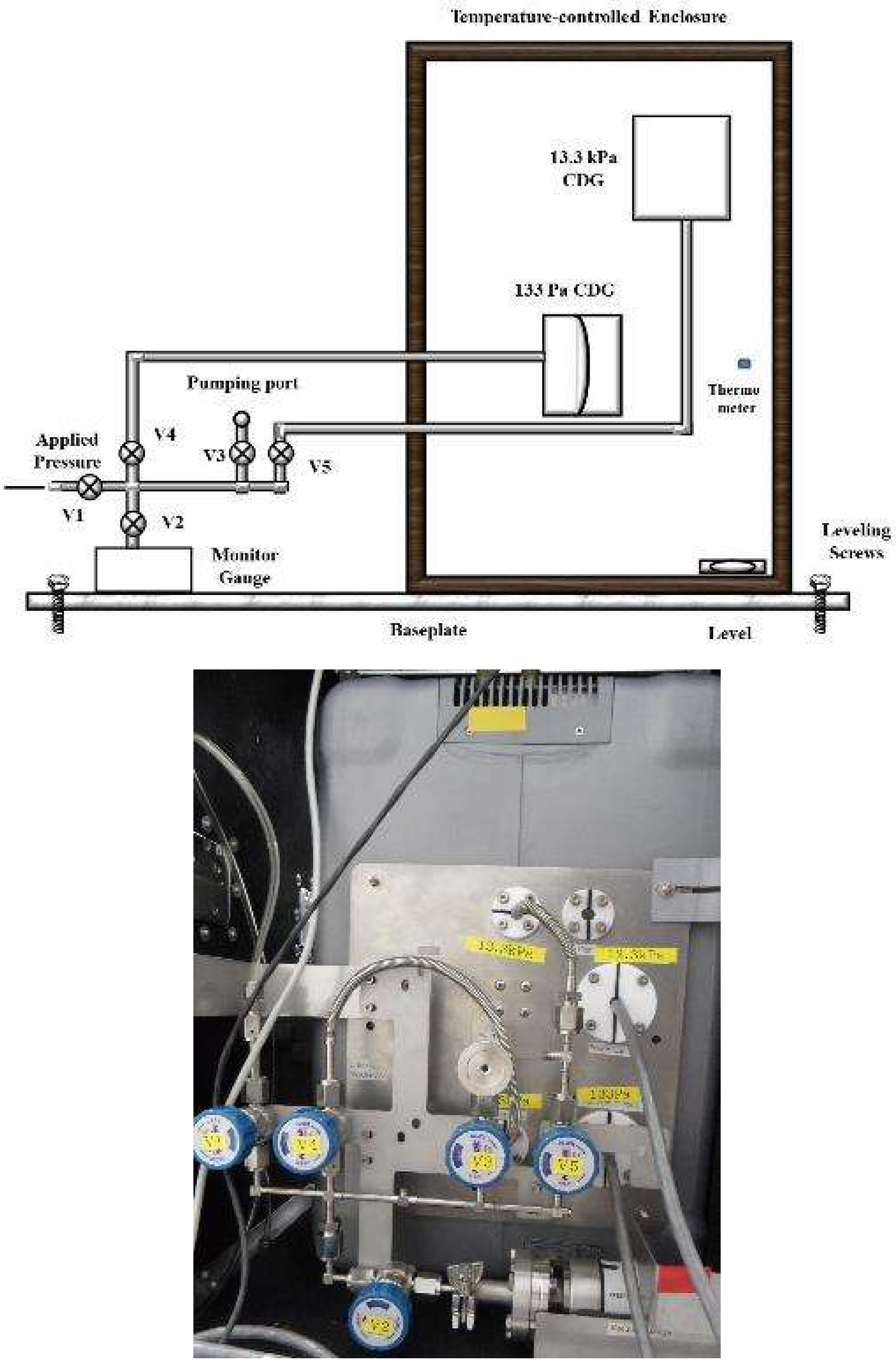


Figure 2. Schematic diagram of the pressure transducer package (PTP).

(a)

(b)

Figure 3. Photograph of Case 1 and the PTP with the front clamshell cover removed((a)). The PTP should be transported with its front adjacent to the recessed wheels of Case 1. The level-meter is shown on the bottom plate in the upper right figure and leveling screws are in figure (b).

Figure 4. Photograph of Case 2 with its front cover removed. The support electronics for the PTP are shock-mounted inside Case 2.

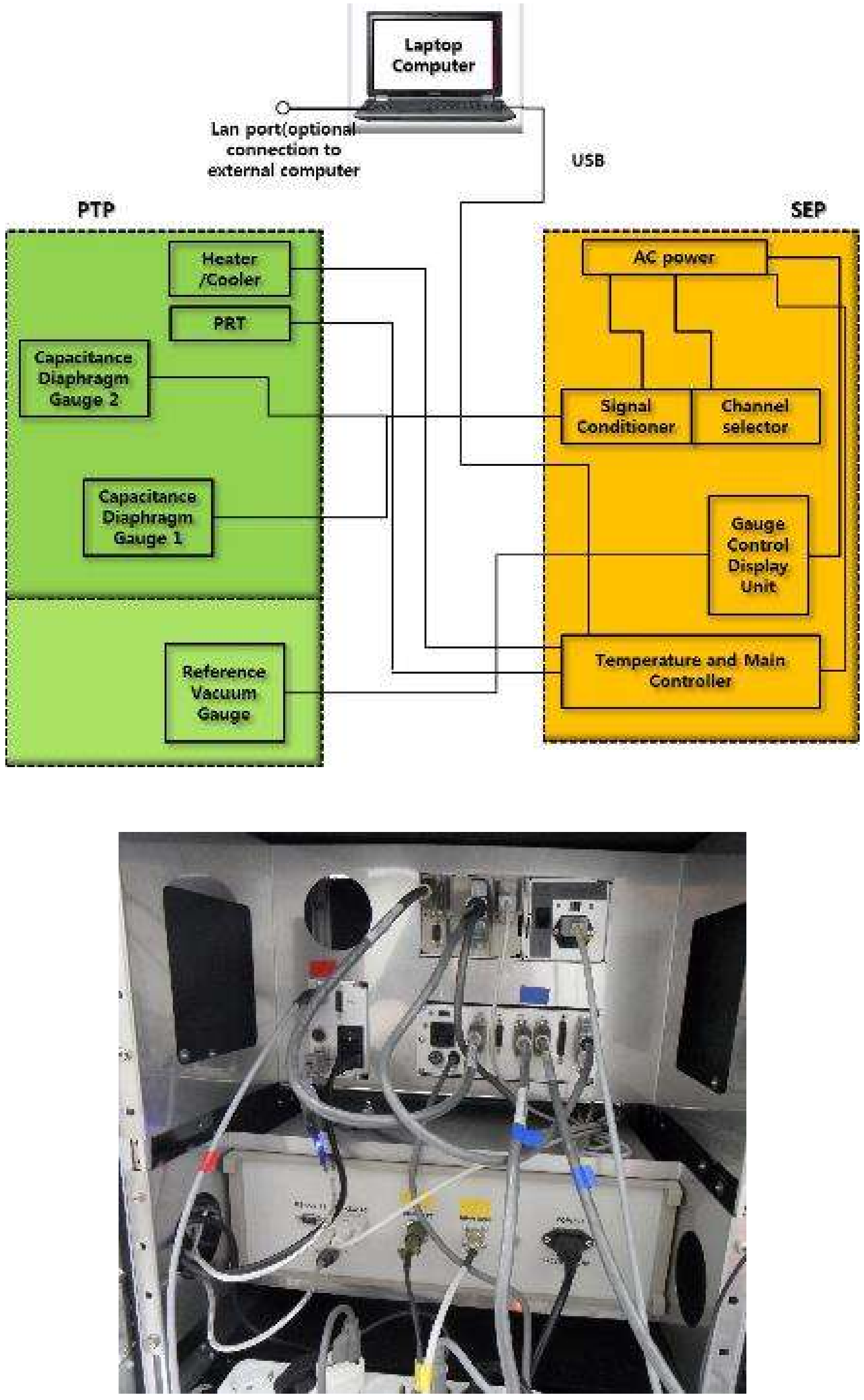


Figure 5. Cable interconnections between the pressure transducer package (PTP) and the support electronics package (SEP).

Table 3. Organization of the Comparison

Laboratory	Date of arrival	Date of Departure
KRISS	Feb. and Mar 2018	14 Mar 2018
NMIJ	16 Apr. 2018	1 June 2018
KRISS	18 June 2018	30 July 2018
CMS/ITRI	7 Aug. 2018	13 Oct. 2018
NPLI	30 Oct. 2018	23 Jan. 2019
NIMT	31 Jan. 2019	09 Apr. 2019
NIM	23 Apr. 2019	05 June 2019
KRISS	July and Aug. 2019	

4.2. PROBLEMS DURING THE COMPARISONS

Overall, the transfer standards package performed well. However, the vacuum fitting was broken during transferring to CMS/ITRI. CMS/ITRI fixed the problem to change the vacuum fitting and measurements were completed.

5. GENERAL CALIBRATION PROCEDURE

The transfer standard should be calibrated with nitrogen gas by comparison with the pressure standard of the participating laboratory at two mandatory cardinal points per decade, nominally at pressures of 1, 3, 10, 30, 100, 300, 1000, 3000, 10 000 Pa. The actual pressures realized by the participant's pressure standard during the calibration should not differ from the cardinal point pressures by more than 2%.

A mandatory five calibration runs should be carried out, with each run proceeding from lowest to highest pressures and each run taken on a different day. Additional calibration runs may be performed at the discretion of the participating laboratory but only data from five runs should be forwarded to the pilot laboratory for purposes of the comparison. It is mandatory that five sets of repeat readings of pressure (and temperature) of the transfer standard transducers and the pressure standard of the participating laboratory be taken at each cardinal point pressure within a given calibration run. Ideally, a reading of the pressure that is measured/generated by the participant's standard should be recorded at approximately the same time a set of readings of the transfer standard are recorded. This can be important for pressure standards that are pressure measurers (e.g., manometers) since the pressure may drift during the time required to record five repeat readings of the transfer standard (~5 min). This may not be an issue for pressure standards that are pressure generators (e.g., volume expansion systems) provided their temperature and hence the generated pressure does not change during repeat readings of the transfer standard.

The laboratories should operate their pressure standards at their normal operating temperature. Thermal transpiration corrections will be made by the pilot laboratory to account for small temperature differences between the participant's pressure standard and the transfer standard. Head corrections shall be referenced to the reference level line indicated on the PTP. NOTE: It is the participant NMI's responsibility to make all corrections specific to your NMI's standard (Head correction, Mercury Vapor Pressure, etc.) prior to sending data to the Pilot lab for analysis. It is also required that the k = 1 uncertainty of each participant's standard is clearly reported at each pressure.

For each set of associated readings of the transfer standard and the participant's pressure standard

{NO, CDG 1(Pa), CDG 2(Pa), T(°C), P_{REF}(Pa)}, [T_{STD}(°C), P_{STD}(Pa), U_{STD}(Pa)]

Where { } indicates data recorded by the notebook computer and also made available on GPIB bus, [ ] indicates data that must be recorded and provided by the participating laboratory. P_{REF} is the reference pressure (vacuum) reading from the reference vacuum gauge controller/display on the SEP and T is the reading of the thermometer located in the PTP (monitors temperature stability). P_{STD} and T_{STD} are the pressure reading and temperature of the participant's pressure standard, and U_{STD} is the uncertainty of the participant's pressure standard (k = 1) in Pa.

• Ten repeat readings of the transfer standard at zero pressure with isolation valves V_1 and V_3 closed and valve V_2, V_4 and V_5 open.

• Five repeat readings of the transfer standard and the pressure standard at each cardinal point pressure, nominally at 1, 3, 10, ... Pa.

• Optional After the first cardinal point pressure, five repeat zero pressure readings of the transfer standard and the pressure standard prior to each subsequent cardinal point pressure.

• Ten repeat readings of the transfer standard after returning to zero pressure at the end of the calibration run with isolation valves V_1 and V_3 closed and valves V_2, V_4 and V_5 open.

6. CORRECTION AND ANALYSIS OF THE REPORTED DATA

The correction and analysis of the key comparison data required that the following factors be addressed:

· Zero-pressure offsets (Section 6.1)

· Thermal transpiration effects (Section 6.2)

· Deviations from target pressures (Section 6.3)

· Calculation of the predicted gauge readings (Section 6.4)

Additionally, the methods for estimating uncertainties (Sections 6.5) and evaluating degrees of equivalence (Section 6.6) are also described. All of these factors were determined by the pilot lab while the participants were responsible for all correction factors individual to their standard (i.e. head correction, Hg vapor pressure, etc.). In addition, the variables used in this report are summarized in Table. 4

Table 4. Variables used in this report

Variables	Definition	Variables	Definition
p'_{rl}	The corrected reading for data point *l* at a given target pressure during calibration run *r*	p_{rl}	The uncorrected gauge reading for data point *l* at a given target pressure during calibration run *r*
$P'_{NMI(j)rl}$	The corrected reading of a NMI's laboratory standard for data point *l* at a given target pressure during calibration run *r*	$P_{NMI(j)rl}$	The uncorrected pressure measured/generated by the laboratory standard operating at absolute temperature $T_{NMI(j)rl}$ at a given target pressure during calibration run *r*
T_{PTPrl}	The absolute temperature (K) of the transfer standard gauge	b_{jr}	The mean ratio of five repeated readings of the transfer standard gauge and NMI laboratory standard *j* at each target pressure during calibration run *r*
p_j	The gauge readings for each pressure measured/generated by laboratory standard *j* at all target pressures	$u_c(p_j)$	The combined uncertainty in the normalized gauge readings
$u_{std}(p_j)$	The Type B uncertainty in p_j due to systematic effects in laboratory standard *j* which is reported by each NMI	$u_{rdm}(p_j)$	The Type A uncertainty in p_j due to the combined effect of short-term random errors of transfer standard gauge and laboratory standard *j* during calibration
$u_{lts}(p_j)$	The uncertainty arising from long-term shifts in the response function of the gauge during the course of the	D_j	The degree of equivalence of the laboratory standard *j* relative to a $KCRV_{APMP}$

	comparison		
$D_{ccm2012}$	The degree of equivalence defined in the report of CCM.P-K4 2012		

6.1. CORRECTIONS FOR ZERO-PRESSURE OFFSETS

The first step was to correct the readings of each gauge, for their zero-pressure offset. At a given target pressure during calibration run *r,* the corrected reading for data point *l* is given by:

$$p'_{rl} = p_{rl} - \langle p_r \rangle_{10} \tag{1}$$

where p_{rl} is the uncorrected gauge reading and $\langle p_r \rangle_{10}$ is the mean of 10 zero-pressure readings taken prior to the start of calibration run *r*.

6.2. CORRECTIONS FOR THERMAL TRANSPIRATION EFFECTS

The difference in temperature of a laboratory standard and the transfer standard gauges can give rise to significant thermal transpiration effects at low pressures [2]. In the present comparison the magnitude of this effect will vary since the laboratory standards were operated at somewhat different temperatures (see Figure 6). The effect of different operating temperatures was minimized by the same method as used in the previous CCM key comparison (CCM.P-K4 2012, [1])

At a given target pressure during calibration run *r*, the corrected reading of a NMI's laboratory standard for data point *l* is given to a good approximation by the Takaishi-Sensui equation [3]:

$$P'_{NMI(j)rl} = P_{NMI(j)rl} f_{tt} = P_{NMI(j)rl} \frac{(aY^2+bY+cY^{0.5}+1)}{(aY^2+bY+cY^{0.5}+\left(\frac{T_{NMI(j)_{rl}}}{T_{PTP_{rl}}}\right)^{0.5})} \tag{2a}$$

where for each NMI j, $P_{NMI(j)rl}$ represents the uncorrected pressure measured/generated by the laboratory standard operating at absolute temperature $T_{NMI(j)rl}$, T_{PTPrl} is the absolute temperature (K) of the transfer standard gauge, a, b, and c, are temperature-independent gas-species dependent constants, and the parameter Y is defined as

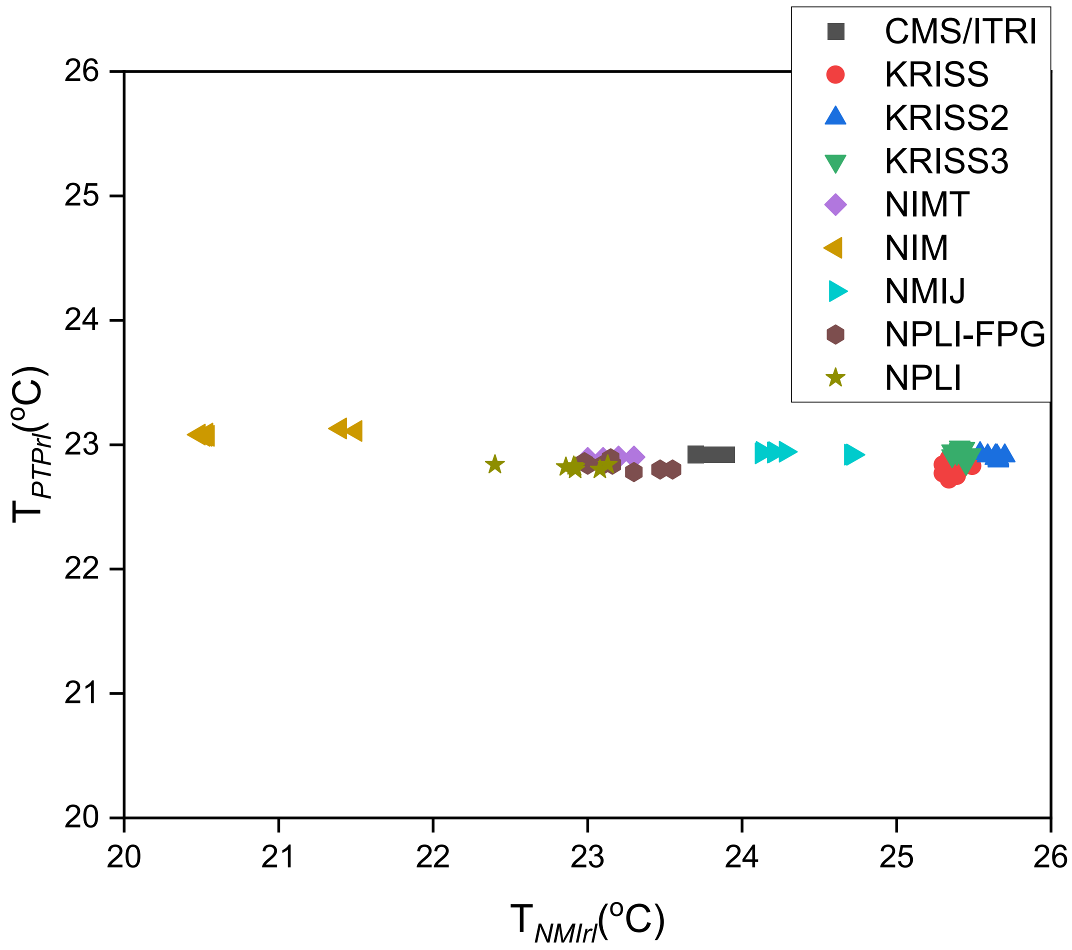


Figure 6. Temperature of laboratory standards vs temperature of transfer standard.

$$Y = \frac{p_{NMI(j)rl} \times d}{133.322\ (\frac{T_{NMI(j)rl} + T_{PTPrl}}{2})} \qquad (2b)$$

and d is the internal diameter of the gauge inlet tubing in mm. The interior temperature of the transfer standard enclosure as measured by the PRT is assumed to closely approximate the temperature of each gauge. It can be seen in Figure 6 that the temperature of the transfer standards was significantly more uniform than the operating temperature of the laboratory standards at different NMIs.

The corrections to the measured/generated pressures (factors f_{tt}) are based on d = 4.3 mm and the following reported values for nitrogen: $a = 1.2 \times 10^6$, $b = 1.0 \times 10^3$, and c = 14 [2,3]. The corrections are largest at lowest pressures and are small, but significant for this comparison. For example, at 1 Pa the value of f_{tt}, from equation (2a), ranged from 0.9909 to 1.0016. Because of the small size of the correction, and the estimated uncertainty of this equation $U_{tt} < 5$ %, the component of uncertainty on pressure is negligible.

6.3. CALCULATION OF CALIBRATION RATIOS

The transfer standard gauges are nominally linear devices and therefore the ratio of transfer standard reading to laboratory standard reading will be essentially independent of pressure for a range of pressures about each target value. These ratios form the basis for the comparison of laboratory standards from different NMIs.

At each target pressure during calibration run r the mean ratio of five repeated readings of the transfer standard gauge and NMI laboratory standard j is given by

$$b_{jr} = \frac{1}{5}\sum_{l=1}^{5} \frac{p'_{rl}}{P'_{NMI(j)rl}} \tag{3}$$

where p'_{rl} and $P'_{NMI(j)rl}$ are the “simultaneous” readings of the gauge and laboratory standard, respectively. The mean of b_{jr} for five calibration runs defines a *calibration ratio* given by

$$b_j = \frac{1}{5}\sum_{r=1}^{5} b_{jr} = \frac{1}{25}\sum_{r=1}^{5}\sum_{l=1}^{5} \frac{p'_{rl}}{P_{NMI(j)rl'}} \tag{4}$$

6.4. CALCULATION OF THE PREDICTED GAUGE READINGS

Degrees of equivalence [4] of the laboratory standards for absolute pressure can be expressed quantitatively by comparing pressure readings of the transfer standard gauges. The basic method adopted here is to use the calibration ratios to predict gauge readings that would be observed had the target pressure generated been equal for each NMI’s laboratory standard. The difference between the calculated predicted gauge readings for the different laboratory standards is taken as a surrogate for the difference between laboratory standards.

At all target pressures, gauge readings for each pressure measured/generated by laboratory standard j may be expressed as follows:

$$p_j = b_j P_{NMI(j)} = b_j p_t \tag{5}$$

where b_j is the calibration ratio, p_j are their respective pressure readings, $P_{\mathrm{NMI(j)}}$ is the measured/generated pressure using the laboratory standard equal to the target pressure, p_t.

For the pilot laboratory, a single value of p_{KRISS} is calculated as the arithmetic mean of three values of p_{jn}. The values of p_{jn} are determined via Equation (5) using calibration ratios b_{jn} obtained from three calibrations (n = 1, 2, 3) of transfer at the pilot lab (KRISS),

$$p_{KRISS} = \frac{p_{j1}+p_{j2}+p_{j3}}{3} \tag{6}$$

6.5. ESTIMATES OF UNCERTAINTIES IN THE NORMALIZED GAUGE READINGS

The combined uncertainty in the normalized gauge readings calculated using Equation (5) may be estimated from the root-sum-square of three component uncertainties [5, 6],

$$u_c(p_j) = \sqrt{u_{std}(p_j)^2 + u_{rdm}(p_j)^2 + u_{lts}(p_j)^2} \tag{7}$$

where $u_{std}(p_j)$ is the Type B uncertainty in p_j due to systematic effects in the laboratory standard j which is reported by each NMI, $u_{rdm}(p_j)$ is the Type A uncertainty in p_j due to the combined effect of short-term random errors of the transfer standard gauge and the laboratory standard j during calibration, and $u_{lts}(p_j)$ is the uncertainty arising from long-term shifts in the response function of the gauge during the course of the comparison. Figure 7 presents the estimated relative uncertainties in pressure in the laboratory standards, as stated by the participants for target pressures used in the comparison.

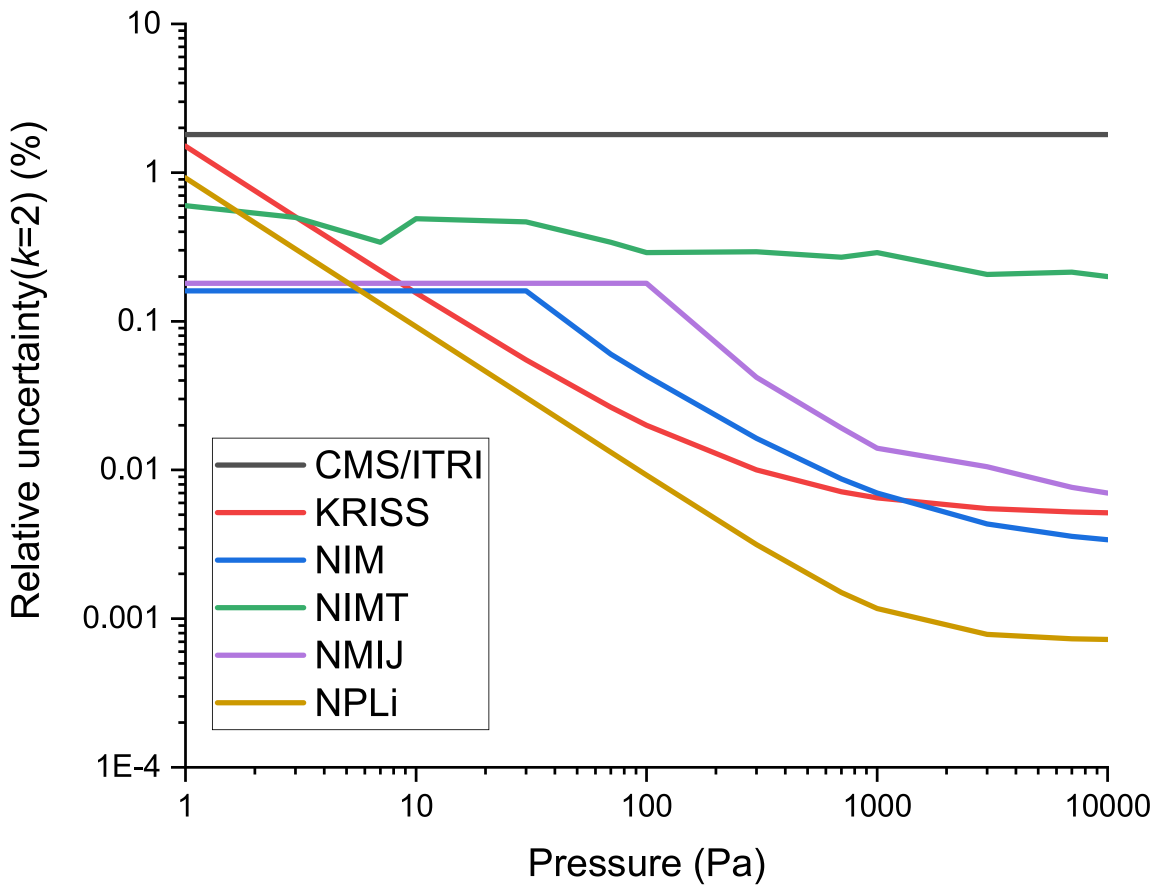


Figure 7. NMI relative uncertainty due to systematic effects in laboratory standards at the participating laboratories as a function of pressure

The relative uncertainty in p_j due to short-term random effects during calibration can be estimated from the corresponding uncertainties in the calibration ratios via Equation (8):

$$\frac{u_{rdm}(p_j)}{p_j} = u_{rdm}(b_j\ ) \tag{8}$$

Similarly, the relative uncertainty in p_{KRISS} due to long-term shifts in the gauge response between calibrations is given by

$$\frac{u_{lts}(p_{KRISS})}{p_{KRISS}} = u_{lts}(b_{KRISS}) \tag{9}$$

The short-term random uncertainty in a calibration ratio, b_j, as given by Equation (4), was estimated by a Type A evaluation as

$$u_{rdm}(b_j\ ) = \frac{\sigma_{jr}}{\sqrt{5}} \tag{10}$$

where σ_{jr} is the standard deviation of five means, b_{jr}, about their mean b_j.

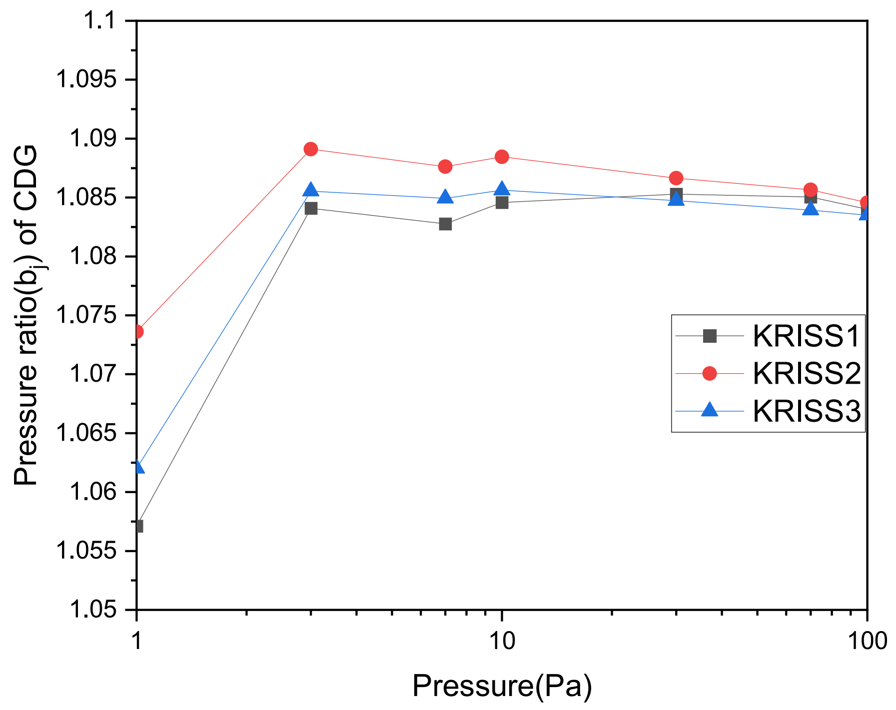
Assuming the observed variability in the gauge response was purely random and considering the small statistical sample of pilot laboratory calibrations (three), a Type B evaluation was used to estimate the uncertainty $u_{lts}(b_{\mathrm{KRISS}})$ for each gauge. At each target pressure, the variation due to long-term shifts was modeled by a normal distribution such that there is a 2 out of 3 chance the calibration ratio lies in the interval between maximum and minimum values of b_j measured during the three calibrations at the pilot laboratory. The standard uncertainty due to this source of error then equals one-half the difference between the maximum and minimum values:

$$u_{lts}(b_{KRISS}) = \frac{(b_{KRISS}\ )_{MAX} - (b_{KRISS}\ )_{MIN}}{2} \tag{11}$$

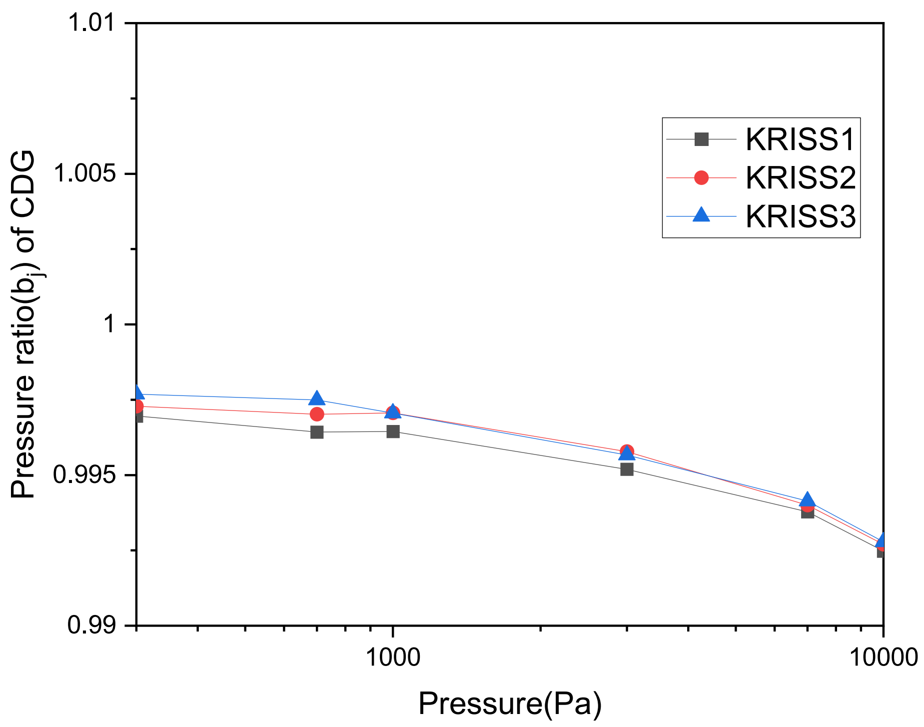
This estimate is unaffected by any systematic bias in the pilot laboratory standard, which would be present in all three calibrations. However, the estimate does assume stability in the laboratory standard at the pilot laboratory and that the observed shifts are attributed to the instability of the gauge.

Figure 8 and Table 5 shows the long-term instability of the transfer standard gauge readings over 2 years during this KC. This plot shows that the long-term stability of the transfer standard over the course of this comparison should be sufficient to resolve any relative biases between different laboratory standards.

The combined uncertainty in the normalized gauge readings, p_j, at each target pressure was estimated by using data from Tables 5 and 7, and Equations (8) to (11), and is given in Table 8.



(a)



(b)

Figure 8. Long-term instability of transfer standard

Table 5. Long-term instability of CDG calibration ratios as measured at the pilot laboratory.

	$u_{lts}(b_j)$	
	CDG1	CDG2
1	0.0082	
3	0.0025	
10	0.0019	
30	0.0010	
100	0.00053	
300		0.00036
1000		0.00031
3000		0.00029
10000		0.00015

6.6. EVALUATION OF DEGREES OF EQUIVALENCE

Before this key comparison in the APMP region, there was an international comparison of CCM.P-K4.2012. Among the NMIs participating in this key comparison, NMIJ participated in CCM.P-K4.2012, and thus in this key comparison, the measured value of NMIJ was used as a key comparison reference value ($KCRV_{APMP}$).

The degree of equivalence of laboratory standard j relative to a $KCRV_{APMP}$ is expressed at each target pressure by two quantities, the deviation of p_j from the reference value p'_{NMIJ}

$$D_j = p_j - p_{KCRV_{APMP}} = p_j - p'_{NMIJ} \tag{12}$$

And p'_{NMIJ} is related to a key comparison reference value ($KCRV_{CCM}$) as follows :

$$D_{CCM2012} = p_{NMIJ2012} - p_{KCRV_{CCM}} \tag{13}$$

where $D_{CCM2012}$ is defined as given in the report of CCM.P-K4 2012 (equation (21) in ref [1]).

Hence, eq.(12) becomes

$$D_j = p_j - p'_{NMIJ} + D_{ccm2012} \tag{14}$$

The expanded uncertainty of this deviation is estimated from the following equation

$$U_j = ku_c(D_j) = k\sqrt{u_c(p_j)^2 + u_c(D_{ccm2012})^2 + u_{rdm}(p_{NMIJ})^2} \tag{15}$$

where u_c (D_j) is the combined standard uncertainty of this deviation, k is the coverage factor (k = 2 was used to represent the expanded uncertainty), u_c (p_j) is the combined uncertainties given by Equations (7), u_c ($D_{ccm2012}$) is the combined uncertainties of deviation for NMIJ defined in ref. [1] and the evaluation of expanded uncertainty of this deviation was discussed in Ref. [8](Table 6). According to the reference, the expanded uncertainty of u_{rdm} (p_{NMIJ}) is the type A uncertainty of NMIJ.

Table 6. Summary of the $D_{CCM2012}$ and $u_c(D_{CCM2012})$

Target Pressure (Pa)	$D_{CCM2012}$ (Pa)	$u_c(D_{CCM2012})$ (Pa)
1	0.0010	0.0070
3	0.0010	0.0075
10	0.0050	0.0240
30	0.0130	0.0675
100	0.0040	0.0295
300	0.0170	0.0855
1000	0.0770	0.1940
3000	-0.0800	0.1550
10000	0	0.3100

7. RESULTS FOR KEY COMPARISON APMP.M.P-K4

7.1. COMPARISON OF NORMALIZED GAUGE READINGS

Table 7 presents a summary of the normalized gauge readings, p_j, obtained from the participating laboratories as a function of nominal target pressures. The calibration ratios for the CDGs were calculated using Equations (3), and are given in the third and the fourth column. Values of p_j, which were calculated via Equation (5), are given in the fifth and sixth columns.

Table 7. Summary of key comparison results for measured calibration ratios, b_j, and calculated values for normalized readings of gauge, p_j, at the target pressure

NMI	Nominal Pressure (Pa)	Mean Ratio		Normalized Gauge Reading (Pa)	
		CDG1	CDG2	CDG1	CDG2
		bj	bj	p_j	p_j
CMS/ITRI	1	1.1006		1.1006	
	3	1.0981		3.2942	
	10	1.0917		10.9171	
	30	1.0882		32.6458	
	100	1.0856		108.55622	

	300		1.0084		302.5235
	1000		1.0041		1004.0676
	3000		0.9971		2991.4349
	10000		0.9938		9937.7581
KRISS	1	1.0642		1.0642	
	3	1.0862		3.2587	
	10	1.0862		10.8622	
	30	1.0855		32.5657	
	100	1.0840		108.4008	
	300		0.9973		299.1799
	1000		0.9969		996.6685
	3000		0.9955		2986.5673
	10000		0.9927		9927.3727
NIM	1	1.0893		1.0893	
	3	1.0905		3.2715	
	10	1.0902		10.9018	
	30	1.0854		32.5615	
	100	1.0846		108.4595	
	300		0.9968		299.0341
	1000		0.9965		996.5064
	3000		0.9957		2987.1198
	10000		0.9932		9931.5332
NIMT	1	1.0918		1.0918	
	3	1.0897		3.2692	
	10	1.0880		10.8803	
	30	1.0854		32.5617	
	100	1.0836		108.3648	
	300		0.9975		299.2550
	1000		0.9966		996.5825
	3000		0.9960		2988.1405
	10000		0.9933		9933.1760
NMIJ	1	1.0897		1.0897	
	3	1.0879		3.2636	
	10	1.0861		10.8605	
	30	1.0849		32.5472	

	100	1.0838		108.3774	
	300		0.9975		299.2647
	1000		0.9969		996.8551
	3000		0.9957		2986.9828
	10000		0.9928		9928.3432
NPLI	1	1.0558		1.0558	
	3	1.0859		3.2577	
	10	1.0853		10.8528	
	30	1.0861		32.5828	
	100	1.0845		108.4464	
	300		0.9966		298.9919
	1000		0.9961		996.1274
	3000		0.9953		2985.8712
	10000		0.9926		9926.2028

Table 8. Summary of key comparison uncertainties due to type B uncertainty $u_{std}(p_j)$, the random effects, $u_{rdm}(b_j)$, and the combined uncertainty $u_c(p_j)$.

NMI	Nominal Pressure (Pa)	$u_{std}(p_j)$ (Pa)	$u_{rdm}(b_j)$		$u_c(p_j)$ (Pa)	
			CDG1	CDG2	CDG1	CDG2
CMS/ITRI	1	0.0060	0.0042		0.0110	
	3	0.0150	0.0013		0.0172	
	10	0.0480	0.0003		0.0518	
	30	0.1400	0.0001		0.1429	
	100	0.3500	0.0002		0.3602	
	300	1.1000		0.0005		1.1138
	1000	3.4000		0.0001		3.4157
	3000	9.8000		0.0001		9.8406
	10000	32.0000		0.0003		32.1976
KRISS	1	0.0075	0.0025		0.0114	
	3	0.0076	0.0005		0.0108	
	10	0.0078	0.0003		0.0211	
	30	0.0083	0.0001		0.0299	
	100	0.0102	0.00004		0.0837	
	300	0.0150		0.0003		0.1338

	1000	0.0324		0.00005		0.3131
	3000	0.0822		0.00002		0.8828
	10000	0.2557		0.00002		1.5424
NIM	1	0.0009	0.0023		0.0086	
	3	0.0027	0.0013		0.0089	
	10	0.0090	0.0009		0.0232	
	30	0.0269	0.0001		0.0393	
	100	0.0215	0.0001		0.0859	
	300	0.0245		0.0001		0.1165
	1000	0.0350		0.00003		0.3115
	3000	0.0650		0.00005		0.8910
	10000	0.1700		0.00004		1.5715
NIMT	1	0.0030	0.0003		0.0088	
	3	0.0073	0.0002		0.0105	
	10	0.0244	0.0001		0.0312	
	30	0.0682	0.00005		0.0739	
	100	0.1470	0.00002		0.1688	
	300	0.4410		0.0001		0.4564
	1000	1.4700		0.0001		1.5028
	3000	3.0958		0.00002		3.2180
	10000	10.0292		0.00001		10.1424
NMIJ	1	0.0009	0.0006		0.0083	
	3	0.0027	0.0003		0.0081	
	10	0.0090	0.0001		0.0214	
	30	0.0270	0.00004		0.0393	
	100	0.0750	0.0002		0.1136	
	300	0.0750		0.0001		0.1357
	1000	0.1000		0.00003		0.3250
	3000	0.1600		0.00002		0.8935
	10000	0.3500		0.00002		1.5607
NPLI	1	0.0050	0.0027		0.0100	
	3	0.0050	0.0010		0.0096	
	10	0.0050	0.0004		0.0204	
	30	0.0050	0.0002		0.0294	
	100	0.0050	0.00005		0.0833	

	300	0.0051		0.0001		0.1173
	1000	0.0062		0.00003		0.3098
	3000	0.0119		0.00001		0.8778
	10000	0.0363		0.000006		1.5108

Table 8 summarizes the uncertainty calculations for the participating laboratories as a function of pressure. Uncertainties in the ratios due to Type A effects, which were obtained by means of Equation (10), are given in the third and fourth columns. The combined standard uncertainties, $u_c(p_j)$, which were calculated according to Equation (7), are given in the fifth and sixth columns.

7.2. DEGREES OF EQUIVALENCE OF THE LABORATORY STANDARDS

These values are used to compare the results of the comparison. Table 9 presents a summary of the final results for the participant NMIs as a function of nominal target pressures. The columns present degrees of equivalence of the measurement standards expressed quantitatively deviations from KCRV. The deviations from KCRV, D_j, were calculated via Equation (14). The expanded uncertainties of these deviations, U_j, were calculated using Equation (15). The gray shaded cells in Table 9 indicate pressures at which the condition for equivalence at a $k = 2$ level of confidence is not satisfied, that is, where $|D_j| > U_j$.

The degrees of equivalence of individual NMIs with respect to key comparison reference values are presented graphically in Figures 9 to 17 as plots of deviations, D_j , versus NMI . Uncertainty bars are the values of U_j, at a k = 2 confidence interval.

Table 9. Degrees of equivalence expressed in two ways, equivalence of NMIs relative to key comparison reference values. The shaded cells indicate apparent nonequivalence of results for which $|Dj|$ exceeds $|Uj|$

NMI	Nominal Pressure (Pa)	*KCRV* (Pa)	
		Dj	U_j
CMS/ITRI	1	0.0120	0.0262
	3	0.0316	0.0377
	10	0.0615	0.1143
	30	0.1116	0.3161
	100	0.1888	0.7239
	300	3.2758	2.2351
	1000	7.2895	6.8427
	3000	4.3722	19.6841
	10000	9.4149	64.3992
KRISS	1	-0.0244	0.0268

	3	-0.0038	0.0263
	10	0.0067	0.0639
	30	0.0315	0.1477
	100	0.0275	0.1819
	300	-0.0678	0.3234
	1000	-0.1096	0.7388
	3000	-0.4955	1.7966
	10000	-0.9706	3.1682
NIM	1	0.0006	0.0222
	3	0.0089	0.0233
	10	0.0463	0.0667
	30	0.0274	0.1563
	100	0.0862	0.1860
	300	-0.2136	0.2955
	1000	-0.2717	0.7360
	3000	0.0571	1.8128
	10000	3.1900	3.2248
NIMT	1	0.0031	0.0225
	3	0.0067	0.0259
	10	0.0248	0.0787
	30	0.0275	0.2002
	100	-0.0086	0.3451
	300	0.0073	0.9308
	1000	-0.1956	3.0311
	3000	1.0778	6.4447
	10000	4.8327	20.2977
NMIJ	1	0.0010	0.0218
	3	0.0010	0.0221
	10	0.0050	0.0643
	30	0.0130	0.1562
	100	0.0040	0.2382
	300	0.0170	0.3267
	1000	0.0770	0.7590
	3000	0.0800	1.8178
	10000	0.0000	3.2038

NPLI	1	-0.0329	0.0245
	3	-0.0048	0.0244
	10	-0.0027	0.0631
	30	0.0486	0.1473
	100	0.0731	0.1812
	300	-0.2558	0.2969
	1000	-0.6507	0.7331
	3000	-1.1916	1.7869
	10000	-2.1404	3.1066

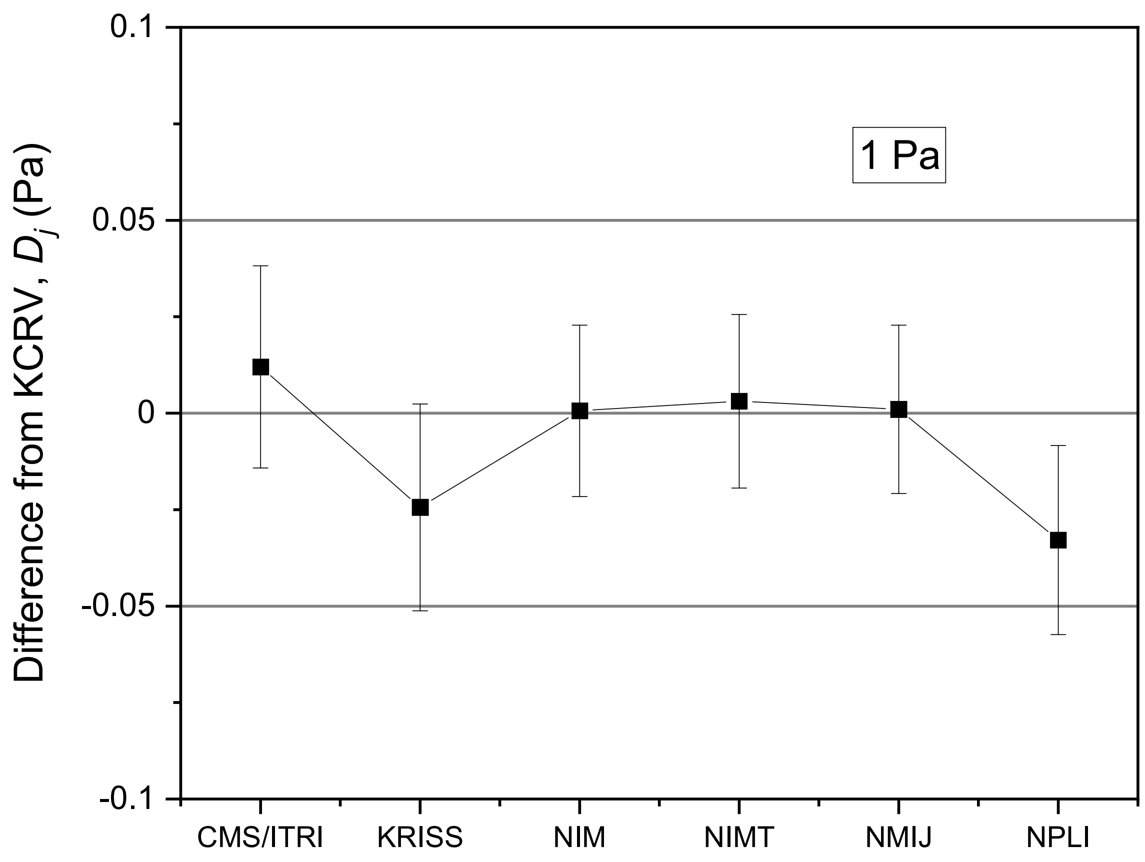


Figure 9. Deviations of corrected mean gauge readings from the key comparison reference values at 1 Pa. Uncertainty bars refer to combined expanded (k = 2) uncertainties of agreement between the NMI and KCRV.

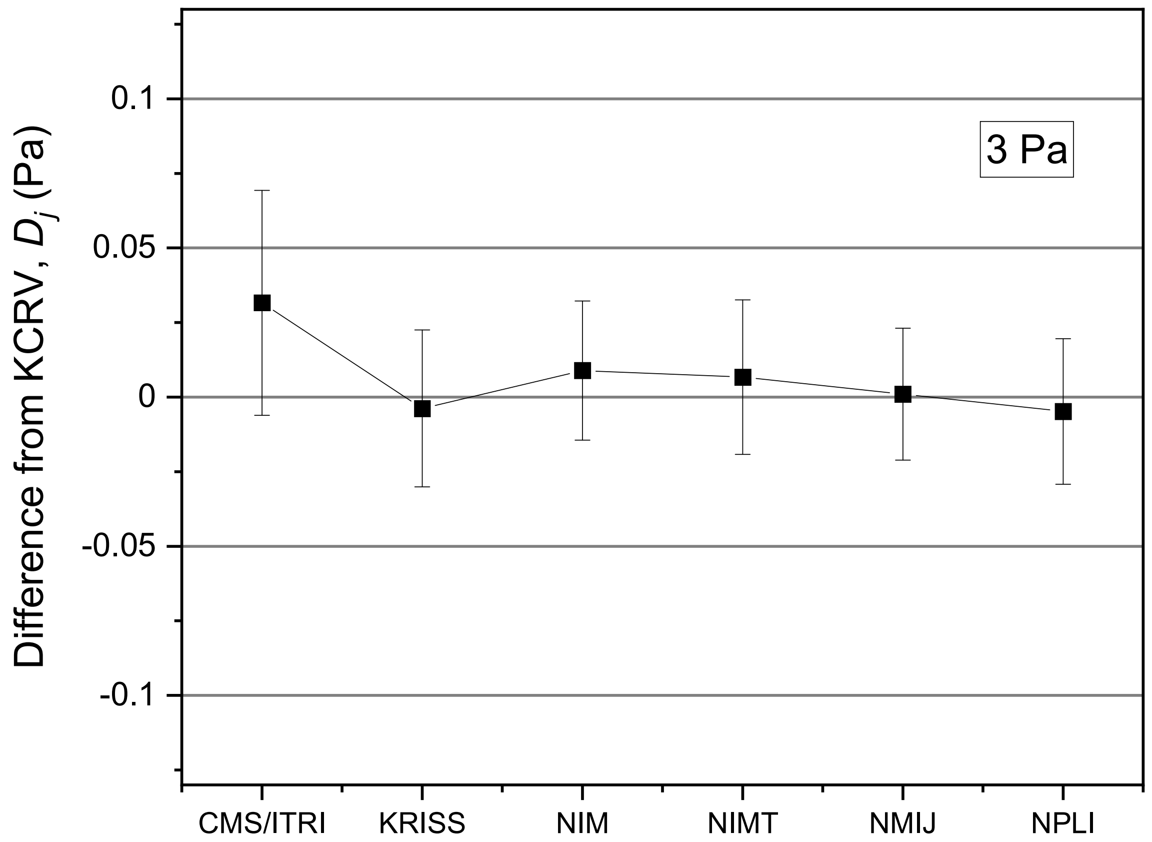


Figure 10. Deviations of corrected mean gauge readings from the key comparison reference values at 3 Pa. Uncertainty bars refer to combined expanded (k = 2) uncertainties of agreement between the NMI and KCRV.

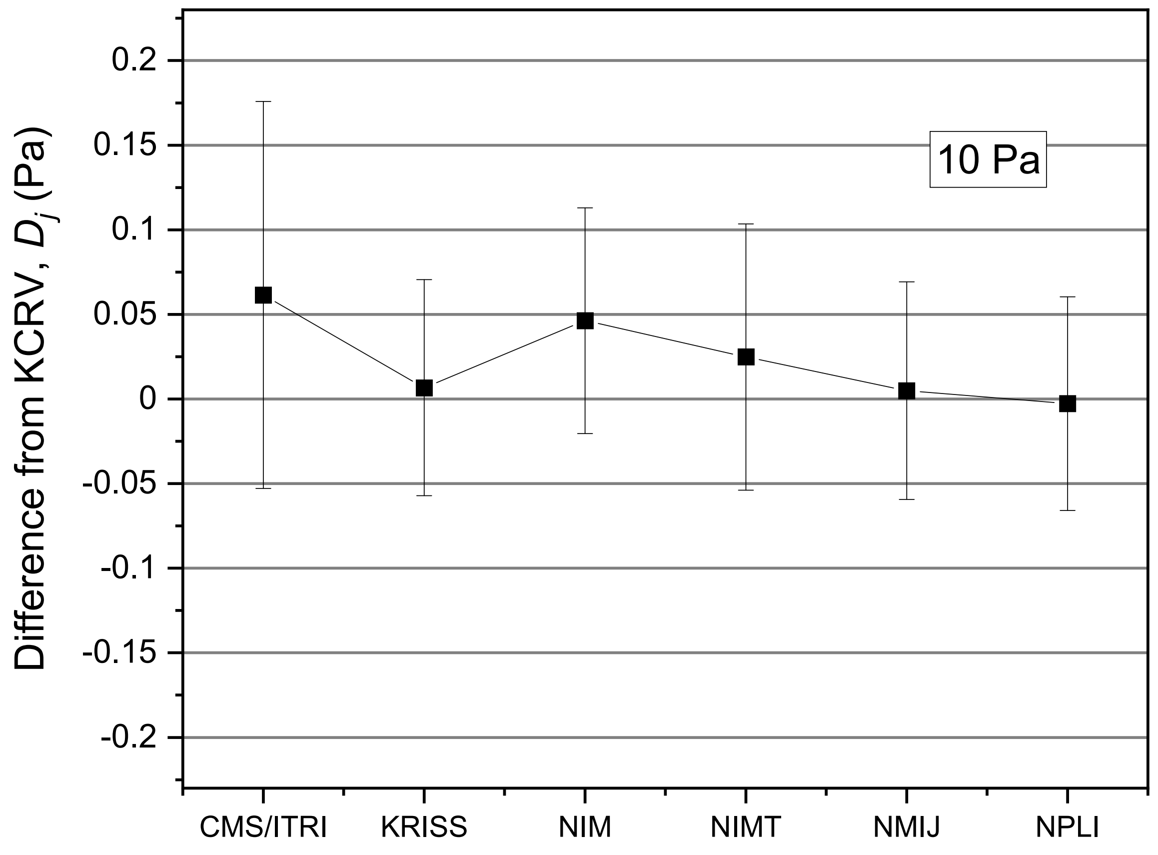


Figure 11. Deviations of corrected mean gauge readings from the key comparison reference values at 10 Pa. Uncertainty bars refer to combined expanded (k = 2) uncertainties of agreement between the NMI and KCRV.

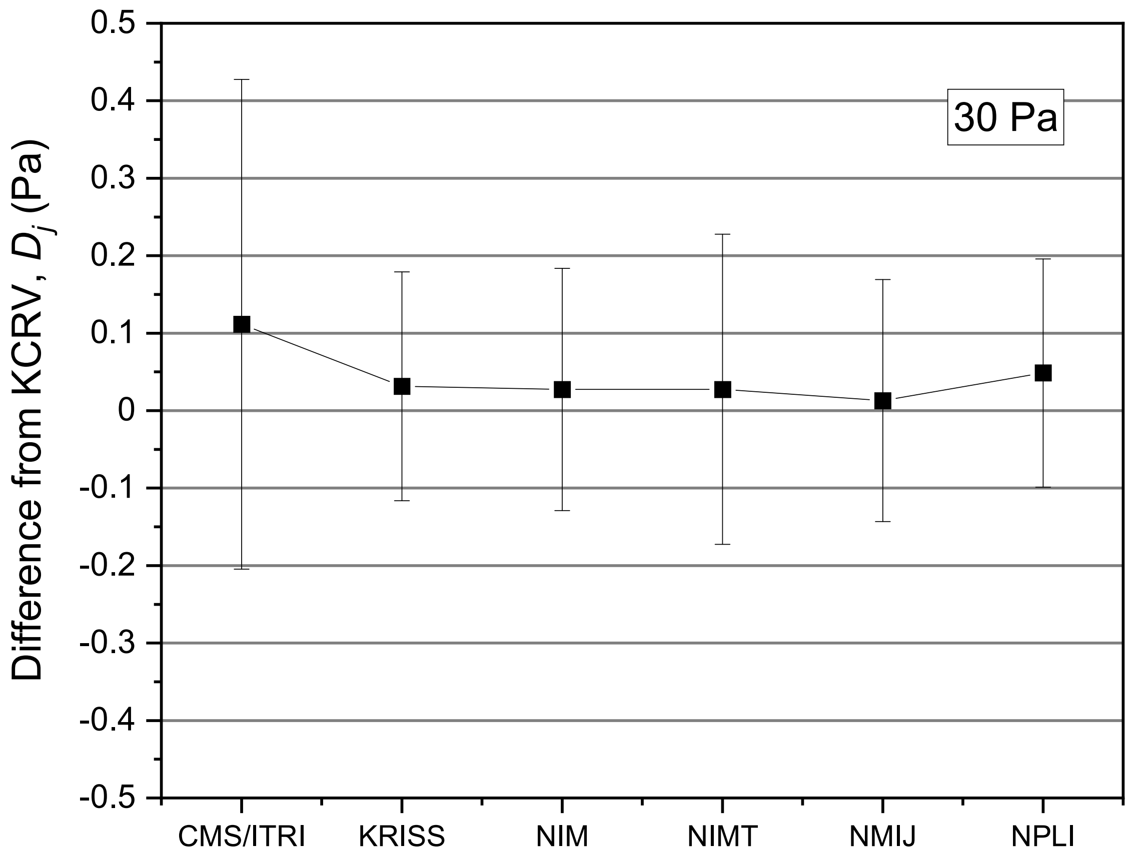


Figure 12. Deviations of corrected mean gauge readings from the key comparison reference values at 30 Pa. Uncertainty bars refer to combined expanded (k = 2) uncertainties of agreement between the NMI and KCRV.

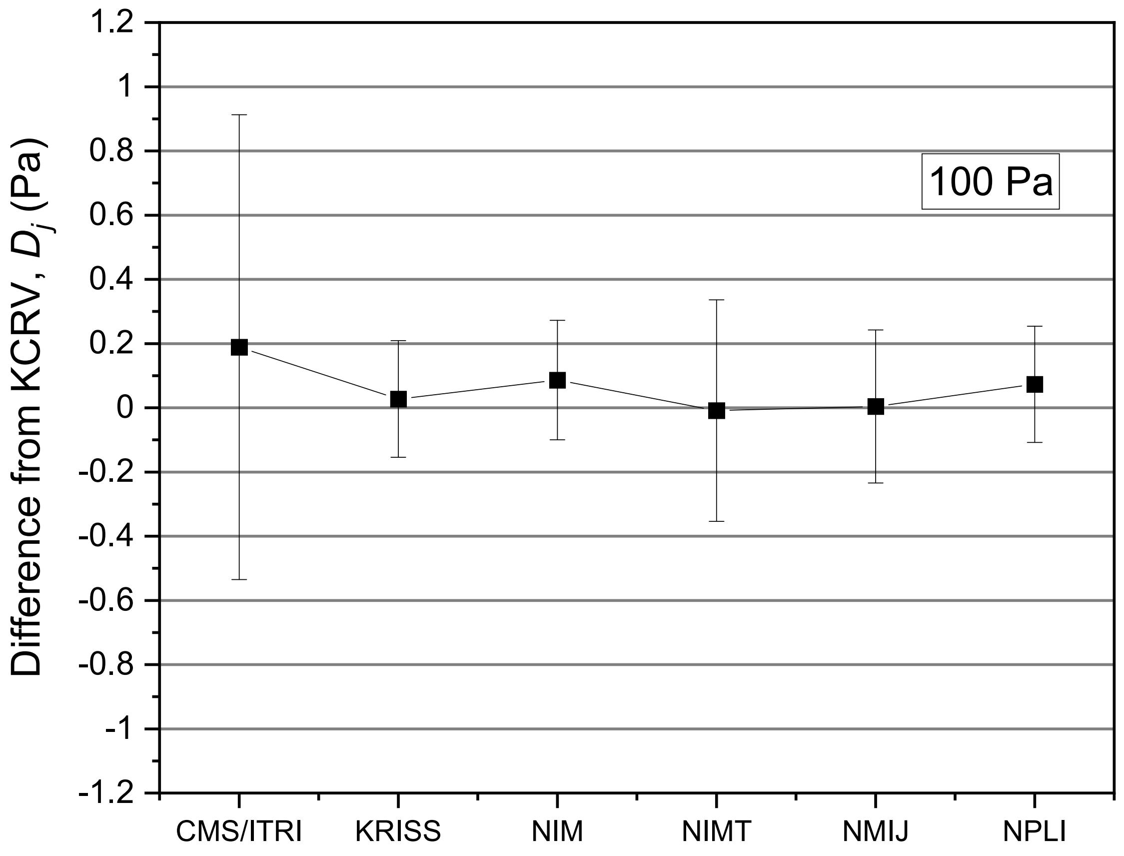


Figure 13. Deviations of corrected mean gauge readings from the key comparison reference values at 100 Pa. Uncertainty bars refer to combined expanded (k = 2) uncertainties of agreement between the NMI and KCRV.

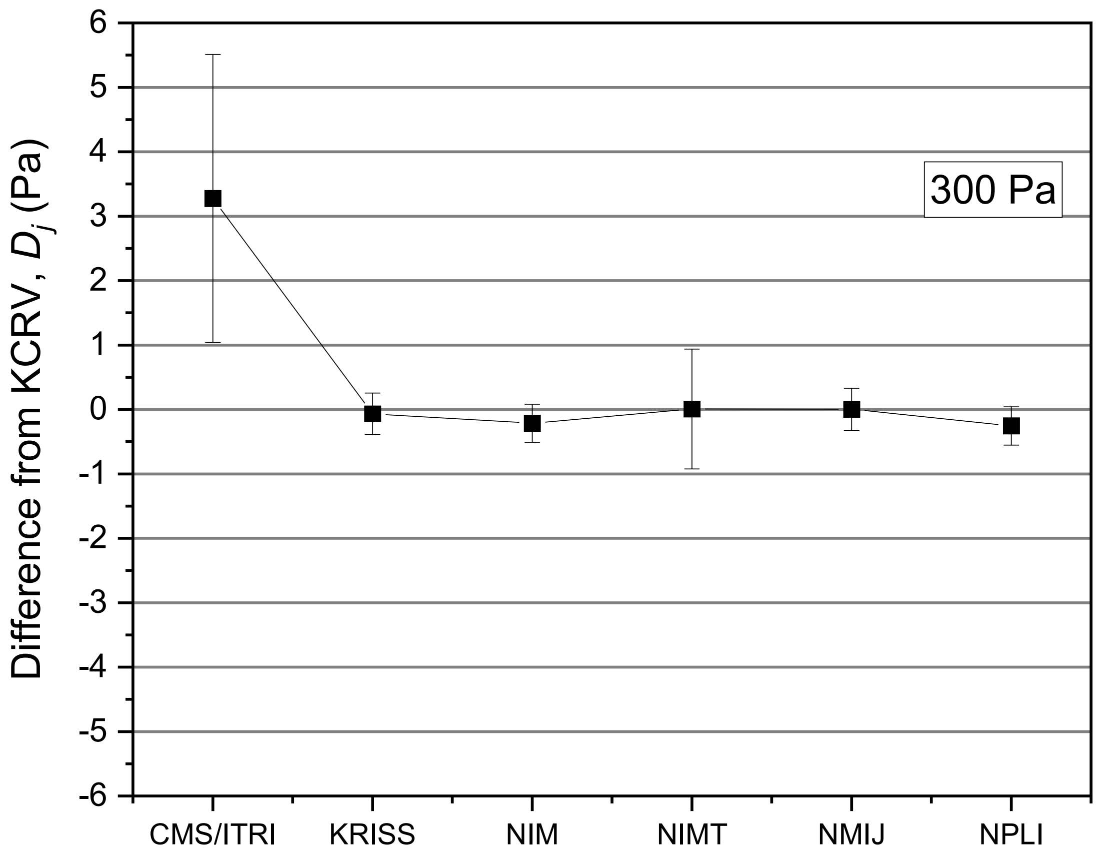


Figure 14. Deviations of corrected mean gauge readings from the key comparison reference values at 300 Pa. Uncertainty bars refer to combined expanded (k = 2) uncertainties of agreement between the NMI and KCRV.

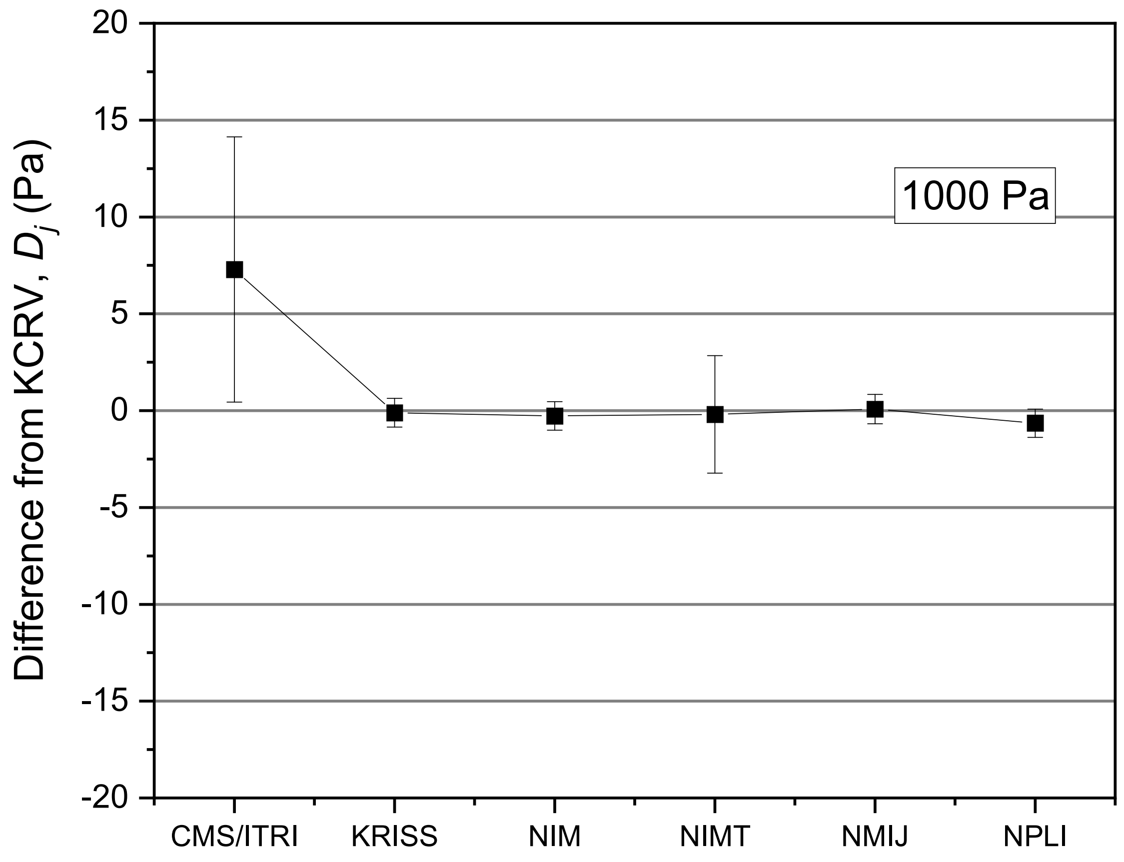


Figure 15. Deviations of corrected mean gauge readings from the key comparison reference values at 1000 Pa. Uncertainty bars refer to combined expanded (k = 2) uncertainties of agreement between the NMI and KCRV.

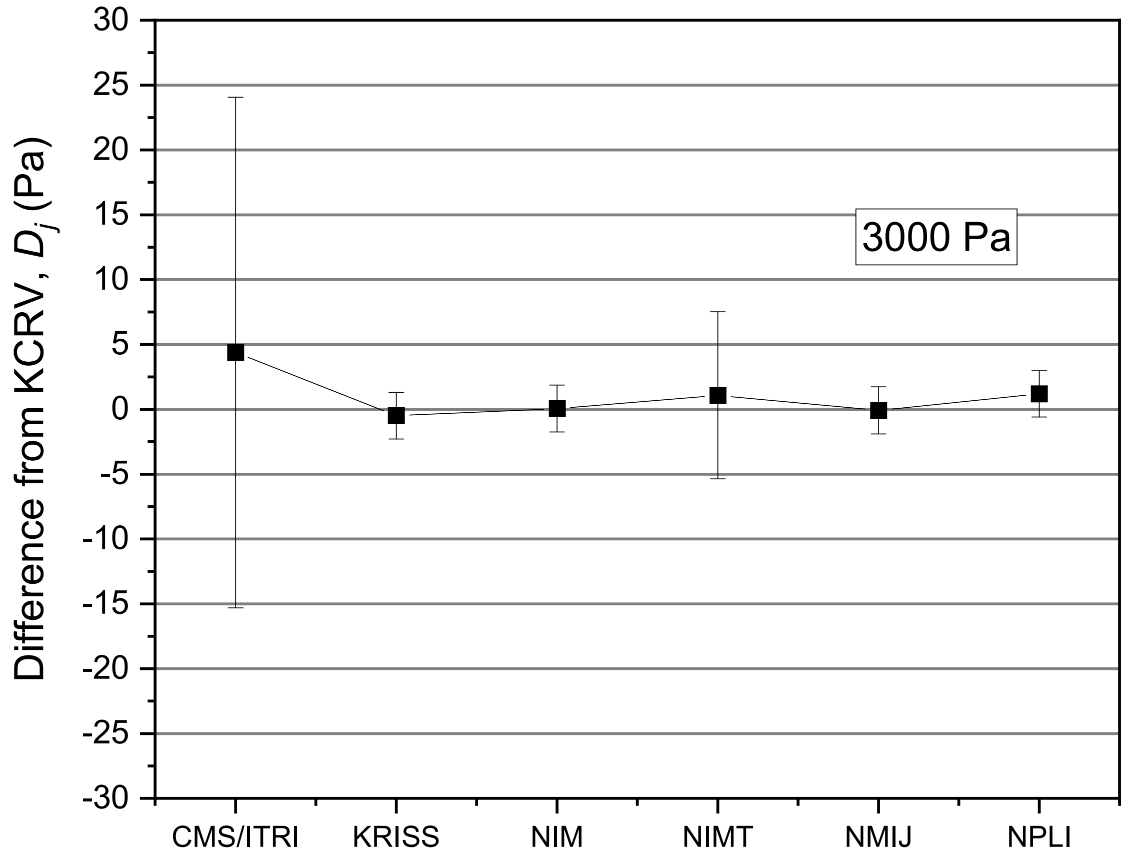


Figure 16. Deviations of corrected mean gauge readings from the key comparison reference values at 3000 Pa. Uncertainty bars refer to combined expanded (k = 2) uncertainties of agreement between the NMI and KCRV.

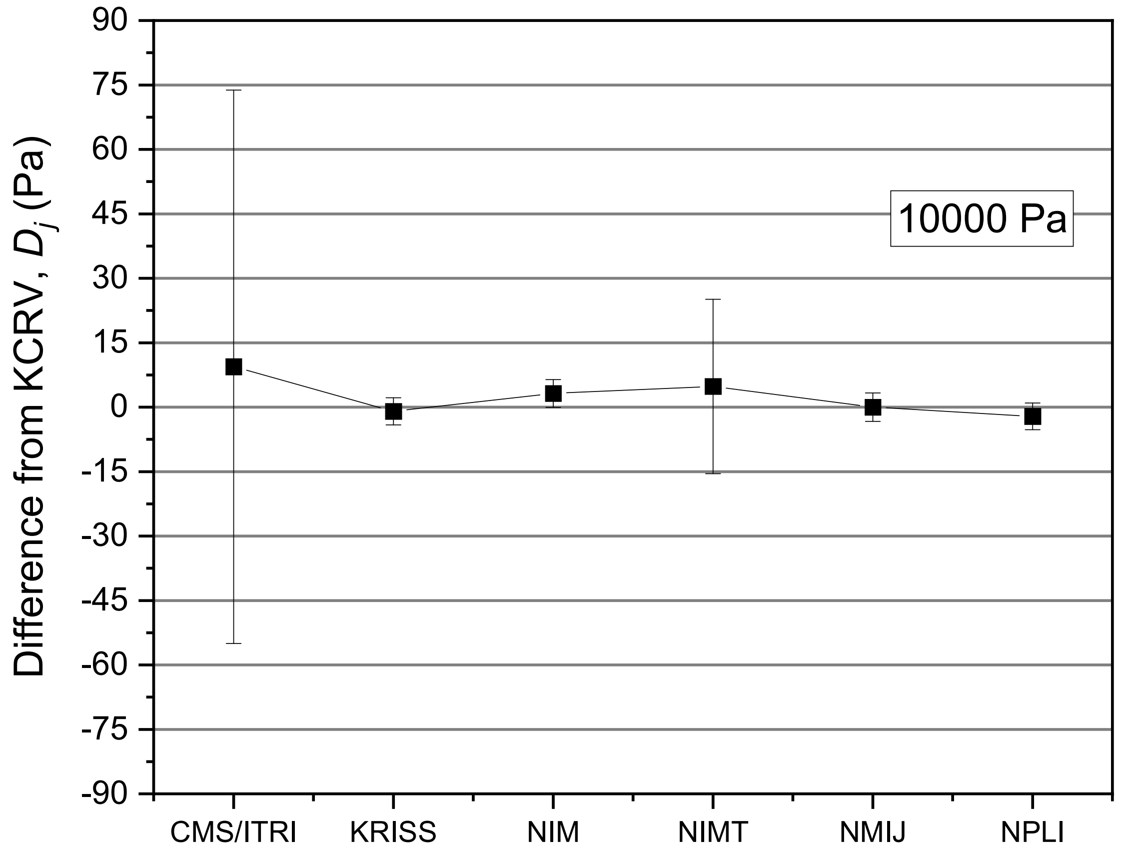


Figure 17. Deviations of corrected mean gauge readings from the key comparison reference values at 10000 Pa. Uncertainty bars refer to combined expanded (k = 2) uncertainties of agreement between the NMI and KCRV.

8. CONCLUSIONS AND DISCUSSION

Overall the comparison was a success, with the transfer package performing well and providing stable artifacts to compare laboratory standards. Among the comparisons outside the stated uncertainty, several have a marginal nonequivalence.

In the present comparison, the degrees of equivalence of the measurement standards were expressed quantitatively in the deviation of corrected mean gauge readings from a key comparison reference value. Dj represents the deviation of the "true" pressure realized by the primary standard j from the corresponding key comparison reference value. However, Dj is not necessarily equal to the deviation of the primary standard j from the SI value. Although the key comparison reference value is likely to be a close approximation to the SI value, it is possible that some of the results (corrected mean gauge readings) from individual NMIs may be even closer.

In the MRA the term 'degree of equivalence of measurement standards' is taken to mean the degree to which a standard is consistent with a key comparison reference value or with a measurement standard at another laboratory. A measure of the degree of consistency is provided by the relative magnitudes of the deviation Dj and its uncertainty, Uj. The shaded cells in Table 7 indicate pressures at which results do not satisfy the condition $| Dj | < Uj$.

Finally, the key comparison established the degrees of equivalence of absolute-pressure standards at six NMIs, both with respect to key comparison reference values as well as between pairs of measurement standards. Apart from results from one NMI that were identified as outliers, the absolute pressure standards of the participating NMIs were generally found to be equivalent.

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