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CCM key comparison in the pressure range 50 kPa to 1000 kPa (gas medium, gauge mode)

Phase A2: Pressure measurements

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Abstract. As part of a wide-ranging key comparison organized by the Consultative Committee for Mass and Related Quantities (CCM) of the Comité International des Poids et Mesures, this report gives the results of a comparison of pressure measurements in the range 0.05 MPa to 1 MPa. The two transfer standards used were pressure balances equipped with large (10 cm^2) effective area piston-cylinder assemblies. The scope of the comparison covered the observation of the behaviour of both piston and cylinder assemblies from two manufacturers and made from different materials. The results show agreement of all the laboratory standards within the estimated expanded uncertainties, expressed with a coverage factor $k = 2$. Most of the difference values (47 out of 54) are inside the standard uncertainties. These results demonstrate the coherency of the standards of the participating laboratories in the range 100 kPa to 1000 kPa for gas pressure, gauge mode.

1. Introduction

In 1996 the CCM High Pressure Working Group decided to organize a comparison in the range 50 kPa to 1 MPa. This work is part of the key comparison in gas media for pressures from 0.05 MPa to 7 MPa, reference CCM.P-K1. The comparison was divided into three phases, A1, A2 and B.

In phase A1, the participants had to determine from dimensional measurements the effective area of two piston-cylinder assemblies with a nominal effective area of 10 cm^2.

In phase A2, the same piston-cylinder assemblies used in phase A1 were circulated with their own balance base. The effective area of each assembly was determined from pressure measurements by comparison with the same laboratory standard.

In phase B, two different piston-cylinder units covering a pressure range up to 7 MPa were circulated between the four laboratories reported here, plus the National Research Laboratory of Metrology (Japan). In this case the comparison results will be analysed on the basis of the effective area determined by participants from a pressure comparison (cross-floating method) against their standards.

The results of phase A1 (CCM.P-K1.a) are given in [1]. The results of phase A2 (CCM.P-K1.b) with a comparison of the results from the two phases are given in [2] and are summarized in this paper.

The objective of the work was the comparison of pressure standards at the lowest uncertainty. The two transfer standards were circulated independently between October 1995 and October 1997.

2. Details of transfer standards

2.1 Description

The two piston-cylinder assemblies transfer standards placed at the working group's disposal by the manufacturers were:

(a) piston-cylinder assembly serial no. DH 6594, manufactured by Desgranges et Huot (DH, France), of the free-deformation type. Both elements are made from tungsten carbide. The assembly was circulated with a type 5111 pressure balance, No. 6593. The PTB determined all the characteristics of the piston-cylinder assembly, except the effective area at null pressure and

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reference temperature A_0. The theoretical pressure distortion coefficient was supplied to each of the laboratories. The PTB also made measurements at the beginning and end of the comparison period (August 1995 and October 1997, respectively), in order to detect the stability of the transfer standard.

(b) piston-cylinder assembly serial no. DHI 107, manufactured by DH Instruments (DHI, USA), of the free-deformation type. The cylinder is made from tungsten carbide and the piston from ceramic. The assembly was circulated with a type PG7101 pressure balance, No. 126. The BNM-LNE performed the same measurements for this standard as the PTB did for the DH standard. The comparison was carried out in September 1995 and March 1997.

In both cases, the piston was loaded using the laboratories' own weights. The maximum mass was 100 kg.

2.2 Stability

Figure 1 shows the stability of the two transfer standards. In order that the effective area of each assembly should remain anonymous, we give the relative differences observed between the two calibrations for each pressure.

The figure gives the differences between the results of the effective area determination before and after circulation. The associated relative standard uncertainties, expressed with a coverage factor $k = 2$, were typically 5×10^{-6} to 7×10^{-6}. No significant shift comparable with the uncertainties was observed.

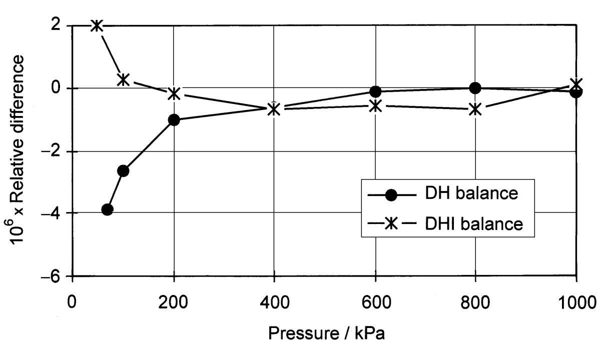

Figure 1. Stability of the piston-cylinder assemblies, as observed at the PTB (DH balance) and the BNM-LNE (DHI balance).

2.3 Direct comparison

As the two standards were at the BNM-LNE at the same time, they were also directly compared in order to obtain more information regarding the ratio of the two effective areas.

The results of the direct comparison are presented in Section 4.3 with the results of all the laboratories, calculated from the ratio of the individual comparisons.

3. Participants' standards

The standards of all the laboratories were pressure balances. All of them were equipped with a simple type of piston-cylinder assembly. Table 1 gives the main specifications of these standards. More detailed descriptions and some important work concerning the standards are given in [3-7].

Table 1. Specifications of the reference standards of participating laboratories.

Laboratory	Effective area A_0/mm^2	Uncertainty of $A_0 \times 10^6$	λ coefficient $\times 10^6$
BNM-LNE	980.524 2	2.2	4 ± 0.2
CNR-IMGC	499.886 57	6	4.2 ± 0.84
NIST	83.982 14	7.9	0 ± 5
PTB	980.491 7	3.2	4 ± 0.8

The effective area of each laboratory standard was measured:

(a) from dimensional measurements at the BNM-LNE and the CNR-IMGC;

(b) by comparison with a mercury column manometer at the NIST;

(c) by a combination of the two methods at the PTB.

4. Analysis of results

4.1 Linearity of transfer standards

Analysis of the results showed that the A_{0i} values calculated by each laboratory are not significantly dependent on pressure. A reference value $A_{0\text{ref1}}$ was calculated as the average of all the values obtained in all the laboratories:

$$A_{0\text{ref1}} = \sum_{i=1}^{4} \sum_{p} A_{0i,p} \Big/ 24.$$

In order to give each laboratory the same weight, only the values obtained for pressures from 100 kPa to 1000 kPa were used for the calculation of the average value. For the PTB and the BNM-LNE, the mean of the two values obtained before and after the circulation of the transfer standard was used when appropriate.

Figures 2 and 3 give the deviations of the laboratory values from $A_{0\text{ref}}$ for the DH and the DHI standard, respectively.

4.2 Results relating to effective area

The results obtained by each laboratory are compared in Table 2 for the DH standard and Table 3 for the DHI standard. The standard deviations observed for each transfer standard are similar in all the laboratories. As indicated in the tables, they are generally less than 1×10^{-6} for pressures equal and above 100 kPa.

Table 2. Synthesis of the results observed for each laboratory using the DH transfer standard. Diff: difference from average; s: standard deviation; u_c: combined standard uncertainty.

	PTB			CNR-IMGC			NIST			BNM-LNE		
Pressure/kPa	Diff. $\times 10^6$	$s \times 10^6$	$u_c \times 10^6$	Diff. $\times 10^6$	$s \times 10^6$	$u_c \times 10^6$	Diff. $\times 10^6$	$s \times 10^6$	$u_c \times 10^6$	Diff. $\times 10^6$	$s \times 10^6$	$u_c \times 10^6$
50 (70)	**4.70**	0.31	*5.1*				**6.13**	1.2	*8.4*	**5.16**	1.06	*3.9*
100	**4.70**	0.40	*5.0*	**–1.75**	0.81	*7.6*	**6.33**	1.5	*8.7*	**–0.92**	0.56	*3.3*
200	**3.78**	0.20	*4.1*	**–3.96**	0.35	*7.0*	**–0.30**	0.8	*9.1*	**–1.45**	0.77	*3.1*
400	**3.99**	0.16	*3.8*	**–3.10**	0.28	*6.8*	**–2.74**	0.6	*10.1*	**–1.20**	0.38	*3.0*
600	**4.24**	0.15	*3.7*	**–3.03**	0.30	*6.7*	**–3.56**	0.8	*11.1*	**–0.24**	0.33	*2.9*
800	**4.19**	0.13	*3.7*	**–1.93**	0.27	*6.6*	**–3.66**	0.9	*12.1*	**0.10**	0.28	*2.9*
1000	**4.24**	0.09	*3.7*	**–1.49**	0.28	*6.6*	**–2.85**	0.9	*13.1*	**0.58**	0.27	*2.9*

Table 3. Synthesis of the results observed for each laboratory using the DHI transfer standard. Diff: difference from average; s: standard deviation; u_c: combined standard uncertainty.

	PTB			CNR-IMGC			NIST			BNM-LNE		
Pressure/kPa	Diff. $\times 10^6$	$s \times 10^6$	$u_c \times 10^6$	Diff. $\times 10^6$	$s \times 10^6$	$u_c \times 10^6$	Diff. $\times 10^6$	$s \times 10^6$	$u_c \times 10^6$	Diff. $\times 10^6$	$s \times 10^6$	$u_c \times 10^6$
50 (70)	**0.46**	1.06	*5.2*				**–4.83**	2.6	*8.8*	**–1.21**	0.82	*3.9*
100	**0.60**	1.06	*5.0*	**4.69**	1.3	*7.6*	**4.94**	0.9	*8.7*	**–4.02**	0.67	*3.4*
200	**1.43**	0.87	*4.1*	**1.87**	0.6	*7.0*	**2.12**	1.0	*9.2*	**–3.50**	0.24	*3.1*
400	**1.23**	0.57	*3.8*	**0.22**	0.5	*6.8*	**2.19**	0.7	*10.1*	**–2.55**	0.17	*3.0*
600	**0.69**	0.49	*3.7*	**–0.60**	0.3	*6.7*	**–0.54**	0.4	*11.1*	**–1.85**	0.18	*2.9*
800	**0.93**	0.29	*3.7*	**–1.13**	0.2	*6.6*	**–1.04**	0.2	*12.1*	**–2.13**	0.15	*2.9*
1000	**1.36**	0.32	*3.7*	**–1.02**	0.1	*6.6*	**–1.23**	0.2	*13.1*	**–2.67**	0.55	*2.9*

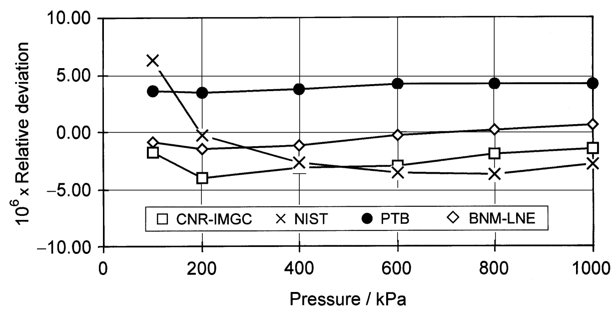

Figure 2. Relative deviation of the effective area from the average value for the DH balance.

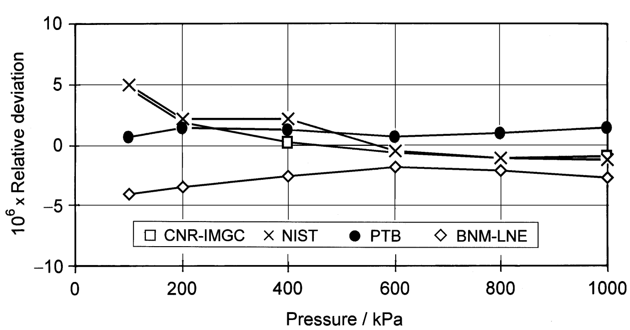

Figure 3. Relative deviation of the effective area from the average value for the DHI balance.

The estimated uncertainties are very different from one laboratory to another: ratios from 1 to 4 can be observed in some cases. The main contributions to the uncertainty arise from the effective area and the pressure distortion coefficient λ of the standards laboratory, as given in Table 1.

The results did not demonstrate any significant difference in the behaviour of the two transfer standards, whether equipped with a piston made from tungsten carbide or ceramic. The results are also represented graphically, for the minimum and maximum pressure alternatively (Figures 4 and 5, for both transfer standards respectively).

Figures 4 and 5 were drawn up in order to demonstrate the equivalence of the results obtained in all the laboratories. The expanded uncertainty of the difference U_{diff} was calculated for each laboratory as the combination of the standard uncertainty of the laboratory and the standard uncertainty of the reference u_{ref}.

$$U_{\mathrm{diff}} = 2 \times \sqrt{u_i^2(\mathrm{lab}) + u_{\mathrm{ref}}^2}.$$

The standard uncertainty of each of the reference values was calculated from the uncertainty estimated by each laboratory as

$$U_{\mathrm{ref}} = \frac{1}{4}\sqrt{\sum_{i=1}^{4} u_i^2(\mathrm{lab})}.$$

As no significant shift was observed in the transfer standards, no uncertainty component arising from the instruments has been taken into account in the reference value.

The standard uncertainties of both reference values were calculated as 6.5×10^{-6} at 100 kPa and 7.7×10^{-6} at 1000 kPa.

Figures 4 and 5 show the equivalence of the results as in all cases the observed differences are less than the estimated expanded uncertainties.

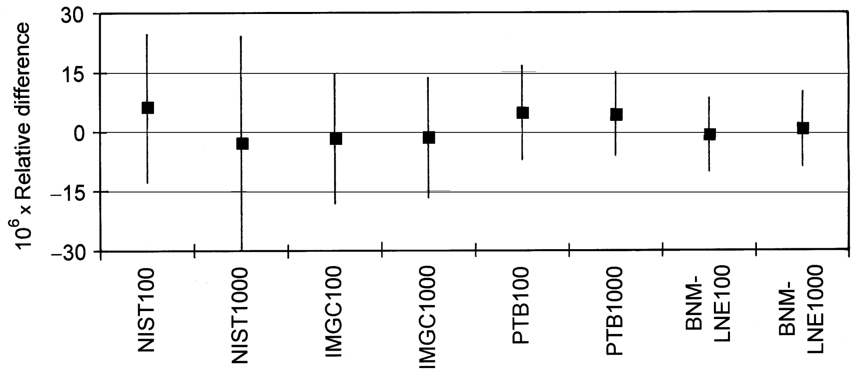

Figure 4. Difference of each laboratory from the reference value, obtained with the DH transfer standard.

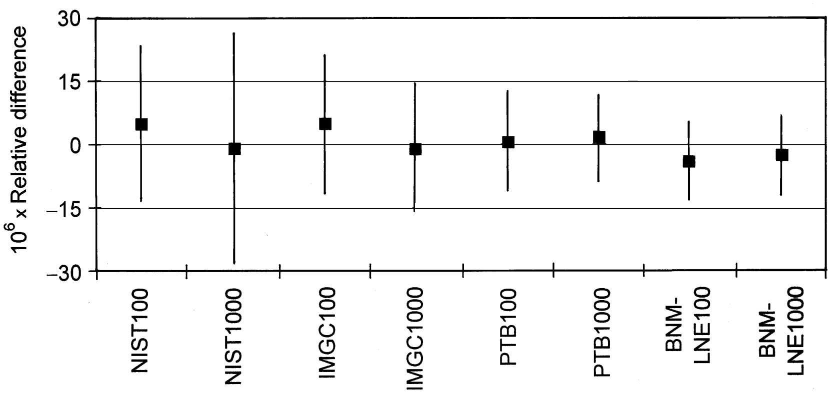

Figure 5. Difference of each laboratory from the reference value, obtained with the DHI transfer standard.

Table 4. Equivalence of pairs of laboratories, at 100 kPa: $10^6 \times$ difference (upper value) and $10^6 \times$ combined expanded uncertainty (lower value).

			NIST	PTB	CNR-IMGC	DHI
PTB	6.5 18		-9.0 19	-4.6 12	-8.7 17	BNM-LNE
NIST	8.1 23	1.6 20		4.3 20	0.2 23	NIST
BNM-LNE	0.8 17	-5.6 12	-7.3 19		-4.1 18	PTB
DH	CNR-IMGC	PTB	NIST			

Table 5. Equivalence of pairs of laboratories, at 1 MPa: $10^6 \times$ difference (upper value) and $10^6 \times$ combined expanded uncertainty (lower value).

			NIST	PTB	CNR-IMGC	DHI
PTB	5.7 15		-1.4 27	-2.7 9.4	-1.7 14	BNM-LNE
NIST	-1.4 29	-7.1 27		-1.2 27	-0.2 29	NIST
BNM-LNE	2.1 14	-3.7 9	3.4 27		1.0 15	PTB
DH	CNR-IMGC	PTB	NIST			

Tables 4 and 5 show the equivalence of pairs of laboratories for the pressures 100 kPa and 1 MPa, respectively. For each pair of laboratories, the tables give the observed differences (upper value) and the uncertainty of the difference calculated as the quadratic combination of the two laboratory uncertainties (lower value). The left part of the tables relates to the DH balance and the right part to the DHI balance.

4.3 Results relating to ratio of effective area

The ratio of the effective areas of both transfer standards was calculated for each pressure from the values determined in each laboratory by individual calibration of each standard. In Figure 6 these results are compared with the experimental values directly measured at the BNM-LNE (see Section 2.3).

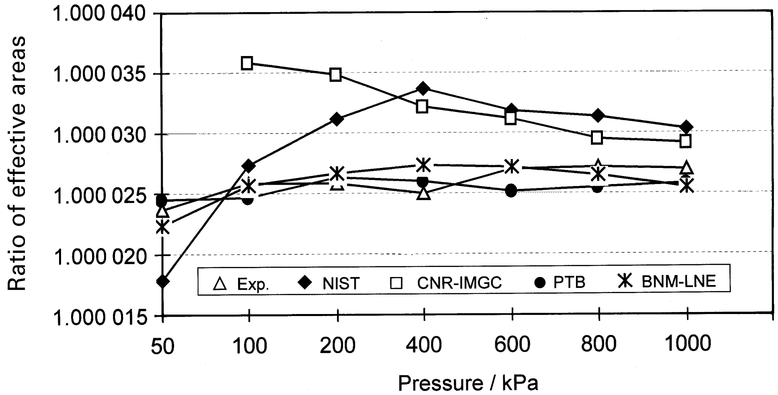

Figure 6. Difference in the ratio of the effective areas determined by each laboratory.

The figure shows an agreement in the ratios within 2×10^{-6} between the results of the PTB and the BNM-LNE, and the experimental determination by direct cross-floating. This can be considered as an estimate of the reproducibility of the transfer standards.

5. Comparison of dimensional and pressure measurements

The effective area of each piston and cylinder under reference conditions was calculated from the dimensional measurements [1]. Table 6 compares these results with the reference values. The differences between the reference values calculated from the dimensional measurements and from the pressure measurements are 2.4 and 2.2, respectively, for both transfer standards.

Table 6. Differences from the reference values observed for each laboratory.

Laboratory	Diff1 $\times 10^6$	U_{diff1} $\times 10^6$	Diff2 $\times 10^6$	U_{diff2} $\times 10^6$
BNM-LNE	–6.5 (–7.4)	8.1 (8.0)	–0.5 (–2.8)	5.8 (5.8)
CNR-IMGC	+1.4 (–3.5)	22 (23)	–2.5 (+0.8)	13 (13)
NIST	–2.0 (+2.0)	11 (11)	–1.1 (+1.0)	26 (26)
PTB	–1.5 (+0.1)	10 (10)	+4.2 (+1.0)	7.4 (7.4)

In the table, diff1 represents the differences for the dimensional measurements and diff2 the differences for the pressure measurements. The expanded uncertainties of the differences were calculated using the method described above for pressure measurements. The two values given in each row correspond to both transfer standards.

The results show an equivalence between all the results, for dimensional measurements as well as for pressure measurements: in all cases the difference is less than the expanded uncertainty.

Also, all the differences are less than 5×10^{-6} except for the dimensional measurements at the BNM-LNE. Thorough investigation demonstrated that a systematic error in the measurement process could explain the discrepancy. These new results will be confirmed through a further comparison.

6. Conclusions

The comparison results may be considered as fully satisfactory as only a very few laboratory differences from the average value are greater than 5×10^{-6}. These larger differences are observed at low pressure where the uncertainty of the force applied to the piston is higher. All the differences are less than the expanded uncertainty, expressed with a coverage factor $k = 2$. Only seven (out of 54) of the difference values are greater than the estimated standard uncertainties.

The two transfer standards used were pressure balances equipped with large (10 cm^2) effective area piston-cylinder assemblies. The piston and cylinder of the DH standard are both made from tungsten carbide, whereas the materials of the DHI standard are ceramic and tungsten carbide for the piston and cylinder, respectively. No significant difference was observed in the behaviour of either standard: in particular, the standard deviations and the deviations from linearity are similar.

These standards have demonstrated a repeatability expressed as a relative standard uncertainty of less than 1×10^{-6} over a large pressure range. As is clear from Figure 6, where the ratios of the effective areas of the two piston-cylinder assemblies calculated at the PTB and the BNM-LNE are in good agreement with those obtained from experiment, this work demonstrates that it is possible to achieve, under certain conditions, comparisons of national standards at the 1×10^{-6} level.

References

1. Molinar G. F., Rebaglia B., Sacconi A., Legras J. C., Vailleau G. P., Schmidt J. W., Stoup J., Flack D. R., Sabuga W., Jusko O., *Dimensional Measurements and Calculation of Effective Area. Phase A1 of the CCM Comparison in the Pressure Range 0.05 to 1 MPa (gas medium, gauge mode)*, PTB Final Report, November 1999; Molinar G. F., Rebaglia B., Sacconi A., Legras J. C., Vailleau G. P., Schmidt J. W., Stoup J. R., Flack D. R., Sabuga W., Jusko O., *Metrologia*, 1999, **36**, 657-662.
2. Legras J. C., Jäger J., Molinar G. F., Schmidt J. W., Phase A2 of the CCM Key Comparison in the Pressure Range 50 – 1000 kPa, Pressure measurements, Report in preparation.
3. Legras J. C., Schatz B., Delajoud P., La référence nationale de pression du BNM dans le domaine de 10 à 400 kPa, *Bull. BNM*, 1986, **65**, 39-53.
4. Maghenzani R., Molinar G., Marzola L., Kulshrestha R. K., *J. Phys. E: Sci. Instrum.*, 1987, **20**, 1173-1179.
5. Klingenberg G., Lüdicke F., Characterization of a pressure balance from dimensional measurements and from pressure comparison experiments, *PTB-Mitteilungen*, 1991, **101**, 7-18.
6. Klingenberg G., Legras J. C., *Metrologia*, 1993/94, **30**, 603-606.
7. Sabuga W., Bestimmung der wirksamen Querschnittsfläche von neuen 5-cm^2-Gas und Öl-Kolbenzylindersystemen, *PTB Annual Report*, 1996, 213-214.