

Report on a bilateral comparison
in the gauge pressure range of 0,7 MPa to 7 MPa. between LNE and
KIM-LIPI
Euramet project 1330

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ABSTRACT

LNE and KIM-LIPI compared their pressure standards in the gauge pressure range of 0,7 MPa to 7 MPa. The results of the comparison can be considered as satisfactory as all the deviations from the reference values are inside the estimated combined uncertainties with a coverage factor $k = 2$. LNE was the pilot laboratory of the comparison.

1. Introduction

This comparison is a part of the EURAMET project n° 1330. The present report describes the results obtained by the participants. The transfer standard was a pressure monitor type RPM4 A7M from DHInstruments, Inc. serial No. 684, operating with nitrogen with a resolution of 1 Pa.

The nominal pressure points for the comparison were 0 hPa; 0,7 MPa; 1,4 MPa; 2,1 MPa; 2,8 MPa; 3,5 MPa; 4,2 MPa; 4,9 MPa; 5,6 MPa; 6,3 MPa and 7MPa.

The results of the present comparison can be compared to those of the key comparison *CCM.P-K1.c*

2. Participants

The participating institutes are presented in table 1. The comparison took place from August 2014 to October 2014.

Table 1. Comparison participants

Institute	Measurement date	Person responsible for the intercomparison
KIM-LIPI	26/08/2014	Rudi Anggoro
Laboratoire National d' Essais	03/09/2014 30/09/2014	Pierre Ota
KIM-LIPI	16/10/2014	Rudi Anggoro

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3. Laboratory standards

KIM-LIPI and LNE pressure standards are PG 7601 type pressure balance manufactured by DH Instruments. Both standards were equipped with DH Instruments 0,5 cm² effective area piston-cylinder assemblies made of tungsten carbide.

It was recommended that each laboratory should use a data sheet reporting the data obtained at each comparison point. The measurements were performed for three cycles. The laboratories were also required to report the standard uncertainty of the deviation.

3.1 KIM-LiPi pressure standard

The details of the KIM-LIPI pressure standard are listed in tables 2.

Table 2. KIM-LIPI pressure standard and measurement conditions

Manufacturer & Model	DH Instruments & PG 7601-AMH
Measurement range in MPa	(0.35 - 7) MPa
Material of piston	Tungsten Carbide
Material of cylinder	Tungsten Carbide
Reference temperature (t_0) in °C	20
Zero-pressure effective area (A_0) at reference temperature in mm ²	49.01695
Relative standard uncertainty of A_0 in 10 ⁻⁶	18
Pressure distortion coefficient (λ) in MPa ⁻¹	-2.35 x 10 ⁻⁶
Uncertainty of λ in MPa ⁻¹	1.0 x 10 ⁻⁶
Linear thermal expansion coefficient of piston (α_p) in °C ⁻¹	4.55 x 10 ⁻⁶
Linear thermal expansion coefficient of cylinder (α_c) in °C ⁻¹	4.55 x 10 ⁻⁶
Local acceleration due to gravity (g) in m/s ²	9.781379
Relative uncertainty of g in 10 ⁻⁶	5
Height difference between laboratory standard (LS) and TS (h , positive if LS is higher than TS) in mm	0.0
Uncertainty of h in mm	5
Operating gas (N ₂)	Yes
Piston rotation speed during measurement in rpm	30~50 rpm
Room Temperature during measurement in °C	(18 to 22) °C with stability 1°C
Traceability	PTB 2007 and 2013

The Pressure Balance Standard was bought in 2007 and is traceable to PTB-Germany (2007 and 2013).

The expanded uncertainty of the pressure measured by the balance in the conditions of calibration at KIM-LIPI is :

$$U(p)_{\text{KIM-LIPI}} = 2,4 \text{ Pa} + 1,7 \cdot 10^{-5} \cdot p + 2,1 \cdot 10^{-13} \cdot p^2$$

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3.2 LNE Pressure standard

The details of the LNE pressure standard are listed in tables 3.

Table 3. LNE pressure standard and measurement conditions

Manufacturer & Model	DH Instruments & PG 7601-AMH
Measurement range in MPa	(0.35 - 7) MPa
Material of piston (N°1051)	Tungsten Carbide
Material of cylinder (N°1051)	Tungsten Carbide
Reference temperature (t_0) in °C	20
Zero-pressure effective area (A_0) at reference temperature in mm ²	49.018 72
Relative standard uncertainty of A_0 in 10 ⁻⁶	5.5
Pressure distortion coefficient (λ) in MPa ⁻¹	-2.35 x 10 ⁻⁶
Standard uncertainty of λ in MPa ⁻¹	1.2 x 10 ⁻⁷
Linear thermal expansion coefficient of piston (α_p) in °C ⁻¹	4.55 x 10 ⁻⁶
Linear thermal expansion coefficient of cylinder (α_c) in °C ⁻¹	4.55 x 10 ⁻⁶
Local acceleration due to gravity (g) in m/s ²	9.809273
Relative standard uncertainty of g in 10 ⁻⁶	0,1
Height difference between laboratory standard (LS) and TS (h , positive if LS is higher than TS) in mm	0.0
Standard uncertainty of h in mm	1
Operating gas (N ₂)	Yes
Piston rotation speed during measurement in rpm	20~40 rpm
Room Temperature during measurement in °C	20 °C with stability 0,5°C
Traceability	Traceable to primary piston-cylinder assembly

The effective area (A_p) of the piston-cylinder assembly n°1051 is traceable to the primary piston-cylinder assembly DH 6594 whose zero-pressure effective area (A_0) is based on dimensional measurements.

The measurements were carried out in an air-conditioned room with the temperature maintained between 19.5 and 20.1 °C during all measurements.

The measurements were performed with the automatic system AMH.

The expanded uncertainty of the pressure measured by the balance in the conditions of calibration at LNE is :

$$U(p)_{LNE} = 1,0 \text{ Pa} + 1,0 \cdot 10^{-5} \cdot p$$

The standard uncertainty of the deviation was calculated combining the uncertainty of the reference pressure, the repeatability between the deviations observed in cycles 1 to 3, and the resolution of the transfer standard.

4. Transfer Standard

4.1 Identification

The transfer standard is a pressure monitor RPM4 A7M, serial No. 684, operating with nitrogen. It has a resolution of 1 Pa. The manufacturer is DHInstruments, Inc. The transfer standard was carried by hand by

4.2 Operating principle

The manometer is composed of an absolute Quartz Reference Pressure Transducer (Q-RPT) and of an internal barometer.

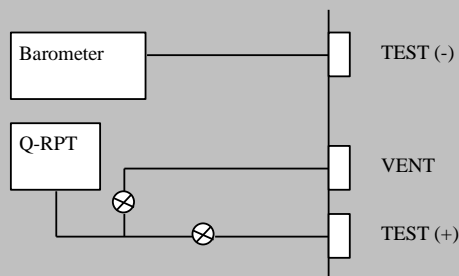


Figure 1: RPM4 pneumatic schematics

The gauge pressure indicated by the RPM4 at the instant t , $P_{RPM}(t)$, is expressed by the following equation:

$$P_{RPM}(t) = P_Q(t) - P_Q(0) + P_{baro}(t) - P_{baro}(0) \quad (1)$$

with the following expressions:

- $P_Q(t)$: Indication of the Q-RPT absolute pressure at the instant t
- $P_Q(0)$: Indication of the Q-RPT at the atmospheric pressure at the time of zeroing execution
- $P_{baro}(0)$: Indication of the barometer at the atmospheric pressure, at the time of zeroing execution
- $P_{baro}(t)$: Indication of the barometer at the atmospheric pressure, at the instant t .

5. Calibration procedure

The calibration of the transfer standard had to be performed after a warm-up time of at least twelve hours in an air conditioned room at 20 °C. It was asked to perform eleven pressure points in an ascending and then descending sequence, repeated three times, at the following nominal gauge pressures: 0 hPa; 0,7 MPa; 1,4 MPa; 2,1 MPa; 2,8 MPa; 3,5 MPa; 4,2 MPa; 4,9 MPa; 5,6 MPa; 6,3 MPa; 7MPa of nitrogen. The stabilisation time at each pressure point was one minute and the recording time of the transfer standard readings at each pressure level thirty seconds.

The one cycle procedure is described below:

- Zeroing of the pressure module by running the "AutoZero" function of the transfer standard after connecting together the TEST(+) port with the TEST(-) port
- Feeding the transfer standard from the reference standard at the successive pressure levels up to 7 MPa, avoiding to come back to zero pressure between the points
- Applying a stabilisation time of five minutes at 7 MPa, then feeding the transfer standard from the reference standard at the successive pressure levels down to zero pressure
- Applying a stabilisation time of five minutes at zero prior to a new zeroing of the pressure module and the beginning of another cycle.

6. Transfer standard stability

The transfer standard was calibrated 2 times at LNE: 03 Sept. 2014 and 30 Sept. 2014. Figure 2 shows the stability of the transfer standard as observed at LNE during this period. A small drift can be

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identified. The standard uncertainty due to the stability of the transfer standard is estimated from the relative difference of the calibration slopes between the two calibrations at LNE :

$$u_{stab} = \frac{|Slope_{LNE\ end} - Slope_{LNE\ begin}|}{\sqrt{3}} \times \frac{n_{Kim-Lipi}}{n_{LNEi}} = 5,7 \cdot 10^{-6} \cdot p \quad (2)$$

Where n_{Lab} represents the number of days separating two calibrations in a laboratory. From their side, KIM-LiPI observed a drift of 4 ppm between the two calibrations.

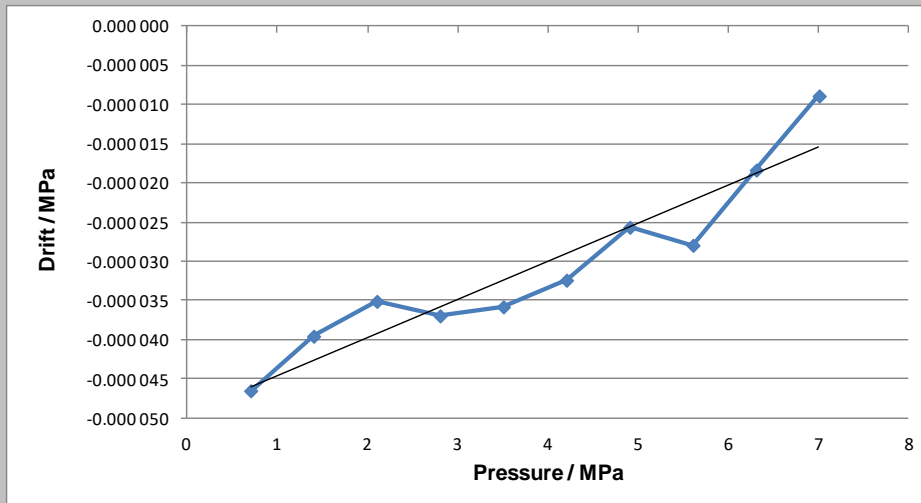


Figure 2. Stability of the transfer standard as observed at LNE. Difference between the two calibrations.

7. Results

The mean deviations ($D_{p,i}$) measured by the participants corrected from zero and their standard uncertainties u (D_{lab}) are presented in Table 7 and figure 3. For both laboratories, the average from the two calibrations is considered.

The standard uncertainties u (D_{lab}) are calculated using the equation:

$$u(D_{Lab}) = \sqrt{\frac{u_{LAB1}^2}{4} + \frac{u_{LAB2}^2}{4} + \frac{1}{2} \rho * (u_{LAB1} \times u_{LAB2})} \quad (3)$$

A correlation coefficient $\rho = 0,8$ is considered between the two calibrations.

Table 7. Mean deviations ($D_{p,i}$) measured by the participants and their standard uncertainties.

Nominal pressure MPa	LNE		KIM LIPI	
	Mean deviations ($D_{p,i}$) MPa	Standard uncertainties MPa	Mean deviations ($D_{p,i}$) MPa	Standard uncertainties MPa
0.0	0.000 000	0.000 009	0.000 000	0.000 002
0.7	0.000 050	0.000 017	0.000 080	0.000 034
1.4	0.000 114	0.000 013	0.000 140	0.000 035
2.1	0.000 159	0.000 016	0.000 191	0.000 055
2.8	0.000 183	0.000 020	0.000 210	0.000 063
3.5	0.000 204	0.000 022	0.000 219	0.000 077
4.2	0.000 215	0.000 025	0.000 226	0.000 090
4.9	0.000 245	0.000 028	0.000 250	0.000 103
5.6	0.000 288	0.000 031	0.000 289	0.000 118
6.3	0.000 350	0.000 035	0.000 350	0.000 132
7.0	0.000 455	0.000 035	0.000 442	0.000 148
7.0	0.000 452	0.000 035	0.000 457	0.000 148
6.3	0.000 428	0.000 031	0.000 432	0.000 133
5.6	0.000 415	0.000 031	0.000 421	0.000 120
4.9	0.000 410	0.000 026	0.000 420	0.000 104
4.2	0.000 402	0.000 023	0.000 420	0.000 090
3.5	0.000 402	0.000 019	0.000 418	0.000 077
2.8	0.000 372	0.000 015	0.000 397	0.000 062
2.1	0.000 331	0.000 011	0.000 362	0.000 049
1.4	0.000 250	0.000 008	0.000 289	0.000 038
0.7	0.000 120	0.000 008	0.000 176	0.000 026
0.0	-0.000 036	0.000 023	0.000 006	0.000 016

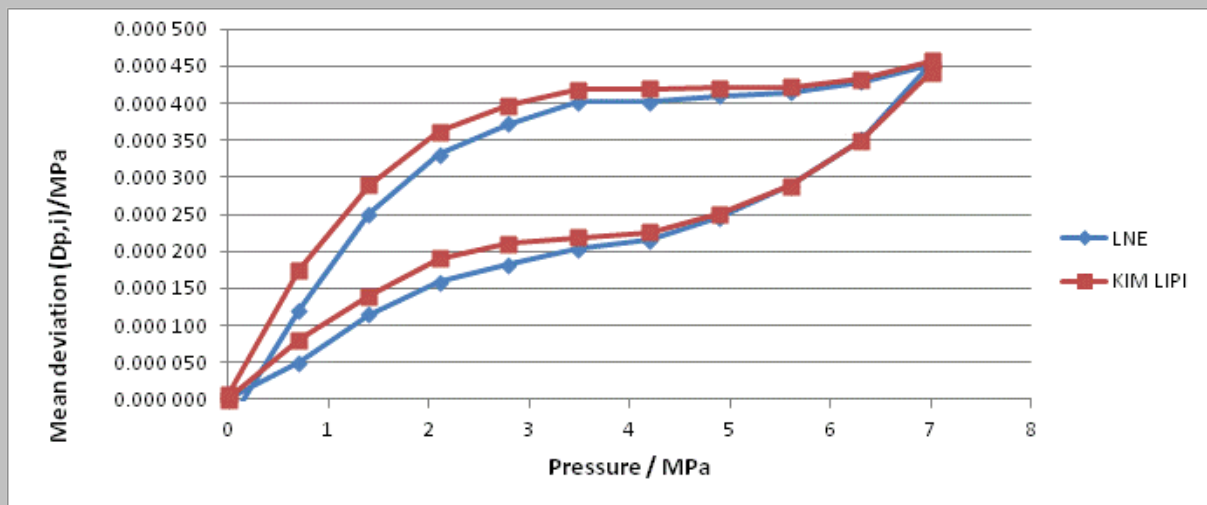


Figure 3. Mean deviations ($D_{p,i}$) measured by the participants.

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8. Reference values

The deviations from LNE (D_{LNE}) are considered as the reference values. Their standard uncertainties are calculated using the equation:

$$u(D_{pref}) = \sqrt{\frac{u_{LNE1}^2}{4} + \frac{u_{LNE2}^2}{4} + \frac{1}{2} \rho^* (u_{LNE1} \times u_{LNE2}) + u_{stab}^2} \quad (4)$$

A correlation coefficient $\rho = 0,8$ is considered between the two calibrations at LNE.

9. Deviation from the reference values, degrees of equivalence

The deviation of KIM-LIPI from the reference values $\delta_p = (D_{KIM-LIPI} - D_{REF})$ and their expanded uncertainties $U(\delta_p)$ are given table 9. $U(\delta_p)$ is calculated as the combination of the uncertainty of the reference value and the uncertainty of KIM-LIPI deviation:

$$U(\delta_p) = 2\sqrt{[u^2(D_{pref}) + u^2(D_{KIM-LIPI})]} \quad (5)$$

The degrees of equivalence E_n are quantified by :

$$E_n = \delta_p / U(\delta_p) \quad (6)$$

Table 9. Differences of the deviations of KIM-LIPI to the reference values δ_p for each pressure, their expanded uncertainties $U(\delta_p)$ and the degrees of equivalence E_n

Nominal pressure MPa	KIM LIPI Deviation from the reference values MPa	Expanded uncertainty of KIM- LIPI deviation from the reference values / MPa	Degree of equivalence
	δ_p MPa	$U(\delta_p)$ MPa	E_n
0.0	0.000 000	0.000 007	
0.7	0.000 030	0.000 096	0.31
1.4	0.000 025	0.000 101	0.25
2.1	0.000 032	0.000 158	0.20
2.8	0.000 028	0.000 182	0.15
3.5	0.000 015	0.000 221	0.07
4.2	0.000 011	0.000 258	0.04
4.9	0.000 005	0.000 297	0.02
5.6	0.000 001	0.000 339	0.00
6.3	0.000 000	0.000 380	0.00
7.0	-0.000 014	0.000 427	-0.03
7.0	0.000 005	0.000 426	0.01
6.3	0.000 004	0.000 384	0.01
5.6	0.000 007	0.000 344	0.02
4.9	0.000 010	0.000 300	0.03
4.2	0.000 018	0.000 259	0.07
3.5	0.000 016	0.000 221	0.07
2.8	0.000 025	0.000 178	0.14
2.1	0.000 031	0.000 141	0.22
1.4	0.000 039	0.000 108	0.36
0.7	0.000 055	0.000 074	0.75
0.0	0.000 041	0.000 045	

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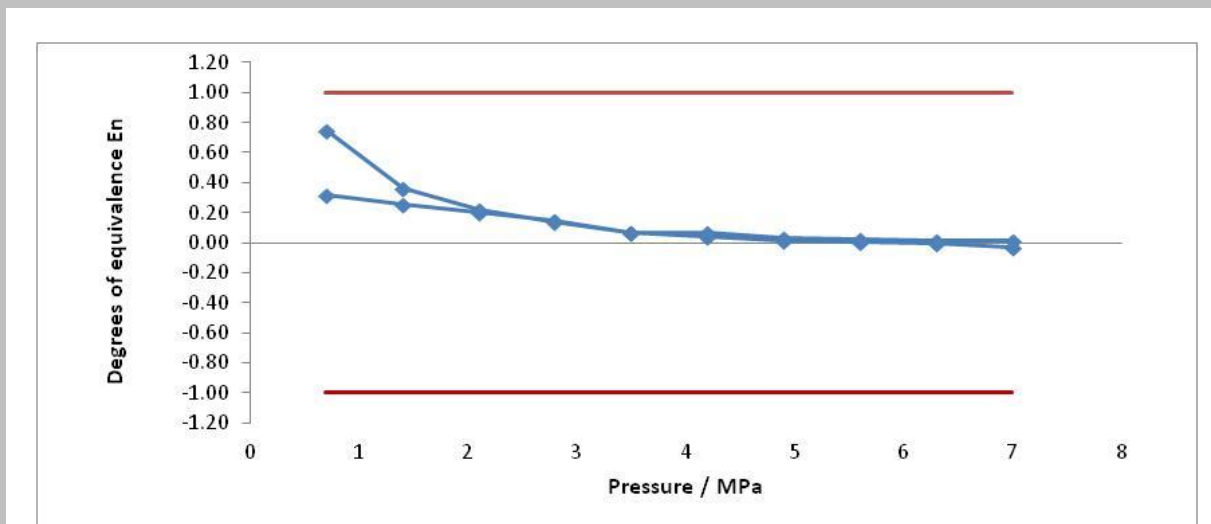


Figure 4. Degrees of equivalence E_n of KIM-LIPI

10. Conclusion

The results of the comparison can be considered as satisfactory. All the values reported by KIM-LIPI agree with the reference values within the expanded uncertainties with a coverage factor $k = 2$. The degrees of equivalence between both laboratories are always less than 1, and in more than half of the cases less than 0,1.

The standard deviation of the deviations of KIM-LIPI from the reference values is less than 17 Pa, representing $2,4 \times 10^{-6}$ of the pressure range.