ASIA-PACIFIC METROLOGY PROGRAMME 20 MPa GAS PRESSURE INTERLABORATORY COMPARISON Comparison Identifier: APMP.M.P-S9

Final Report on Supplementary Comparison APMP.M.P-S9 in Gas Gauge Pressure from 2 MPa to 20 MPa

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Likit Sainoo¹, Chaveng Khamnounsak¹, Tasanee Priruenrom¹, Wladimir Sabuga², Oliver Ott², Radley F. Manalo³, Maryness I. Salazar³

¹ NIMT (Pilot institute): National Institute of Metrology (Thailand), 3/4 - 5 Moo 3, Klong 5, Klong Luang, Pathumthani 12120, Thailand

² PTB: Physikalisch-Technische Bundesanstalt, Bundesallee 100, 38116 Braunschweig, Germany
 ³ NMLPHIL: National Metrology Laboratory of the Philippines, Metrology Building, General Santos Avenue, Bicutan, Taguig City, Metro Manila, Philippines

Abstract

This report describes the results of a supplementary comparison of gas pressure standards at three National Metrology Institutes - National Institute of Metrology (Thailand) (NIMT), Physikalisch-Technische Bundesanstalt (PTB) and National Metrology Laboratory of the Philippines (NMLPHIL) - carried out within the framework of Asia-Pacific Metrology Programme (APMP) in order to determine their degrees of equivalence at pressures in the range from 2 MPa to 20 MPa for gauge mode. The pilot institute was NIMT. The measurements were carried out from February 2016 to July 2016. All participating institutes used pressure balances as their pressure standards. A reference pressure monitor was used as a transfer standard. Characteristics of the transfer standard were evaluated at the pilot institute and then used for uncertainty estimations. After completing the comparison measurements, the transfer standard showed a less stable behaviour than expected, resulting in a significant contribution to the uncertainties of the results. The degrees of equivalence that were evaluated in terms of differences of the participant's results and the comparison reference values divided by their expanded uncertainties (k=2) are all within the unity. This demonstrates equivalence of the compared standards on the uncertainty level, which is unfortunately dominated by the uncertainty of the transfer standard.

1. Introduction

The objective of this supplementary comparison (SC) was to check equivalence of gas pressure standards of three National Metrology Institutes (NMIs), National Institute of Metrology (Thailand) (NIMT), Physikalisch-Technische Bundesanstalt (PTB, Germany) and National Metrology Laboratory of the Philippines (NMLPHIL), in the pressure range 2 MPa to 20 MPa in gauge mode. The results of this comparison will be essential to support the submission of calibration and measurement capabilities (CMCs) of the NMIs to the Appendix C of the key comparison database of BIPM.

The pilot laboratory of this SC was NIMT. The transfer standard (TS) was a new reference pressure monitor that belongs to NMLPHIL. It was bought for using as the measurement artifact in this SC.

2. Participating institutes and description of pressure standards

2.1 List of participating institutes

Three NMIs participated in this comparison, including the pilot institute. The NMIs along with their addresses and contact persons are listed in Table 2.1.

No.		Participating Institutes
1	Institute	: National Institute of Metrology (Thailand)
	Acronym	: NIMT (Pilot institute)
	Address	: 3/4 - 5 Moo 3, Klong 5, Klong Luang, Pathumthani 12120, Thailand
	Contact P	Person : Mr. Likit Sainoo and Dr. Tasanee Priruenrom
	Phone	: +66-2577-5100 ext. 2106
	Fax	: +66-2577-3658
	E-mail	: <u>likit@nimt.or.th, tasanee@nimt.or.th</u>
2	Institute	: Physikalisch-Technische Bundesanstalt (Germany)
	Acronym	: PTB
	Address	: Bundesallee 100, D-38116 Braunschweig, Germany
	Contact P	Person : Dr. Wladimir Sabuga, Dr. Oliver Ott and Ms. Silke Schierding
	Phone	: +49 (0) 531 592-3230, +49 (0) 531 592-3344
	Fax	: +49 (0) 531 592-69 3344
	E-mail	: wladimir.sabuga@ptb.de, oliver.ott@ptb.de, silke.schierding@ptb.de
3	Institute	: National Metrology Laboratory- Industrial Technology Development
		Institute (Philippines)
	Acronym	: NMLPHIL
	Address	: Metrology Bldg., Gen. Santos Ave., Bicutan, Taguig City, Metro Manila, Philippines
	Contact P	Person : Mr. Radley F. Manalo, Ms. Maryness I. Salazar
	Phone	: +63-2-837-2071 ext. 2264
	Fax	: +63-2-837-6150
	E-mail	: <u>radleymanalo@yahoo.com</u> , <u>nhet28@yahoo.com</u>

Table 2.1: List of participating institutes

2.2 Description of pressure standards

All the participating institutes used pressure balances series 5500 of DH-Budenberg (France) as their laboratory standards. NIMT and PTB used the 5503 model, whereas NMLPHIL used the 5502 model. The pressure balances were equipped with re-entrant type's piston-cylinder assemblies (liquid-lubricated, gas-operated). Each institute provided the pilot institute with information about its standard that was used to calibrate the transfer standard. Table 2.2 and Table 2.3 list the information about the laboratory standards, including traceability, the pressure balance base, the type and material of piston-cylinder unit (PCU), the method and rotation rate of the piston, the effective area with associated standard uncertainty, the reference temperature, the pressure distortion coefficient with associated standard uncertainty, and the linear thermal expansion coefficient of the PCU.

j	Institute	Traceability	Pressure balan	Pressure balance base		Piston-cylinder		Rotation	
			Manufacture	Model	Туре	Material	Method	[rpm]	
1	NIMT	Independent	DH-	5503	Liquid-	WC/WC	Hand	20 - 30	
		-	Budenberg,		lubricated				
			France		gas type				
					(re-entrant)				
2	PTB	Independent	DH-	5503	Liquid-	WC/WC	Hand	20 - 30	
			Budenberg,		lubricated				
			France		gas type				
					(re-entrant)				
3	NMLPHIL	NIMT	DH-	5502	Liquid-	WC/WC	Hand	24	
			Budenberg,		lubricated				
			France		gas type				
					(re-entrant)				

Table 2.2: Summary of laboratory standards

Table 2.3: Effective area and pressure distortion coefficient of laboratory standards. All uncertainties are expressed as standard ones.

j	Institute	Effective area (A_0)			Ref.	Disto	ortion	Thermal expansion
		at reference temperature (ref. temp.)			temp.	coeffic	ient (λ)	coefficient ($\alpha_p + \alpha_c$)
		Value	Unc.	Unc.	t _r	Value	Unc.	Value
		[m ²]	[m ²]	[10-6]	[°C]	[MPa ⁻¹]	[MPa ⁻¹]	[°C-1]
1	NIMT	1.961120.10-5	$1.70 \cdot 10^{-10}$	8.7	20	9.7·10 ⁻⁷	$1.7 \cdot 10^{-7}$	9.0·10 ⁻⁶
2	PTB	1.960898·10 ⁻⁵	$2.55 \cdot 10^{-10}$	13	20	2.0.10-7	1.0.10-7	1.1.10-5
3	NMLPHIL	1.961190.10-5	3.15.10-10	16	20	6.6·10 ⁻⁷	1.1.10-7	9.0.10-6

3. Transfer standard

A reference pressure monitor that belongs to NMLPHIL was used as a TS for this comparison. The details are listed in Table 3.1 and shown in Figure 3.1. The TS is a product of DH Instruments, Inc., USA.

Name of equipment	Reference pressure monitor	
Manufacturer	DH Instruments, Inc.	
Model	RPM4 A20Ms	
Serial number	2404	
Maximum range	20 MPa	
Resolution	0.01 kPa	
Power supply	85 to 264 VAC, 50/60 Hz	

Table 3.1: Details of transfer standard



Figure 3.1: Gas reference pressure monitor, Model RPM4 A20Ms, S/N 2404

4. Circulation of the transfer standard

According to the protocol, the TS was circulated during the period February to July 2016 with the measurements done at the pilot institute (NIMT) at start and end of the comparison. Table 4.1 presents the actual chronology of measurements in this comparison. The TS was measured first by NIMT. Then it was carried to the participants for measurement by logistic agencies. After returning to NIMT, the TS was calibrated again at the end of comparison in order to confirm that there is no significant drift occurred during its travelling.

 Table 4.1: Chronology of measurements

No.	Period of Measurement	NMIs
1	February, 2016	NIMT
2	April, 2016	NMLPHIL
3	May, 2016	РТВ
4	July, 2016	NIMT

5. Measurement

The general procedure that required each participating institute to measure the transfer standard was described in the Technical Protocol of this comparison.

5.1 Preparation

All participants were required to use clean nitrogen (N_2) as a working fluid. The pressure standard of each participating institute was operated at the normal operating temperature of the institute. The environmental conditions, such as atmospheric pressure, ambient temperature and ambient relative humidity during the calibration were measured using the participant's own devices. For the preparation of the calibration, the following was recommended: (i) At least, 12 hours before starting the measurement procedure, the TS should be connected to a power supply and should be turned on for warming up and stabilization. (ii) The power supply for the TS should be maintained during all the calibrations at the participating institute. (iii) After the installation, the TS should be pressurized using the participant's standard up to the maximum pressure, which is 20 MPa and the leak in the test system should be checked.

5.2 Height difference and head correction

The pressure generated by a pressure standard at the reference level of TS, *P*, is represented by the following equation:

$$P = P_{\rm e} + (\rho_{\rm f} - \rho_{\rm a}) \cdot g_1 \cdot \Delta h \tag{1}$$

where P_e is the pressure generated by the participant's pressure standard at its reference level and $(\rho_f - \rho_a) \cdot g_1 \cdot \Delta h$ is the head correction, in which ρ_f is the density of the working fluid, ρ_a is the air density, g_1 is the local acceleration due to gravity and Δh is the vertical distance between the reference levels of the institute's standard and the TS. Δh is positive if the level of the institute's standard is higher. Each participant had to make an appropriate correction for the height difference between the reference levels of the laboratory standard and the TS and to include its uncertainty contribution in the uncertainty of the applied pressure.

5.3 Measurement method

At nominal target pressures of 0 MPa, 2 MPa, 4 MPa, 6 MPa, 8 MPa, 10 MPa, 12 MPa, 14 MPa, 16 MPa, 18 MPa and 20 MPa, the reference pressure was applied, and the readings of the TS were recorded. The reference pressure and the TS reading values, together with their uncertainties, were the main basis of the comparison.



Figure 5.1: Example of calibration setup

5.3.1 Measurement at 0 MPa

At the beginning and end of each cycle, zero-pressure readings of the TS were recorded. These data were used to correct calibration data for zero-pressure offsets. To apply zero gauge pressure to the TS, the valve V3 was opened (see Figure 5.1). After waiting ten minutes, during next five minutes, 20 readings of the TS were taken, and their average and its corresponding standard deviation, σ , were calculated. The environmental conditions were also measured. These data were recorded and reported by the participants using report sheets provided in Appendix A1 of the technical protocol. An example of the report sheet is shown in Table 5.1.

ſ	Nom	Local	Atm	Atm	Atm	Reading,	R_TS	Applied	$u(P)^{*2}$
	Pres.	Time	Temp.	R.H.	Pres.	[kPa]]	Pressure, P^{*1}	(<i>k</i> =1)
	[MPa]		[°C]	[%]	[kPa]			[kPa]	[kPa]
						Average	σ		
	0	9:00	20.0	55.0	101.3	-0.2	0.1	Not requ	uired
ſ	20	14:30	20.0	55.0	101.3	20000.9	0.2	20000.59	0.30

Table 5.1: Example of measurement results at pressure of 0 MPa and 20 MPa

^{*1} pressure generated by the participant's pressure standard at the reference level of the TS and calculated using equation (1).

*2 standard uncertainty of P(k=1).

5.3.2 Measurement at (2, 4, 6, 8, 10, 12, 14, 16, 18, 20) MPa

The pressure generated by the participant's standard was applied to the transfer standard by closing valve V3 and opening valves V1 and V2. The position of the piston of the pressure balance was kept in the floating range to maintain the applied pressure. After the waiting time of ten minutes for stabilization of the pressure, within the next five minutes, the reading, which was the resulting average of twenty measurements and its corresponding standard deviation σ of TS, was taken. Also, the environmental conditions were recorded. Then, the applied pressure with the associated standard uncertainty at the reference level of the TS was calculated. Any influence quantity having effect on the measurement was appropriately taken into account in the uncertainty estimation by each participant. The correction due to the height difference between the reference levels of the participating institute's standard and the TS was considered. The data were recorded and reported by the participants using report sheets provided in Appendix A1 of the technical protocol. An example is shown in Table 5.1.

5.3.3 Complete measurement cycle

One complete measurement cycle consisted of recording the TS readings for 11 points from 0 MPa to 20 MPa for ascending pressures and 11 points from 20 MPa to 0 MPa for descending pressures as shown in the Figure 5.2. The ascending pressure measurement cycle had to start from 0 MPa, while the descending pressure measurement cycle had to start from 20 MPa. The results of measurement cycle had to be recorded on the measurement result sheet as shown in Appendix A1 of the technical protocol. Totally, for one cycle the 22 measurement points were obtained. One complete measurement cycle was performed in a day. For this comparison three complete measurement cycles were required, with each cycle being on a separate day.



Figure 5.2: Pressure measurement cycle

5.4 Results to be reported

After the measurements were completed at the participating institute, the measurement results were collected into the sheets prepared by the pilot institute and sent to the pilot institute. They included:

- (i) Measured and calculated values at nominal pressures specified, each with the uncertainty in the measurement and the date(s) on which the measurement cycles were performed (3 cycles).
- (ii) Details of the participating institute's standard used in the comparison together with its traceability to the SI unit (shown in Table 2.2).
- (iii) Details of the parameters used for the comparison. These were local gravity and difference in height between the reference levels of the participating institute's standard and the TS together with their measurement uncertainties (shown in Table 5.2).

5.5 Parameters used by each participating institute

The local gravity and the height difference with standard uncertainties of each participating institute are presented in Table 5.2.

Table 5.2: Details of the parameters used by each participating institute. All the uncertainties are expressed as the standard ones.

		Local g	ravity g	Height difference Δh	
j Institute	Institute	Value	Rel. uncertainty	Value	Uncertainty
		(m/s^2)	(10^{-6})	(mm)	(mm)
1	NIMT	9.7831243	0.50	-2.0	5
2	PTB	9.812533	0.54	-10.97	0.7
3	NMLPHIL	9.783551	20	0.0	5

6. Analysis of reported data

Data obtained from one complete measurement cycle consists of the recordings of pressure obtained from the TS, the pressure applied by the laboratory standard and environment parameters

for the 22 points. The 22 points consisted of 11 pressure points from 0 MPa to 20 MPa in steps of 2 MPa in an ascending sequence and 11 pressure points from 20 MPa to 0 MPa in steps of 2 MPa in a descending sequence. Therefore, the following data sets were obtained from the reported results.

$$\{R(j, y, w, i), P(j, y, w, i)\},\$$

where the meanings of the parameters are as follows:

- R = Raw reading of TS, [kPa]
- P = Applied pressure at the reference level of TS by pressure standard *j*, [kPa]
- j = Index for participating institute, (1 = NIMT, 2 = PTB, 3 = NMLPHIL)
- y = Index for measurement cycle, (1, 2, or 3)
- w = Index for indicating ascending or descending measurements, (1 or 2)
- i = Index for indicating pressure, $i \times 2$ MPa, i = 0 10

In this section, the reduction and analysis of the data are described in the following sequence:

- (i) Correction by the zero-pressure offset,
- (ii) Correction by the difference between nominal pressure and actual pressure,
- (iii) Calculation of the mean pressure of participating institute,
- (iv) Estimation of the measurement uncertainty of participating institute.

6.1 Correction by the zero-pressure offset

The first step of the analysis was to correct the reading of each pressure with zero-pressure offset. The correction was conducted for each calibration cycle. There were two 0 MPa measurement data in one cycle. The reading at the beginning of each cycle was used for the offset correction. By subtracting the offset from the raw reading R, the corrected reading R_{c0} was obtained as follows:

$$R_{c0}(j, y, w, i) = R(j, y, w, i) - R(j, y, l, 0).$$
⁽²⁾

6.2 Correction by the difference between nominal pressure and actual pressure

 R_{c0} is the reading of the TS corrected by the zero-pressure offset according to equation (2). Normally, the readings from the TS are linear, and the ratio of the readings to the actual pressures is generally independent of pressure for the pressure range if the deviation of the actual pressure from the nominal target pressure is small. As described in the technical protocol, the difference between the actual applied pressure and the nominal target pressure was adjusted to be within a thousandth of the nominal pressure. The ratios can be used to correct the readings for deviation of the applied pressure from the nominal pressure. When an exact nominal pressure P_n is applied as the common measurement result of the TS for all participants, the respective reading of the TS, R_{c1} , can be calculated by:

$$R_{c1}(j, y, w, i) = \frac{R_{c0}(j, y, w, i)}{P(j, y, w, i)} \cdot P_{n}(i),$$
(3)

where R_{c1} and P are the simultaneous reading of the TS and the actual applied pressure, respectively. Herewith, the ratio of the participant pressure to nominal pressure at each measurement point can be used to correct the reading for deviation of the pressure standard from the nominal pressure without significant effect.

6.3 Calculation of mean pressure of participating institute

The mean pressure of participating institute, P(j,i), is calculated by

$$P(j,i) = \frac{1}{6} \sum_{w=1}^{2} \sum_{y=1}^{3} R_{c1}(j, y, w, i).$$
(4)

P(j,i) is taken as an indicator of the TS pressure when the pressure standard of the participating institute generates the nominal target pressure. The results for P(j,i) from individual institutes are presented in Table 6.1. For NIMT that performed two measurement campaigns, at the beginning and the end of the comparison, the results from the 1st campaign were used and are given in Table 6.1.

		P (j,i) [MPa]					
	j	-	1	2	3		
i	P _n [MPa]	NIMT-1 ¹⁾	NIMT-2 ²⁾	PTB	NMLPHIL		
1	2	1.99994	2.00025	1.99990	1.99974		
2	4	4.00020	4.00048	3.99983	4.00012		
3	6	6.00010	6.00042	5.99970	5.99990		
4	8	8.00013	8.00050	7.99966	8.00002		
5	10	10.00015	10.00057	9.99961	10.00007		
6	12	12.00025	12.00069	11.99962	12.00025		
7	14	14.00021	14.00068	13.99952	14.00030		
8	16	16.00021	16.00071	15.99947	16.00029		
9	18	18.00018	18.00072	17.99938	18.00029		
10	20	20.00016	20.00071	19.99932	19.99978		

Table 6.1: TS mean pressures of the institutes for nominal target pressures, P(j,i)

1) NIMT results from the 1st measurement period. They were used as NIMT results in the comparison.

2) NIMT results from the 2^{nd} measurement period.

6.4 Estimation of uncertainties

In this subsection, all the uncertainties are expressed as the standard ones. The relative combined standard uncertainty in the mean pressure of j-th participating institute can be estimated from the root-sum-square of four component uncertainties as follows.

$$u_{c}\left\{P(j,i)\right\} = \sqrt{u_{std}^{2}\left\{P(j,i)\right\} + u_{rdm}^{2}\left\{P(j,i)\right\} + u_{temp}^{2}\left\{P(j,i)\right\} + u_{lts}^{2}\left\{P(j,i)\right\}},$$
(5)

where $u_{std}\{P\}$ is the relative standard uncertainty due to systematic effects in pressure standard of each participant, $u_{rdm}\{P\}$ is the relative standard uncertainty due to combined effect of short-term random errors of TS and pressure standard during measurement, $u_{temp}\{P\}$ is the relative standard uncertainty due to the effect of temperature and $u_{lts}\{P\}$ is the relative standard uncertainty arising from long-term shift in the characteristics of the TS calibrated at the pilot institute.

6.4.1 Uncertainty due to systematic effect in pressure standard

The relative standard uncertainty due to systematic effect in pressure standard, $u_{std}\{P(j,i)\}$, was estimated from

$$u_{\rm std}\left\{P\left(j,i\right)\right\} = \frac{u\left\{P_{\rm std}\left(j,i\right)\right\}}{P_{\rm n}\left(i\right)},\tag{6}$$

where $u\{P_{std}(j,i)\}$ is the standard uncertainty due to systematic effect in pressure standard for each participant and $P_n(i)$ is the nominal target pressure.

Table 6.2 presents the estimated relative standard uncertainties arising from systematic effects in the pressure standards used in the comparison, as reported by the participating institutes for nominal target pressures. The uncertainty due to the hydrostatic head correction was

considered as included in the uncertainty of the pressure standard. The main contributions in this uncertainty came from the effective area and the pressure distortion coefficient of the pressure standard of the participating institute.

Table 6.2: Relative standard uncertainties, as claimed by the participants, due to systematic effects in their pressure standards, $u_{std}{P(j,i)}$. All the uncertainties are expressed as the standard ones.

			$u_{std}{P(j,i)}$ [10 ⁻⁶]	
j		1	2	3
i	P _n [MPa]	NIMT	PTB	NMLPHIL
1	2	11.8	13.5	25.0
2	4	11.8	13.2	25.0
3	6	11.8	13.3	25.0
4	8	11.8	13.7	25.0
5	10	11.8	14.0	25.0
6	12	11.8	13.3	25.0
7	14	11.8	13.6	25.0
8	16	11.8	13.7	25.0
9	18	11.8	13.3	25.0
10	20	11.8	13.5	25.0

6.4.2 Uncertainty due to combined effect of short-term random errors

The relative standard uncertainty in P(j,i) due to combined effect of short-term random errors of the TS and pressure standard during measurement, $u_{rdm}\{P(j,i)\}$, was estimated on the basis of standard deviation by the equation below.

$$u_{\rm rdm}\left\{P(j,i)\right\} = \frac{\sqrt{\sigma^2 \{R_{c1}(j, y, w, i)\}/6}}{P_{\rm n}(i)},\tag{7}$$

where σ { $R_{c1}(j,y,w,i)$ } is the standard deviation of *n* values of $R_{c1}(j,y,w,i)$ from their mean with n = 6.

Table 6.3 and Figure 6.1 present the estimated standard uncertainties due to combined effect of short-term random errors calculated by equation (7).

Table 6.3: Relative standard uncertainties due to combined effect of short-term random errors, $u_{rdm}\{P(j,i)\}$. All the uncertainties are expressed as the standard ones.

		$u_{rdm}\{P(j,i)\}$ [10 ⁻⁶]			
j		1	2	3	
i	<i>P</i> _n [MPa]	NIMT	PTB	NMLPHIL	
1	2	15.7	60.1	16.2	
2	4	7.4	34.7	10.1	
3	6	6.8	22.8	9.9	
4	8	6.5	16.4	7.4	
5	10	5.8	12.5	6.4	
6	12	4.9	9.2	5.6	
7	14	3.6	6.9	3.5	
8	16	2.8	4.8	3.3	
9	18	2.0	3.4	2.4	
10	20	1.4	2.4	1.5	



Figure 6.1: Relative standard uncertainties due to short-term random errors as a function of nominal target pressure.

As it is discussed in section 6.4.4, the standard uncertainties presented here are caused not only by random effects but, to high extent, by hysteresis of the transfer standard.

6.4.3 Uncertainty due to the effect of temperature

The reading is normally affected by the temperature. However, the pilot institute did not analyse the temperature coefficient correction. Therefore, it does not have enough information to correct the results of TS for the effect of temperature change. Thus, the effect of temperature was estimated as the uncertainty for all participating institutes. Using the information from manufacturer, the relative standard uncertainty due to the effect of temperature can be determined by equation (8) and is shown in Table 6.4.

$$u_{\text{temp}}\left\{P(j,i)\right\} = \frac{7 \cdot 10^{-7} \text{ rdg } + 1 \cdot 10^{-6} \text{ span}}{P_n(i)},$$
(8)

where rdg is the reading pressure and span is the maximum pressure range of the TS (20 MPa). The uncertainty part $1 \cdot 10^{-6}$ span was considered under the limit of temperature deviation taken from all participants, within ± 2 °C, see Figure 6.2.



Room temperatures during measurements

Figure 6.2: Averaged room temperatures of the participating institutes for nominal target pressures.

i	P _n [MPa]	$u_{\text{temp}}\{P(j,i)\} [10^{-6}]$
1	2	10.7
2	4	5.7
3	6	4.0
4	8	3.2
5	10	2.7
6	12	2.4
7	14	2.1
8	16	2.0
9	18	1.8
10	20	1.7

Table 6.4: Relative standard uncertainties due to the effect of temperature, $u_{\text{temp}}\{P(j,i)\}$.

6.4.4 Uncertainty arising from the long-term shift

The TS was measured at pilot institute twice, before and after circulating the TS to the participating institutes. Therefore, it does not have enough information to correct the effect of long term instability to the results of the TS. In this comparison, the difference between two measurement periods at the pilot institute was estimated as the uncertainty of long-term shift for all participating institutes. If the NIMT reference standard was stable between the 1st and 2nd measurements, the temperature and the random errors $u_{rdm}{P(j,i)}$ were in both measurements the same, NIMT results in 1st and 2nd measurements will be consistent with such a long-term shift uncertainty contribution $u_{lts}{P}$ at which the following condition is fulfilled:

$$\frac{\left|P(1,i)_{2nd} - P(1,i)_{1st}\right| / P_n(i)}{\left[2 \cdot u_{rdm}^2 \{P(1,i)\} + u_{lts}^2 \{P(j,i)\}\right]^{0.5}} \le 1.$$
(9)

If $|P(1,i)_{2nd} - P(1,i)_{1st}| / P_n(i) \le 2^{0.5} \times u_{rdm} \{P(1,i)\}$, condition (9) is fulfilled even if $u_{lts} \{P(j,i)\} = 0$. This means that NIMT results in 1st and 2nd measurements are consistent within the random uncertainty of the TS, and no additional uncertainty contribution due to drift is required. Otherwise, the relative standard uncertainty due to instability of the TS can be estimated by equation (9a) and is shown in Table 6.5.

$$u_{\rm lts}\{P(j,i)\} = \left[\left(P(1,i)_{\rm 2nd} - P(1,i)_{\rm 1st} \right)^2 / P_{\rm n}^2(i) - 2 \cdot u_{\rm rdm}^2 \{P(1,i)\} \right]^{0.5},$$
(9a)

where $P(1,i)_{1st}$ and $P(1,i)_{2nd}$ are the mean pressures at the pilot institute for the first and the second measurement periods, respectively.

i	<i>P</i> _n [MPa]	$u_{\text{lts}}\{P(j,i)\} [10^{-6}]$
1	2	75.4
2	4	34.5
3	6	26.6
4	8	22.6
5	10	20.2
6	12	17.8
7	14	16.5
8	16	15.4
9	18	14.8
10	20	13.7

Table 6.5: Relative standard uncertainties due to instability of TS, $u_{\text{lts}}\{P(j,i)\}$.

Moreover, the instability of the TS can be estimated by analysing hysteresis behaviour of the TS in the measurements of the NMIs. Indeed, all participants have used pressure balances as reference standards, which therefore are assumed to have no hysteresis. The differences of the TS readings from the reference pressures are shown in Figure 6.3.



Figure 6.3: Differences between TS readings and nominal target pressures in the measurements of three NMIs.

These demonstrate that the TS hysteresis behaviour was rather different. The maximum difference of hysteresis amplitudes observed by the 3 participants is equal to 0.59 kPa. It was observed at 2 MPa and, in relative units, was equal to $2.93 \cdot 10^{-4}$.

6.4.5 Combined uncertainty of the mean pressure for participating institutes

The relative combined standard uncertainty of the mean pressure of participating institutes, $u_c\{P(j,i)\}$, calculated by equation (5) is shown in Table 6.6 and Figure 6.4.

Table 6.6: Relative combined standard uncertainties of the mean pressures of participating institutes, $u_c\{P(j,i)\}$. All the uncertainties are expressed as the standard ones.

		$u_{c}\{P(j,i)\} [10^{-6}]$					
j		1	2	3			
i	P _n [MPa]	NIMT	PTB	NMLPHIL			
1	2	78.7	98.0	81.8			
2	4	37.6	51.0	44.1			
3	6	30.2	37.7	38.1			
4	8	26.5	31.3	34.6			
5	10	24.2	27.7	32.9			
6	12	22.1	24.2	31.3			
7	14	20.7	22.6	30.2			
8	16	19.7	21.3	29.6			
9	18	19.1	20.3	29.2			
10	20	18.2	19.5	28.6			



Figure 6.4: Relative combined standard uncertainties of the mean pressures of participating institutes as a function of nominal target pressure.

7. Results for supplementary comparison APMP.M.P-S9

7.1 Comparison reference values

The standard uncertainties of participants are rather different. Therefore, the comparison reference values, P(ref,i), and their uncertainties, $u\{P(\text{ref},i)\}$, were determined from the results of all participating institutes by the weighted-mean method using equations (10). The values are shown in Table 7.1.

$$P(\text{ref},i) = \frac{\sum_{j} \frac{P(j,i)}{u_c^2 \{P(j,i)\}}}{\sum_{j} \frac{1}{u_c^2 \{P(j,i)\}}}, \text{ and } u\{P(\text{ref},i)\} = \left[\sum_{j} \frac{1}{u_c^2 \{P(j,i)\}}\right]^{-0.5}$$
(10)

i	P _n [MPa]	P (ref , <i>i</i>) [MPa]	<i>u</i> { <i>P</i> (ref , <i>i</i>)} [10 ⁻⁶]
1	2	1.99986	49.1
2	4	4.00009	25.0
3	6	5.99993	20.0
4	8	7.99995	17.5
5	10	9.99996	15.9
6	12	12.00002	14.5
7	14	13.99998	13.6
8	16	15.99995	13.0
9	18	17.99989	12.6
10	20	19.99977	12.1

Table 7	7.1:	Comparison	reference	values and	l their	standard	uncertainties
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7.2 Degree of equivalence

The degree of equivalence of the participating institute's result from the reference value is expressed by the two quantities: the deviation of the participating institute's result from the reference value, D(j,i), and the expanded uncertainty (k=2) of this deviation, U(j,i), as the following:

$$D(j,i) = P(j,i) - P(\operatorname{ref},i), \tag{11}$$

$$U(j,i) = 2 \cdot \sqrt{u_c^2 \{P(j,i)\} - u^2 \{P(\text{ref},i)\}} \cdot P_n.$$
(12)

The two quantities, D(j,i) and U(j,i), as well as their ratios are summarized in Table 7.2. All ratios D(j,i) / U(j,i) are within the unity, meaning that the results of the participating institute are equivalent with the comparison reference values. The deviation of the participating institute's result from the reference value D(j,i) is plotted with the error bar representing the expanded uncertainty U(j,i), in Figure 7.1.

Table 7.2: Degrees of equivalence of the participating institute's results in relation to the comparison reference value

j		1			2			3		
		NIMT			PTB			NMLPHIL		
i	P _n [MPa]	D(j,i) [MPa]	<i>U(j,i)</i> [MPa]	D(j,i) / U(j,i)	D(j,i) [MPa]	<i>U(j,i)</i> [MPa]	D(j,i) / U(j,i)	D(j,i) [MPa]	<i>U(j,i)</i> [MPa]	D(j,i) / U(j,i)
1	2	0.00008	0.00025	0.34	0.00004	0.00034	0.13	-0.00012	0.00026	-0.46
2	4	0.00011	0.00023	0.49	-0.00026	0.00036	-0.72	0.00004	0.00029	0.13
3	6	0.00017	0.00027	0.61	-0.00023	0.00038	-0.59	-0.00003	0.00039	-0.08
4	8	0.00017	0.00032	0.54	-0.00030	0.00042	-0.71	0.00007	0.00048	0.14
5	10	0.00020	0.00037	0.54	-0.00034	0.00045	-0.75	0.00012	0.00057	0.20
6	12	0.00023	0.00040	0.57	-0.00041	0.00047	-0.87	0.00022	0.00067	0.33
7	14	0.00024	0.00044	0.54	-0.00046	0.00050	-0.91	0.00032	0.00076	0.42
8	16	0.00026	0.00048	0.55	-0.00048	0.00054	-0.89	0.00034	0.00085	0.40
9	18	0.00029	0.00052	0.55	-0.00051	0.00057	-0.89	0.00039	0.00095	0.41
10	20	0.00039	0.00055	0.72	-0.00045	0.00061	-0.74	0.00001	0.00104	0.01



Figure 7.1: Deviation of the participating institute's results from the reference value

8. Conclusions

A supplementary comparison of gas pressure standards was conducted among three National Metrology Institutes; National Institute of Metrology (Thailand) (NIMT), Physikalisch-Technische Bundesanstalt (PTB) and National Metrology Laboratory of the Philippines (NMLPHIL), within the framework of Asia-Pacific Metrology Programme (APMP) in order to determine their degrees of equivalence at pressures in the range from 2 MPa to 20 MPa for gauge mode. The measurements were carried out from February 2016 to July 2016. The uncertainties of the participants' results were calculated from the uncertainty of their reference standards, repeatability of the measurements, which included the hysteresis effect, temperature dependence of the transfer standard and its instability during the comparison. The reference values were calculated by the weighted mean method. At all pressures, the results of the participants are in agreement with the reference values and with each other within the expanded uncertainties (k=2). However, the uncertainties ascribed to the participants' results are much bigger than the uncertainties of their standards and are mostly caused by instability of the transfer standard during the comparison. Therefore, equivalence of the compared standards can be stated only on the uncertainty level of the comparison results, which is dominated by the uncertainty of the transfer standard. As for the performance of the transfer standard, it is necessary to note that the precision of the TS (including linearity, hysteresis and repeatability) and the measurement uncertainty of the TS were at least two to four times better than predicted by the manufacturer.

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