EUROMET.M.P-S4 FINAL REPORT

BILATERAL PRESSURE COMPARISON IN GAS MEDIA BETWEEN LNE (FRANCE) and UME (TURKEY) (GAUGE MODE)

IN THE RANGE FROM 0,04 to 1,75 MPa

Ilknur KOCAS[1], Jean-Claude LEGRAS[2], Pierre OTAL[2]

1 TUBITAK UME, P.O. Box 54, 41470 Gebze-KOCAELI, TURKEY
e-mail: ilknur.kocas@ume.tubitak.gov.tr

2 Laboratoire National d'Essais (LNE), 1, rue Gaston Boissier
75724 PARIS CEDEX 15 France,
e-mail: Jean-Claude.Legras@lne.fr and pierre.otal@lne.fr

ABSTRACT

The EUROMET project N° 861 is organised using the same procedures as for the CCM.P-K1 (b) key comparison, in the pressure range 0,04 to 1,75 MPa. LNE participated in the CCM.P-K1 (b) comparison as co-pilot. Key pressure points 100 kPa, 500 kPa and 1 MPa have been selected in the present comparison in order to allow a link to the CCM one.

A pressure balance equipped with a 200 mm^2-effective area piston-cylinder is compared to the pressure standards of each participating institute in the range 0,04 to 1,75 MPa. From these measurements, the effective area at null pressure of the assembly is calculated. The results are compared to those obtained in the CCM.P-K1b comparison.

1. Introduction

There has been considerable interest in international and bilateral key comparisons to ensure worldwide uniformity of measurements and their traceability to the International System of Units (SI). Also, sharing of experiences between the laboratories is one of the important issue and value in this kind of co-operations.

This bilateral inter-laboratory comparison is organized in the frame of EUROMET Technical Committee for Mass and Related Quantities as the EUROMET Project N° 861. The target is to linking this bilateral comparison to the corresponding CIPM key comparison identified as CCM.P-K1b [1]. This bilateral comparison was also carried out as per "Guidelines of the CIPM key Comparisons".

The pneumatic piston gage is used as a transfer standard in this comparison.

The measurements on the transfer Piston-Cylinder assembly used in this comparison were performed firstly in France with LNE base and mass set, then the same measurements were realised in Turkey with UME base and mass set. The cross-floating method was used as a calibration method to determine the effective area of transfer piston-cylinder unit. All temperature probes and mass sets have been calibrated before the comparison measurements.

2. Apparatus

a. Transfer standard

The transfer standard has a 200 mm² nominal effective area. It is produced by DH Instruments as PG7601 model. It has been under operation since 2000 in UME laboratory. Transfer standard's characteristics and the first calibration values are listed in Table 1.

Piston-cylinder characteristics	
Nominal sensitivity	50 kPa/kg
Calculated pressure distortion coefficient	$-1,67x10^{-6}$ 1/MPa
Thermal expansion coefficient	$(9,0 \times 10^{-6})$ °C^{-1}
Minimum operating pressure (gauge mode)	10 kPa
Maximum operating pressure (with 50 kg)	2500 kPa
Maximum drop rate	0,5 mm/min

Piston characteristics	
Nominal diameter	16 mm
Piston material	Piston-tungsten carbide
Equivalent piston density	6910 kg/m^3
Young's modulus	600 GPa
Poisson's ratio	0,22
Cylinder characteristics	
Cylinder material	Tungsten carbide
Linear thermal expansion coefficient	$(4,5x10^{-6})$ °C^{-1}
Young's modulus	600 GPa
Poisson's ratio	0,22
Mounting system used	Negative, free deformation (measured pressure applied outside the cylinder)

Table 1 – Data relating to the transfer piston-cylinder assembly

b. Laboratory standards

The main characteristics of the laboratory standards are reported in table 2.

LNE standard.

The LNE standard used for the comparison is a secondary one. The 2 MPa-unit is one of the piston-cylinder assemblies used for defining the LNE gas-operated pressure scale. It is used for confirming the extrapolation of the scale from the primary standard "APX" [2] to the 10 MPa pressure balance. The effective area at null pressure has been determined by direct comparison with the APX in the range from 40 to 1000 kPa. The pressure distortion coefficient was calculated using Lamé's equations. The linear thermal expansion coefficient of the transfer standard has been determined in [3] for example.

It is the first comparison realised with this standard. LNE participated to several comparisons in overlapping ranges:

- CCM.P-K1 b and EUROMET.M.P-K3b in the range 10 to 1000 kPa [3, 4]
- CCM.P-K1 c in the range 0,1 to 7 MPa [5]
- EUROMET.M.P-K5 in the range 0,1 to 4 MPa [6]

UME standard

UME laboratory standard's effective area and uncertainty values are taken from PTB certificate (0089 PTB05). The same piston-cylinder assembly has been measured dimensionally on 2 February 2006 by LNE through the DH Budenberg Company. The effective area issued from these measurements was 490, 2597 mm^2 with a standard uncertainty lower then 3×10^{-6} [3, 8]. These values are not used in this work because they were obtained only by diameter measurements.

Other comparisons have been carried out in a similar range by different NMIs.

- EUROMET 537 in the range 15 kPa to 7 MPa [10]
- Bilateral comparison between PTB and LNE [8]
- Bilateral comparison between NIST and NPLI [9]

	LNE	UME
Piston-cylinder serial number	6394	9145
Manufacturer	Desgranges et Huot	Desgranges et Huot
Model	DH 5111	DH 5111
Measurement range	0,04 -2 MPa	0,04 -2 MPa
Piston material	Tungsten carbide	Tungsten carbide
Cylinder material	Tungsten carbide	Tungsten carbide
Operation mode, free deformation or controlled clearance	Free deformation	Free deformation
Zero- pressure effective area A_0 at reference temperature, in mm^2	490,2530	490,2587
Relative uncertainty of A_0, in 10^{-6} (k=1)	4,7	9,9
Pressure distortion coefficient (λ) in MPa^{-1}	1,2 $x10^{-6}$	1,37 $x10^{-6}$
Uncertainty of λ, in MPa^{-1}(k=1)	0,12 $x10^{-6}$	0,15 $x10^{-6}$
Relative uncertainty of mass pieces, in 10^{-6} (k=1)	0,75	0,60
Linear thermal expansion coefficient of piston (α_p), in °C^{-1}	(4,5$x10^{-6}$)	(4,5$x10^{-6}$)
Linear thermal expansion coefficient of piston (α_c), in °C^{-1}	(4,5$x10^{-6}$)	(4,5$x10^{-6}$)
Reference temperature (t_0), in °C^{-1}	20	20
Local gravity (g), in m/s^2	9,809273	9,802310
Relative uncertainty of g, in 10^{-6} (k=1)	0,1	0,1
Height difference between laboratory standard (LS) and transfer standard(TS)	225	29,6
Uncertainty of h in mm (k=1)	0,5 mm	0,6 mm

Table 2 – Characteristics of the Laboratory standards

3. Calibration procedure

Height difference between reference gauge and transfer gauge are minimized, piston gauges are rotated manually during the measurement process. Before the measurements, piston gauges were loaded to maximum pressure value to check their performances (cleanliness, piston fall rate, leakage).

The calibration procedure followed well-known methods for cross-float comparison between laboratory standard and transfer standard. Piston gages were loaded with masses in ratios approximately the same as the ratios in effective area. The pressure was increased to float the pistons, and the pressure equilibrium between laboratory standard and transfer standard was determined by the fall rate analyse method. If the piston gages were not in equilibrium, small fractional masses were added or subtracted from the laboratory standard. High purity nitrogen was used during the comparison.

During the comparison:

- Temperature of the piston gauges was measured and monitored with platinum resistance thermometers (PRTs) attached near the pistons,
- For minimising air flow effects around piston gauges, bases are housed in an appropriate cabin,
- Both piston gauges were mounted on a rigid base to minimize vibration and magnetic effects,
- A pressure head correction term was applied to compensate for the difference in the reference levels of the pistons.

Before the measurement cycle, each piston was levelled to ensure the verticality of its axis and the system was checked for leaks to its full-scale pressure value of 1,75 MPa.

The comparison was performed at LNE in August 2005 and at UME in March 2006.

The pressures of the comparison were 0,04-0,1-0,35-0,5-0,6-0,8-1,0-1,25-1,50-1,75 MPa. Five cycles with 10 measuring point in ascending and decreasing pressure were realised. About 35 minutes time was adequate for changes in pressure to bring the piston gauges in to equilibrium.

4. Calculations

1. Mathematical model

The effective area at null pressure has been calculated from (1).

$$\mathbf{A}_o^{'} = \frac{\sum_i \left[\mathbf{m}_i \mathbf{g}(1-\rho_a/\rho_{mi})\right] \pm \rho \mathbf{g} \Delta \mathbf{h}}{\mathbf{p}^{'}\left[1+(\alpha_p+\alpha_c)(\mathbf{t}^{'}-\mathbf{t}_o^{'})\right]\left[1+\lambda_{th}^{'}\mathbf{p}\right]} \quad (1)$$

$A_0^{'}$: Zero- pressure effective area A0 at reference temperature

m_i : Applied mass

g : Local gravity

ρ_a : Air density

ρ_{mi} : Mass density

$p^{'}$: Pressure measured by the laboratory standard at its reference level

λ : Transfer standard pressure distortion coefficient

α_p : Transfer standard linear thermal expansion coefficient

α_c : Transfer standard linear thermal expansion coefficient

$t^{'}$: Measurement temperature

$t_0^{'}$: Reference temperature (20 °C)

The results of the comparison are presented in Tables 3 and 4, and Figure 1 and 2, for LNE and UME respectively.

LNE							
Nom.Pres	A'_0 **(mm²)**					A'_0 **(mm²)**	**Standard deviation**
MPa	**Cycle 1**	**Cycle 2**	**Cycle 3**	**Cycle 4**	**Cycle 5**	**Mean**	**(x 10^{-4} mm²)**
0,04	196,1079	196,1077	196,1077	196,1078	196,1084	196,1079	1,5
0,1	196,1067	196,1059	196,1067	196,1062	196,1069	196,1065	2,0
0,35	196,1080	196,1076	196,1080	196,1080	196,1077	196,1079	0,9
0,5	196,1072	196,1074	196,1072	196,1074	196,1075	196,1073	0,7
0,6	196,1074	196,1076	196,1076	196,1072	196,1078	196,1075	1,3
0,8	196,1070	196,1072	196,1071	196,1072	196,1075	196,1072	1,1
1	196,1076	196,1076	196,1071	196,1074	196,1075	196,1074	1,1
1,25	196,1077	196,1078	196,1076	196,1076	196,1078	196,1077	0,4
1,5	196,1078	196,1073	196,1072	196,1075	196,1077	196,1075	1,3
1,75	196,1073	196,1077	196,1076	196,1077	196,1078	196,1076	0,9

Table 3- Effective area values against the pressure for each cycle (LNE)

UME							
Nom.Pres	A'_0 **(mm²)**					A'_0 **mm²)**	**Standard deviation**
MPa	**Cycle 1**	**Cycle 2**	**Cycle 3**	**Cycle 4**	**Cycle 5**	**Mean**	**(x 10^{-4} mm²)**
0,04	196,1085	196,1086	196,1091	196,1082	196,1085	196,1086	1,5
0,1	196,1071	196,1076	196,1080	196,1073	196,1074	196,1075	1,8
0,35	196,1079	196,1082	196,1082	196,1078	196,1080	196,1080	0,9
0,5	196,1081	196,1081	196,1084	196,1079	196,1082	196,1081	0,9
0,6	196,1080	196,1083	196,1083	196,1079	196,1081	196,1081	0,8
0,8	196,1075	196,1081	196,1082	196,1080	196,1080	196,1079	1,4
1	196,1082	196,1081	196,1083	196,1079	196,1081	196,1081	0,8
1,25	196,1085	196,1084	196,1084	196,1077	196,1080	196,1082	1,6
1,5	196,1083	196,1083	196,1081	196,1078	196,1080	196,1081	1,1
1,75	196,1085	196,1081	196,1085	196,1081	196,1081	196,1083	1,0

Table 4- Effective area values against the pressure for each cycle (UME)

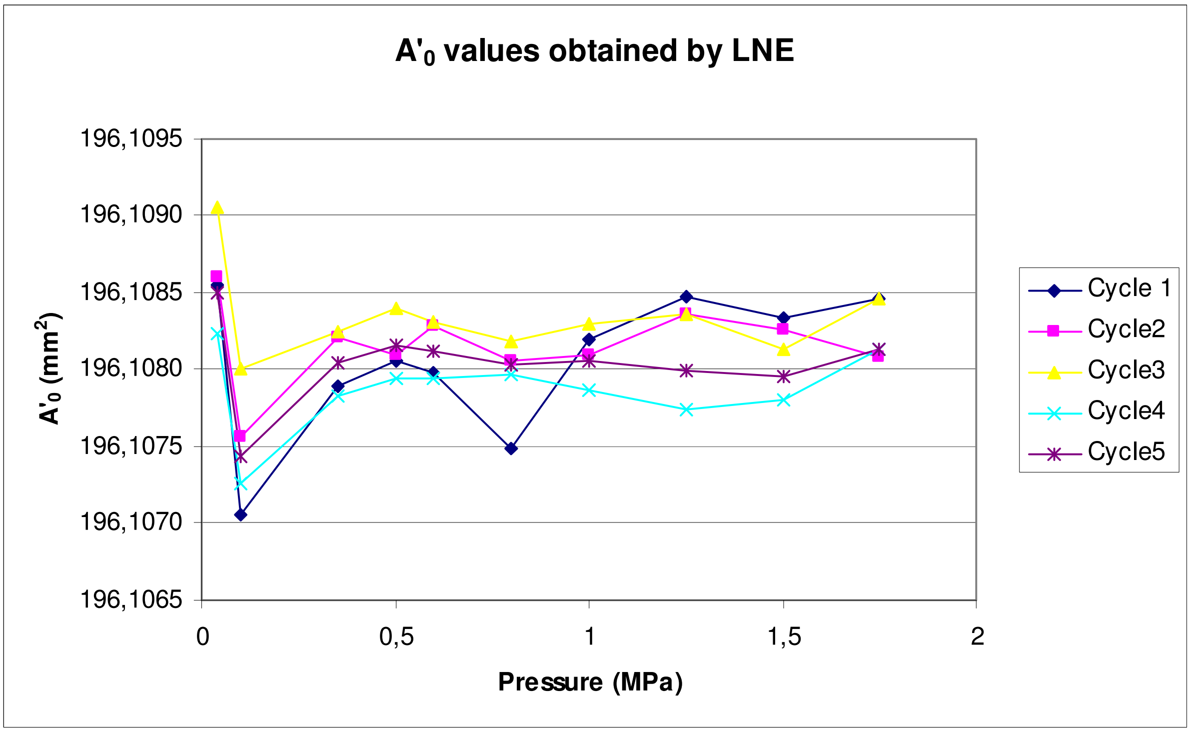

Figure 1- Effective area values against the pressure for each cycle (LNE)

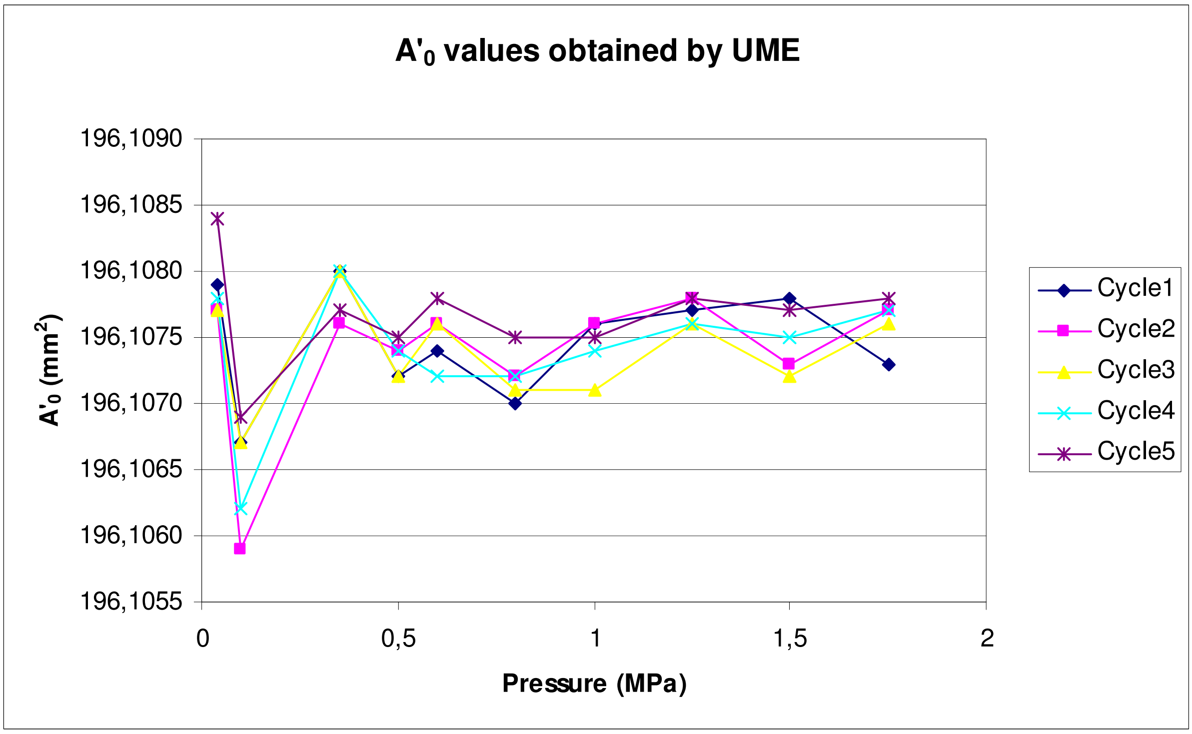

Figure 2- Effective area values against the pressure for each cycle (UME)

The mean effective area determined by both laboratories is given as a function of pressure in Figure 3.

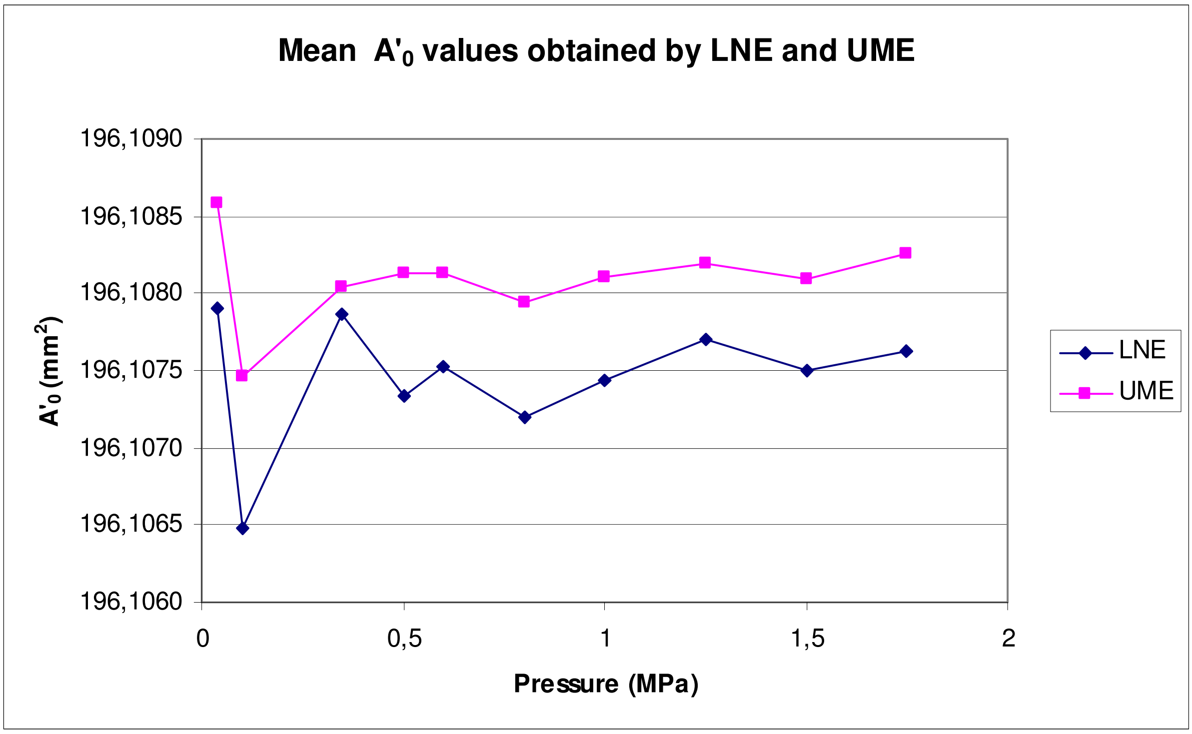

Figure 3 - Mean effective area values vs pressure for each cycle obtained by LNE and UME

The uncertainties in the measurements of effective area arise from Type A and Type B sources. The following mathematical expression (2) is used to calculate A type uncertainty component.

$$u_A(A'_0) = \sqrt{\frac{\sum_{k=1}^{n}\left(A'_{0,k} - A'_{0,av}\right)^2}{n(n-1)}} \qquad (2)$$

Type A uncertainty is added in quadrature with Type B uncertainty to give the combined uncertainty in the average effective area (3).

$$u_C(A'_0) = \sqrt{u_A(A'_0)^2 + u_B(A'_0)^2} \quad (3)$$

Relative standard uncertainty values $u_C(A'_0)/A'_0$ are evaluated at each pressure.

LNE						
Nominal pressure	**Average A'o value**	**Standard deviation of A'o**	**Uncertainty due to p'**	**Uncertainty due to M'**	**Uncertainty due to λ'**	**Uncertainty of A'o**
(MPa)	**(mm^2)**	**(x 10^6)**	**(x 10^6)**	**(x 10^6)**	**(x 10^6)**	**(x 10^6)**
0,04	196,1079	1,5	6,4	3,8	0,003	7,6
0,1	196,1065	2,0	5,6	2,0	0,008	6,3
0,35	196,1078	0,9	5,2	1,1	0,029	5,4
0,5	196,1073	0,7	5,2	1,0	0,042	5,3
0,6	196,1075	1,3	5,2	1,0	0,050	5,4
0,8	196,1072	1,1	5,2	0,95	0,067	5,4
1	196,1074	1,1	5,2	0,92	0,084	5,3
1,25	196,1077	0,4	5,1	0,90	0,11	5,2
1,5	196,1075	1,3	5,1	0,88	0,13	5,4
1,75	196,1076	0,9	5,1	0,87	0,15	5,3

Table 5 - Uncertainty components (LNE)

UME						
Nominal pressure	**Average A'o value**	**Standard deviation of A'o**	**Uncertainty due to p'**	**Uncertainty due to M'**	**Uncertainty due to λ'**	**Uncertainty of A'o**
(MPa)	**(mm^2)**	**(x 10^6)**	**(x 10^6)**	**(x 10^6)**	**(x 10^6)**	**(x 10^6)**
0,04	196,1086	1,5	10,3	2,2	0,007	10,6
0,1	196,1075	1,8	9,9	0,9	0,018	10,1
0,35	196,1080	0,9	9,8	0,7	0,063	9,9
0,5	196,1081	0,9	9,8	0,6	0,089	9,8
0,6	196,1081	0,8	9,8	0,5	0,107	9,8
0,8	196,1079	1,4	9,8	0,44	0,143	9,9
1	196,1081	0,8	9,7	0,40	0,179	9,8
1,25	196,1082	1,6	9,8	0,36	0,224	10,0
1,5	196,1081	1,1	9,8	0,33	0,268	9,8
1,75	196,1083	1,0	9,7	0,29	0,313	9,8

Table 6 - Uncertainty components (UME)

b. Degree of equivalence

The degree of equivalence is evaluated using the standard method of a CCM comparison by calculating the difference in the average effective area of the transfer standard, as found by LNE and UME, at similar pressure points. The degree of equivalence, represented for each pressure point as the normalised error E_n calculated with help of well-known mathematical expression below is presented in table 7:

$$E_n = \frac{\lfloor A_{0(LNE)} - A_{0(UME)} \rfloor}{\sqrt{U(A_0)^2_{LNE} + U(A_0)^2_{UME}}} \qquad (5)$$

The combined uncertainty is expressed as u_c and the expended uncertainty U is expressed using a coverage factor k=2 and with the mathematical expression (6):

$$U = \frac{2\sqrt{u(A_0)^2_{LNE} + u(A_0)^2_{UME}}}{(A_0)_{\mathrm{Re}f}} \qquad (6)$$

c. Results

As a result of 5 cycles of measurements, the value of the effective areas of the transfer standard are listed in Table 5 for LNE and Table 6 for UME. In the same tables the standard deviations of effective areas are listed.

Nominal pressure	**LNE**		**UME**		**Difference LNE-UME**	**Combined uncertainty (k=2)**	**E_n value**
	Average of A'o value	**Uncertainty of A'o (k=2)**	**Average of A'o value**	**Uncertainty of A'o (k=2)**			
(MPa)	**(mm^2)**	**(x 10^6)**	**(mm^2)**	**(x 10^6)**	**(x 10^6)**	**(x 10^6)**	**-**
0,04	196,1079	15,1	196,1086	21,3	-3,5	26	-0,13
0,1	196,1065	12,5	196,1075	20,3	-4,9	24	-0,21
0,35	196,1078	10,9	196,1080	19,7	-1,2	23	-0,05
0,5	196,1073	10,7	196,1081	19,7	-4,2	22	-0,19
0,6	196,1075	10,9	196,1081	19,6	-3,2	22	-0,14
0,8	196,1072	10,7	196,1079	19,8	-3,8	23	-0,17
1	196,1074	10,7	196,1081	19,6	-3,6	22	-0,16
1,25	196,1077	10,5	196,1082	19,9	-2,5	22	-0,11
1,5	196,1075	10,7	196,1081	19,7	-3,0	22	-0,14
1,75	196,1076	10,6	196,1083	19,6	-3,3	22	-0,15

Table 7 - Degree of equivalence (E_n)

For realising the link to CCM.P-K1b key comparison, the results of this comparison are compared with the results of CCM.P-K1b key comparison. UME results have been added in Tables 8, 9 and 10 to be compared with the other laboratories using the formulas below:

$(D_{UME,j})_{CCM} = (D_{UME,LNE})_{EUR} + (D_{LNE,,j})_{CCM}$

where $(D_{UME,j})_{CCM}$ is the difference between UME and the laboratory of rank j in the CCM comparison.

$(U_{UME,j})_{CCM} = \{u^2[(D_{UME,LNE})_{EUR}] + u^2[(D_{LNE,,j})_{CCM}] + 2\, u[(D_{LNE})_{CCM} \,.\, u[(D_{LNE})_{CCM} \,.\, \rho(LNE_{CCM}, LNE_{EUR})\}^{0,5}$

where $\rho(LNE_{CCM}, LNE_{EUR})$ is the correlation coefficient of the LNE values for the two comparisons, estimated to be 0,7.

These formulas have been established by considering a perfect coherency between the two standards used at LNE for both comparisons.

p =100 kPa										
Lab.	LNE		INRIM		NIST		PTB		UME	
	D_{ij} (x 10^6)	**U_{ij} (x 10^6)**	**D_{ij} (x 10^6)**	**U_{ij} (x 10^6)**	**D_{ij} (x 10^6)**	**U_{ij} (x 10^6)**	**D_{ij} (x 10^6)**	**U_{ij} (x 10^6)**	**D_{ij} (x 10^6)**	**U_{ij} (x 10^6)**
LNE			+ 0,8	17	- 7,3	19	- 5,6	12	- 5,1	24
INRIM	- 0,8	17			- 8,1	23	- 6,5	18	- 5,9	27
NIST	+ 7,3	19	+ 8,1	23			+ 1,6	20	+ 2,2	28
PTB	+ 5,6	12	+ 6,5	18	- 1,6	20			+ 0,5	24
UME	+ 5,1	24	+ 5,9	27	- 2,2	28	- 0,5	24		

Table 8- CCM.P-K1.b results which is included UME at 100 kPa

p = 600 kPa										
Lab.	LNE		INRIM		NIST		PTB		UME	
	D_{ij} (x 10^6)	U_{ij} (x 10^6)	D_{ij} (x 10^6)	U_{ij} (x 10^6)	D_{ij} (x 10^6)	U_{ij} (x 10^6)	D_{ij} (x 10^6)	U_{ij} (x 10^6)	D_{ij} (x 10^6)	U_{ij} (x 10^6)
LNE			+ 2,8	15	+ 3,3	23	- 4,5	10	- 3,1	22
INRIM	- 2,8	15			+ 0,5	26	- 7,3	15	- 5,9	25
NIST	- 3,3	23	- 0,5	26			- 7,8	23	- 6,4	31
PTB	+ 4,5	10	+ 7,3	15	+ 7,8	23			+ 1,4	22
UME	+ 3,1	22	+ 5,9	25	+ 6,4	31	- 1,4	22		

Table 9 - CCM.P-K1.b results which is included UME at 600 kPa

p =1000 kPa										
Lab.	LNE		INRIM		NIST		PTB		UME	
	D_{ij} (x 10^6)	U_{ij} (x 10^6)	D_{ij} (x 10^6)	U_{ij} (x 10^6)	D_{ij} (x 10^6)	U_{ij} (x 10^6)	D_{ij} (x 10^6)	U_{ij} (x 10^6)	D_{ij} (x 10^6)	U_{ij} (x 10^6)
LNE			+ 2,1	15	+ 3,4	27	- 3,7	10	- 3,6	22
INRIM	- 2,1	15			+ 1,4	29	- 5,7	15	- 5,6	25
NIST	- 3,4	27	- 1,4	29			- 7,1	27	- 7,0	34
PTB	+ 3,7	10	+ 5,7	15	+ 7,1	27			+ 0,1	22
UME	+ 3,6	22	+ 5,6	25	+ 7,0	34	- 0,1	22		

Table 10 - CCM.P-K1.b results which is included UME at 1000 kPa

5. Conclusion

The effective area of the transfer standard used in this bilateral comparison was determined by cross-floating method. During the comparison period only the piston-cylinder assembly was circulated between laboratories. Other elements, such as the base, the temperature probe and the measuring system for the fall rate of the piston, were provided by both laboratories.

The effective area and the standard uncertainty of the effective area were calculated for each pressure value. The results obtained by both laboratories are in agreement, as all the E_n values are less than 0,2. Both laboratories used the same type of piston cylinder assembly produced by the same manufacturer with similar physical characteristics, but the characterisation methods were different.

If the UME laboratory standard piston-cylinder assembly had been fully measured dimensionally the uncertainty of effective area of this assembly could be in the same level uncertainty as the LNE piston-cylinder assembly.

The bilateral comparison results validate and also demonstrate the method employed for linking the present comparison to the CIPM -K1b key comparison.

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