Final Report on COOMET Key Comparison of National Pressure Standards in the Range 100 Pa to 5 kPa of Gauge Pressure (COOMET.M.P-K14)

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ABSTRACT

This report describes a COOMET key comparison of pneumatic gauge pressure standards of four National Metrology Institutes, listed in the chronological order of their measurements, that was carried out in the period from November 2008 to March 2010 in order to determine their degree of equivalence in the range of 100 Pa to 5 kPa of gauge pressure. The pilot laboratory was PTB. The reference pressure standards of the participants were of different design. The transfer standard was a piston gauge model V1600 of the company *Pressurements*. The quantity under comparison was the effective area of the transfer standard at different pressure values reported together with uncertainty contributions and the conclusive combined uncertainty of measurement. All participants' results agree with the key comparison reference values within the expanded uncertainties calculated with a coverage factor 2, all but one results even within the standard uncertainties. For the participants' results compared in pairs, all of totally 48 pairs show agreement within the expanded and 46 pairs within the standard uncertainties. The results of the comparison demonstrate equivalence of the laboratory standards and support their measurement capabilities stated in the KCDB of BIPM.

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1. INTRODUCTION

At the COOMET TCM meeting held at KazInMetr, Astana, on 8 October 2008, it was decided to carry out a key comparison (KC) in the range of 5 kPa of pneumatic gauge pressure. Point of aim of this comparison was to state the equivalence of the national pressure standards of the participating national metrological laboratories in the 5 kPa range. Two participants in this comparison, the Physikalisch-Technische Bundesanstalt (PTB), Germany, and the Czech Metrology Institute (CMI), Czech Republic, are also participants in the respective KCs of EURAMET, EURAMET.M.P-K4.2010, being in progress, and a CCM KC being still in preparation. This will allow a link of the current COOMET KC to the corresponding EURAMET and CCM KCs.
PTB was agreed to be a pilot laboratory in this KC and provided a transfer standard (TS) for this KC.

The comparison was carried out in accordance with its Technical Protocol which specified the procedures to be followed in the comparison and had been prepared in accordance with the Guidelines for CIPM Key Comparisons, 1 March 1999.

2. PARTICIPANTS

The laboratory standards (LS) used in this comparison were of different types, namely a diving bell manometer, deadweight and force-controlled piston gauges, and a water column micromanometer. Different methods were applied by the participants to compare their standards with the TS. All uncertainties relating to the participants standards and their results in this report are standard ones if not explicitly indicated as expanded.

2.1 PTB pressure standards and measurement method

Two different laboratory standards were used by PTB in this KC. For the pressure values up to nominally 4 kPa, the diving bell known as TGM 5 with the mass set identified by g01ma2 was compared to TS. This bell manometer was developed at the ASMW [1] and was transferred to PTB in 1990 acting there as a reference standard for small gauge and differential pressures up to 4 kPa. In order to cover this pressure range it was equipped with Fluorinert instead of nonane as a working fluid, and, moreover, the load mechanism was directly attached to the bell. It was used in a comparison of European differential pressure standards from 3 Pa to 1 kPa [2]. The effective area of the diving bell is traceable to a primary mercury manometer of PTB. Table 1 summarizes properties of the diving bell manometers and the related measurement conditions.

Table 1. PTB diving bell manometer for pressures up to 4 kPa and measurement conditions

Type of device	Diving bell manometer
Manufacturer	ASMW, Germany
Measurement range in Pa, mode	(1 – 4000), gauge
Material of diving bell or piston	brass
Working liquid (for diving bell manometer)	Fluorinert
Pressure-transmitting gas	Air or nitrogen
Zero-pressure effective area (A_0) at reference temperature in m^2	$486.880 \cdot 10^{-4}$
Relative uncertainty of A_0 in 10^{-6}	35.5
Uncertainty of mass (m) pieces or of mass measurement	$125\ \text{mg} + 2 \cdot 10^{-6} m$
Linear thermal expansion coefficient of diving bell manometer (α_p) in °C^{-1}	$36 \cdot 10^{-6}$
Reference temperature (t_0) in °C	20
Local gravity (g) in m/s^2	9.812533
Relative uncertainty of g in 10^{-6}	0.6
Height difference between laboratory standard (LS) and TS (h, positive if LS is higher than TS) in mm	0
Uncertainty of h in mm	5

For pressure values 4800 and 5000 Pa a gas-operated pressure balance TL1568 from *Ruska* company was used together with the mass set p04ma. The properties of this pressure balance are presented in Table 2.

Table 2. PTB pressure balance for pressures of 4.8 and 5 kPa

Type of device	TL1568
Manufacturer	Ruska
Measurement range in kPa, mode	1.4 – 180, gauge and absolute
Material of diving bell or piston	steel
Material of cylinder (for pressure balance)	Tungsten carbide
Pressure-transmitting gas	Dry gas/nitrogen
Zero-pressure effective area (A_0) at reference temperature in m^2	3.356606
Relative uncertainty of A_0 in 10^{-6}	5.7
Uncertainty of mass pieces at p_{max}, mg	3
Linear thermal expansion coefficient of diving bell or piston (α_p) in °C^{-1}	$1.1 \cdot 10^{-5}$
Linear thermal expansion coefficient of cylinder (for pressure balance, α_c) in °C^{-1}	$4 \cdot 10^{-6}$
Reference temperature (t_0) in °C	20
Local gravity (g) in m/s^2	9.812533
Relative uncertainty of g in 10^{-6}	0.6
Height difference between laboratory standard (LS) and TS (h, positive if LS is higher than TS) in mm	-64.0
Uncertainty of h in mm	5

2.2 VNIIM pressure standard and measurement method

The VNIIM pressure standard is a water column micromanometer which is a part of the national special standard of unit of pressure GET 95-75 (GOST 8.187-76 «State system for ensuring the uniformity of measurements. The State special standard and All-Union verification schedule for means of measurements of the difference of pressures up to $4 \cdot 10^4$ Pa») having designation "МКШ". Physically, it is a water-based compensated micromanometer working in a pressure range up to 5 kPa with a traceability of its properties to VNIIM standards. With the TS used in the KC and under the conditions of the experiment, the maximum pressure of the laboratory standard was limited by 4.8 kPa. Hence, deviating from the Technical Protocol, the highest measured pressure was 4.8 kPa. TS was transported to VNIIM by the pilot laboratory on 21 November 2008 and measured there between 24 and 26 November 2008. It was returned to PTB on 28 November 2008. The properties of the VNIIM pressure standard and the measurement conditions are given in Table 3.

Table 3. VNIIM water column micromanometer and measurement conditions

Type of device	Water column micromanometer
Manufacturer	VNIIM, Russia
Pressure (*p*) measurement range in Pa	100 - 5000
Relative uncertainty of pressure	for 100 Pa: $2\cdot10^{-4}$
	for 5000 Pa: $1.6\cdot10^{-5}$
Working liquid	Distilled water
Density of working liquid (ρ_l) kg/m^3	998.202
Relative uncertainty of ρ_l in 10^{-6}	1
Reference temperature of working liquid (t_0) in °C	20
Uncertainty of t_0 in °C	0.1
Pressure-transmitting gas	Nitrogen
Local gravity (g) in m/s^2	9.819308
Relative uncertainty of g in 10^{-6}	0.2
Height difference between laboratory standard (LS) and TS (h, positive if LS is higher than TS) in mm	20
Uncertainty of h in mm	1.0

2.3 CMI pressure standard and measurement method

The CMI pressure standard used in this comparison is a force-balanced piston gauge (FPG) manufactured by *DH Instruments*, USA, model FPG 8601, whose description of the physical principle is described in [3] and the commercial realisation in [4]. The setup of the CMI FPG was modified with respect to the commercial one in such a way that a turbo-molecular pump was added to the pumping system of the reference side in order to reduce the residual pressure on the reference side and, therefore, its uncertainty contribution. A thorough evaluation of the CMI system is presented in [5]. The effective area was evaluated both by a dimensional measurement of the piston-cylinder geometry and by cross-floating techniques comparing it against the CMI PG 7601 pressure balance. These comparisons were performed with the CMI standards during the year 2002. An intercomparison with the Slovak national metrology institute (SMU) was performed in December 2002 from 2 to 15 kPa in both gauge and absolute pressure mode with a Bell and Howell pressure balance as a transfer standard. Another intercomparison with the Finnish national metrology institute, (MIKES), was realized in July 2003 from 1 Pa to 15 kPa in gauge pressure mode and from 6 Pa to 15 kPa in absolute pressure mode, the MIKES standard being another FPG 8601. A third bilateral comparison, EURAMET.M.P-S2, was carried out with PTB that used an FRS5 force-compensated pressure balance manufactured by *Furness Controls*, UK.
For the actual measurements an MKS Baratron differential pressure cell (DPC) was used as a zero pressure indicator installed between the CMI FPG and the TS. With this DPC CMI could automate the measurements using software Compass of the FPG. The technical details of the CMI standard and the measurement conditions are given in Table 4.

The transfer standard was transported to CMI by the pilot laboratory on 11 May 2009. The measurements were performed in the period from 12 to 13 May 2009, according to the Technical Protocol. Afterwards, TS was brought back to PTB on 15 May 2009.

Table 4. CMI force-balanced piston gauge and measurement conditions

Type of device	FPG 8601
Manufacturer	*DH Instruments*
Measurement range in Pa	1 – 15000, gauge and absolute
Material of diving bell or piston	tungsten-carbide
Material of cylinder (for pressure balance)	tungsten-carbide
Pressure-transmitting gas	nitrogen
Zero-pressure effective area (A_0) at reference temperature in m^2	$9.80527 \cdot 10^{-4}$
Relative uncertainty of A_0 in 10^{-6}	12.5
Uncertainty of mass (m) pieces or of mass measurement	0.5 mg + $2 \cdot 10^{-6} m$
Linear thermal expansion coefficient of diving bell or piston (α_p) in °C^{-1}	$4.5 \cdot 10^{-6}$
Linear thermal expansion coefficient of cylinder (for pressure balance, α_c) in °C^{-1}	$4.5 \cdot 10^{-6}$
Reference temperature (t_0) in °C	20
Local gravity (g) in m/s^2	9.809272
Relative uncertainty of g in 10^{-6}	0.8
Height difference between laboratory standard (LS) and TS (h, positive if LS is higher than TS) in mm	124
Uncertainty of h in mm	$2/\sqrt{3}$

2.4 VMC pressure standard and measurement method

The VMC laboratory standard was a piston gauge Metran-505, Vozduch I, manufactured by *Metran*, Russia, serial number 094, made in 2008. The device was calibrated at PTB (Germany) in August 2009. Thus, its pressure value is traceable to the German national pressure standards. The pressure values realised with pistons M and B in combination with weights and the uncertainties of that pressure values were determined at PTB and reported in Calibration certificate Nr. 0050 PTB 2009. Finally, the effective area $A(p)$ of piston M assembly and piston B assembly were calculated at VMC. The technical details of the VMC standard and the measurement conditions are given in Table 5.

Table 5. VMC piston gauge and measurement conditions

Type of device	Metran-505, Vozduch I, serial Nr. 094
Manufacturer	*Metran*
Measurement range, mode	20 Pa to 25 kPa: gauge pressure 5 Pa to 25 kPa: differential pressure
Material of pistons	up to 125 Pa - aluminium 160 Pa to 25 kPa - stainless steel
Material of cylinder (for pressure balance)	stainless steel
Pressure-transmitting gas	Air/nitrogen
Zero-pressure effective area (A_0) at reference temperature in m^2	3 pistons $4.91 \cdot 10^{-4}$
Relative uncertainty of A_0 in 10^{-6}	19
Uncertainty of mass (m) pieces or of mass measurement, mg	7.1
Linear thermal expansion coefficient of piston and cylinder (α_p) in °C^{-1}	$(23 \pm 2) \cdot 10^{-6}$
Reference temperature (t_0) in °C	20
Local gravity (g) in m/s^2	9.814380
Relative uncertainty of g in 10^{-6}	5
Height difference between LS and TS (h, positive if LS is higher than TS) in mm	-6
Uncertainty of h in mm	5

TS and the laboratory standard were interconnected via a DPC using nitrogen as a pressure-transmitting medium. TS and the laboratory standard were loaded with masses corresponding to the nominal pressures as specified in the Technical Protocol and the residual pressure differences between them were measured with the DPC. Hence, the uncertainty of the DPC, which according to the pilot laboratory is

Uncertainty of zero indicator	0.02 Pa

was added to the uncertainty of the VMC standard. TS was brought to VMC on 23 November 2009 and measured between 24 and 27 November, before it was returned to PTB on 27 November 2009.

3. TRANSFER STANDARD

The transfer standard was described in detail in the technical protocol. It was a piston gauge V1600/1D manufactured by *Pressurements* Ltd., Bedfordshire, England, in 1998 and identified by serial number 10533-98. The assembly consisted of a cylinder identified by serial number B 192, which is engraved on the cylinder face, and three different pistons all carrying number N 192 on the lower piston face and identified by markings "1 mbar", "2 mbar" and "L". The nominal effective area of the assembly was $A_{0,\mathrm{nom}}$ = 4.91 cm^2.
The cylinder’s material was stainless steel, whereas piston "1 mbar" was made of aluminium and pistons "2 mbar" and "L" were fabricated again from stainless steel.

Following the manufacturer information, the thermal expansion coefficient of the assembly of cylinder and any of the pistons (either of steel or of aluminium) was taken as $(22 \pm 2)\cdot 10^{-6}$ °C^{-1} with its standard uncertainty based both on the manufacturer information and the experience of the pilot laboratory.

Table 6. Masses and densities of pistons

Piston	True mass in g	Density in kg/m^3
"1 mbar"	4.9999 ± 0.0005	2650 ± 25
"2 mbar"	9.9986 ± 0.0005	7920 ± 25
"L"	10.7333 ± 0.0005	7920 ± 25

The weight carrier was marked with "L" and made of aluminium.

Table 7. Masses and densities of the weight carrier

Weight carrier	True mass in g	Density in kg/m^3
	9.2776 ± 0.0005	2650 ± 25

Seven weights were identified by markings "1 mbar", "5 mbar", "10 mbar", "20_1 mbar", "20_2 mbar", and additionally by serial number N192 on each piece.

Table 8. Masses and densities of the weights

Weight	True mass in g	Density in kg/m^3
"1 mbar"	5.0042 ± 0.0005	2650 ± 25
"2_1 mbar"	10.0112 ± 0.0005	2650 ± 25
"2_2 mbar"	10.0110 ± 0.0005	2650 ± 25
"5 mbar"	25.0271 ± 0.0005	2650 ± 25
"10 mbar"	50.0398 ± 0.0005	7920 ± 25
"20_1 mbar"	100.0825 ± 0.0005	7920 ± 25
"20_2 mbar"	100.0824 ± 0.0005	7920 ± 25

In combination with the piston gauge, a zero pressure indicator FC 014 was delivered to the participants, which had been manufactured by *Furness Controls* and had serial number 92021254-1. This indicator had a scale interval of 0.02 Pa and a maximum differential pressure range of 100 Pa at a maximum acceptable line pressure of 100 kPa and a maximum overload differential pressure of 100 kPa.

4. ORGANIZATION AND CHRONOLOGY OF THE COMPARISON

The measurements were performed in the order and times given in Table 9.

Table 9. Chronology of measurements

Institute	Measurement start date	Measurement end date
PTB, initial investigation of TS	4 Nov 2008	17 Nov 2008
VNIIM	24 Nov 2008	26 Nov 2008
PTB, intermediate check of TS	30 Mar 2009	3 Apr 2009
CMI	12 May 2009	13 May 2009
PTB, intermediate check of TS	12 Nov 2009	12 Nov 2009
VMC	24 Nov 2009	27 Nov 2009
PTB, final check of TS	4 Mar 2010	9 Mar 2010

The pilot laboratory performed three measurement cycles and one half-cycle measurement (November 2009) of TS. The measurement in March 2010 was taken as a PTB contribution to this KC.

5. MEASUREMENT PROCEDURES

The transfer standard had to be handled and the piston-cylinder assembly mounted in accordance with the instructions given in the User's Manual Reference of the V1600 provided to the participants. TS was operated throughout all measurements by one member of the pilot laboratory (Mrs. Ahrendt).

The TS was recommended to be located close to the laboratory's reference standard to keep the pressure line between the two instruments as short as possible. The piston gauge V1600/ID of TS was equipped with two cylinders. The cylinder identified by "B" was used in the comparison measurements. The reference level of TS was the upper face of cylinder B. The horizontality of TS expected to be better than 0.1 mm/m was checked with a spirit level placed on the upper face of cylinder B. The temperature of TS was measured with a thermometer of the participant. It was attached to cylinder B of TS. The reference temperature of the comparison was 20 °C. For measurements performed at a temperature deviating from 20 °C, the effective area of the TS was referred to 20 °C using the piston-cylinder thermal expansion coefficient. The zero-pressure indicator was switched in the pressure line between the gauge of TS and the laboratory standard to control equality of the pressures generated by the two pressure standards and to avoid pressure gradients along the line. A bypass line with a valve was connecting both sides of the zero indicator to set its zero reading.
The working gas of the assembly was either dry air or nitrogen. The piston gauge of TS did not have any electronics to be warmed up, but the zero-pressure indicator had to be switched on at least 5 minute before the measurements. The piston had to be in a self-centred stable floating position. The time between a pressure level change and the acquisition of the data corresponding to the equilibrium of the laboratory standard and TS had to be not shorter than 3 minutes.
The measurements included four cycles each with nominal pressures generated in the following order (100, 200, 500, 1000, 2000, 3000 350, 4000, 5000, 5000, 4000, 3000, 2000, 1000, 500, 200, 100) Pa. Thus, 64 measurements were performed in total.

Table 10. Load of TS to generate the nominal pressures

Nominal pressure in Pa	Loads (pistons, weight carrier, weights)
100	Piston "1 mbar"
200	Piston "2 mbar"
500	Piston "L" + weight carrier "L"+ "1 mbar"
1000	Piston "L" + weight carrier "L" + "1 mbar" + "5 mbar"
2000	Piston "L" + weight carrier "L" + "1 mbar" + "5 mbar" + "10 mbar"
3000	Piston "L" + weight carrier "L" + "1 mbar" + "5 mbar" + "20_1 mbar"
4000	Piston "L" + weight carrier "L" + "1 mbar" + "5 mbar" + "10 mbar" + "20_1 mbar"
5000	Piston "L" + weight carrier "L" + "1 mbar" + "5 mbar" + "20_1 mbar" +"20_2 mbar"

Due to measurement set-up of the actual comparison, VNIIM was only able to measure a nominal pressure value of 4800 Pa as the highest achievable, see section 2.2. The corresponding combination of mass pieces for this point was the following:

Table 11. Load of TS to generate the nominal pressure of 4800 Pa

Nominal pressure in Pa	Loads (pistons, weight carrier, weights)
4800	Piston "L" + weight carrier "L" + "2_1 mbar" +"2_2 mbar" + "20_1 mbar" +"20_2 mbar"

Comparability of the results obtained at this pressure with the results of other participants obtained at 5 kPa was determined by measurements of the pilot laboratory performed at both nominal pressures, 4.8 and 5 kPa.
No additional/different loads were applied to the pistons of TS. The equilibrium between TS and the laboratory standard, which was controlled with the help of the zero-pressure indicator, was achieved by adjusting the pressure in the laboratory standard. In the case of VMC using a similar device to TS as a laboratory standard, the zero indicator reading was used as a direct measure of the pressure difference between TS and LS.

The effective area of the TS determined for a particular measurement (A_p) referred to 20 °C was calculated with the equation

$$A_p = \frac{g \sum m_i \cdot (1 - \rho_a / \rho_i)}{p\,[1 + (\alpha + \beta)(t - 20\,°\mathrm{C})\,]}, \qquad (1)$$

where

m_i are true masses of the piston, the weight carrier and the mass pieces placed on the weight carrier of TS;

ρ_i are densities of the parts with masses m_i;

ρ_a is air density;
g is local gravity acceleration;
p is pressure generated by the laboratory standard at the TS reference level;
$(\alpha+\beta)$ is thermal expansion coefficient of the piston-cylinder;
t is temperature of TS.

The values of p and ρ_a were calculated and t was measured by the participating laboratory. The conditions of the measurements are given in Annexes A1-A4.

6. RESULTS

6.1 Stability of the transfer standard

Prior to the comparison, the long-term stability of TS has been evaluated on the basis of calibration results obtained in 1999, 2001 and 2007 (Figs. 1 and 2).

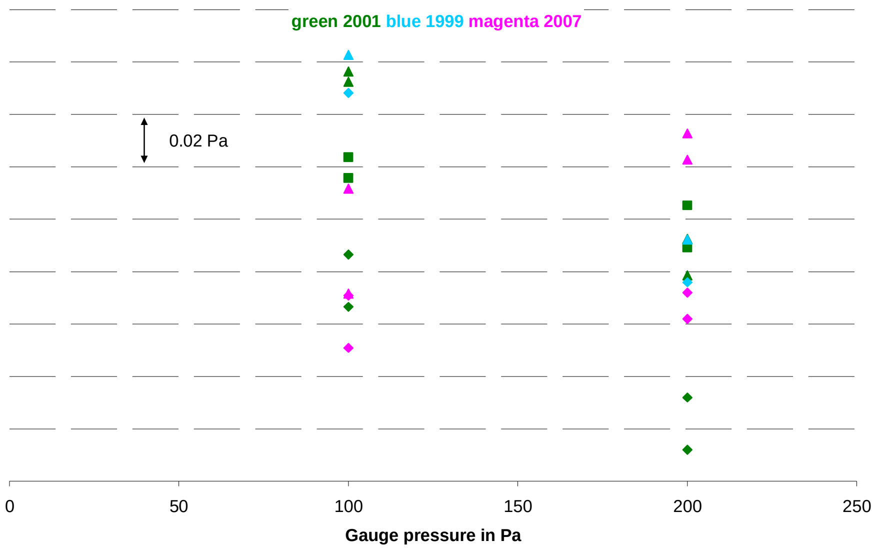

Figure 1. Deviations from reference pressure, pistons "1 mbar" and "2 mbar"

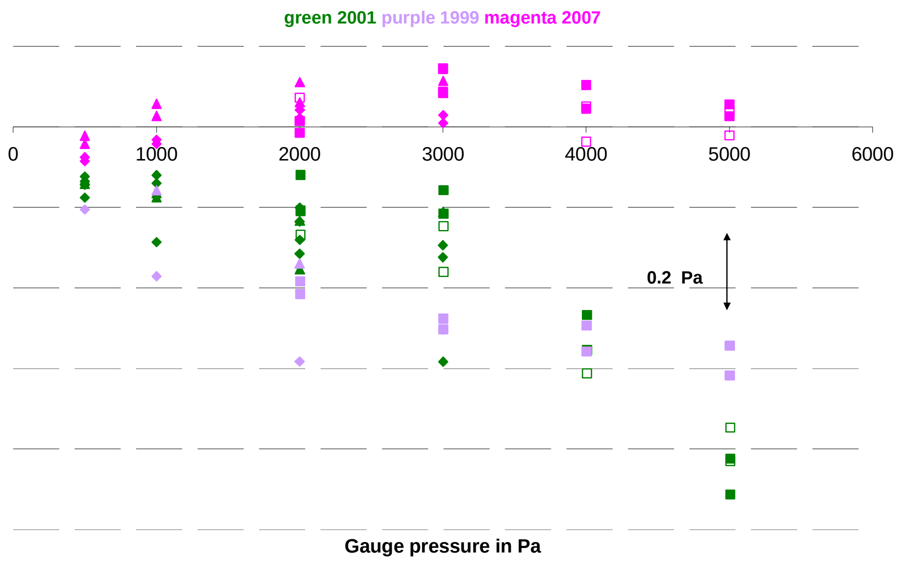

Figure 2. Deviations from reference pressure, piston "L"

As a result, no systematic changes could be seen at pressures up to 200 Pa. At the maximum pressure of 5 kPa, the differences did not exceed 1 Pa.
During the comparison, the TS stability was measured by the pilot laboratory at the beginning, in the middle, and at the end of the comparison. The results for the one year period of the actual KC are presented in Figs. 3 and 4. During the time of the KC, the pilot laboratory performed four measurements in March 2010, November 2009, March 2009 and November 2008, see table 8, although the measurements of November 2009 included only one measurement at each pressure.

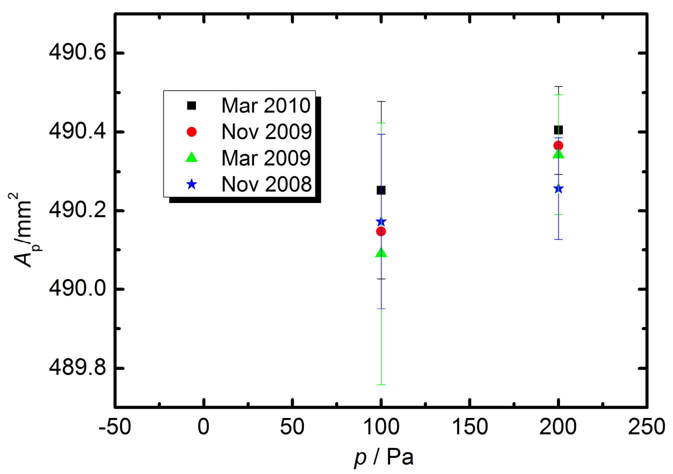

Figure 3. Stability of the transfer standard. Mean effective areas and their standard uncertainties for pistons "1 mbar" and "2 mbar" measured by PTB in 2008, 2009 and 2010

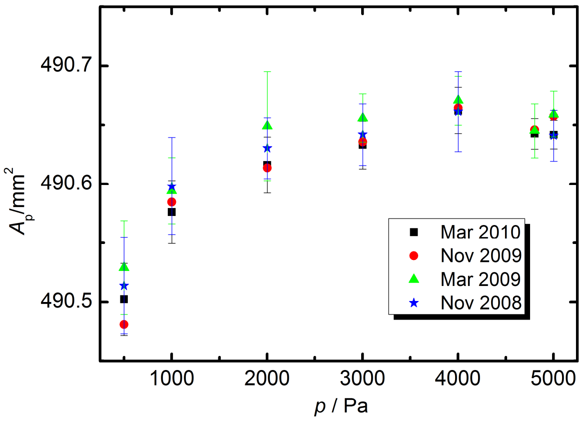

Figure 4. Stability of the transfer standard. Mean effective areas and their standard uncertainties for piston "L" measured by PTB in 2008, 2009 and 2010

From an analysis of these results, see Appendix C, it could be concluded that the TS did not underlay any systematic time drift during this KC. Therefore, no time dependent correction was applied to the participants' results, and the instability of TS, $u_{\text{instab}}(A_p)$, was expressed by the standard deviation of the mean values of the effective area measured by the pilot lab for each nominal pressure, $A_{p,\text{pilot, mean}}$:

$$A_{p,\text{ pilot, mean}} = \sum_{i=1}^{N} A_{p,\text{ pilot, }i} \Big/ N \,, \tag{2}$$

$$u_{\text{instab}}\left(A_p\right) = \left[\sum_{i=1}^{N}\left(A_{p,\text{ pilot, }i} - A_{p,\text{ pilot, mean}}\right)^2 \Big/ \left(N-1\right)\right]^{0.5} , \tag{3}$$

where N is number of pilot laboratory measurements over the time of the comparison, N = 4. The uncertainty of the TS determined in this way is presented in Table 12.

Table 12. Uncertainty contribution due to instability of TS, $u_{\text{instab}}(A_p)$

Nominal pressure in Pa	$u_{\text{instab}}(A_p)$ / mm^2	$u_{\text{instab}}(A_p)/A_p \times 10^6$
100	0.067	137
200	0.063	128
500	0.020	41
1000	0.010	20
2000	0.016	33
3000	0.010	21
4000	0.0042	9
4800	0.0018	4
5000	0.0098	20

Figures 5 and 6 show relative deviations of the effective areas measured by the pilot four times from their mean values. It is obvious that no systematic time drift occurred. Hence, the participants' values of the effective areas did not have to be corrected for the comparison.

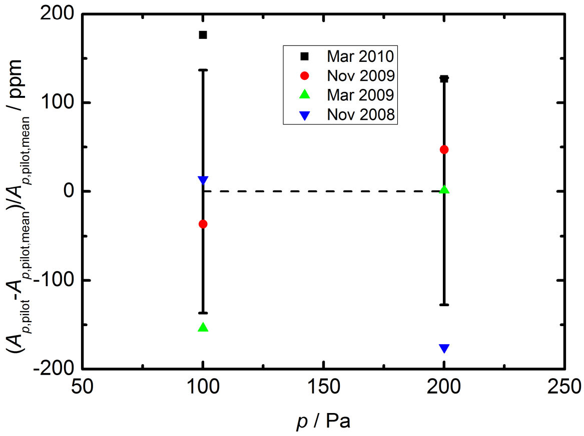

Figure 5. Instability of TS: Relative deviations of the single effective areas from the mean ones of pistons "1 mbar" and "2 mbar" measured by the pilot laboratory together with the relative standard deviations of the mean values (uncertainty bars) extracted from Table 12

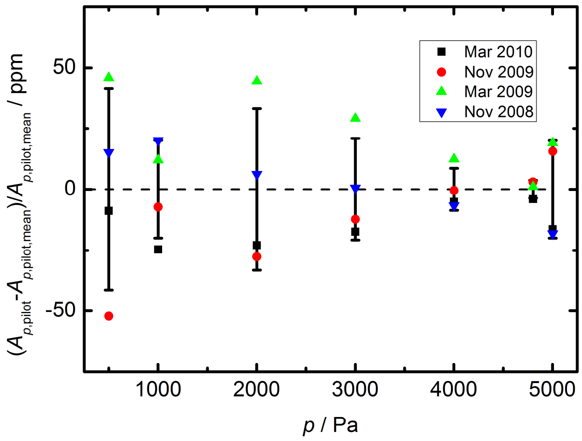

Figure 6. Instability of TS: Relative deviations of the single effective areas from the mean ones of piston "L" measured by the pilot laboratory together with the relative standard deviations of the mean values (uncertainty bars) extracted from Table 12

6.2 Results of the Participants

The mean effective areas, the standard deviations of the effective areas at each pressure and the combined relative standard uncertainties of the effective areas reported by the participants are presented in Table 13. The graphical visualization of the summary of all measurement cycles and participants is given in Figures 7 and 8.

Table 13. Mean effective areas ($\langle A_p \rangle$), their relative standard deviations ($s(A_p)/A_p$), relative standard uncertainties of pressures ($u(p)/p$), standard uncertainty of the temperature of TS ($u(t)$) and combined standard uncertainties of the mean effective areas ($u(A_p)/A_p$)

	PTB					VNIIM					CMI					VMC				
Nominal pressure in Pa	$\langle A_p \rangle$ / mm^2	$s(A_p)/A_p \times 10^6$	$u(p)/p \times 10^6$	$u(t)$ / °C	$u(A_p)/A_p \times 10^6$	$\langle A_p \rangle$ / mm^2	$s(A_p)/A_p \times 10^6$	$u(p)/p \times 10^6$	$u(t)$ / °C	$u(A_p)/A_p \times 10^6$	$\langle A_p \rangle$ / mm^2	$s(A_p)/A_p \times 10^6$	$u(p)/p \times 10^6$	$u(t)$ / °C	$u(A_p)/A_p \times 10^6$	$\langle A_p \rangle$ / mm^2	$s(A_p)/A_p \times 10^6$	$u(p)/p \times 10^6$	$u(t)$ / °C	$u(A_p)/A_p \times 10^6$
100	490.252	459	234	0.2	525	490.372	1594	350	0.1	1622	490.344	69	90	0.1	151	490.278	306	628	0.29	934
200	490.404	224	120	0.2	259	490.341	796	270	0.1	841	490.451	67	50	0.1	98	490.421	103	304	0.29	439
500	490.502	53	56	0.2	98	490.555	123	190	0.1	227	490.549	20	27	0.1	69	490.626	77	126	0.29	192
1000	490.576	44	39	0.2	71	490.601	84	130	0.2	155	490.596	10	20	0.1	46	490.615	52	98	0.29	156
2000	490.616	36	34	0.2	55	490.636	75	79	0.1	109	490.629	18	17	0.1	35	490.634	58	99	0.29	157
3000	490.633	27	32	0.2	45	490.656	69	46	0.1	83	490.646	18	16	0.1	29	490.638	59	88	0.29	146
4000	490.662	25	32	0.2	43	490.662	76	39	0.1	86	490.653	9	15	0.1	24	490.670	78	57	0.29	115
4800	490.642	15	21	0.2	28	490.672	72	37	0.1	81										
5000	490.642	15	20	0.2	27						490.667	5	15	0.1	20	490.685	32	38	0.29	96

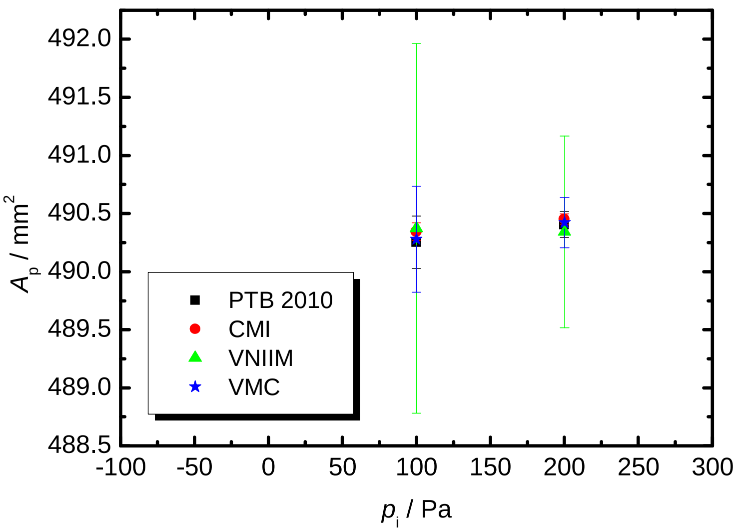

Figure 7. Mean effective areas and their uncertainties of TS with pistons "1 mbar" and "2 mbar" determined by the participants

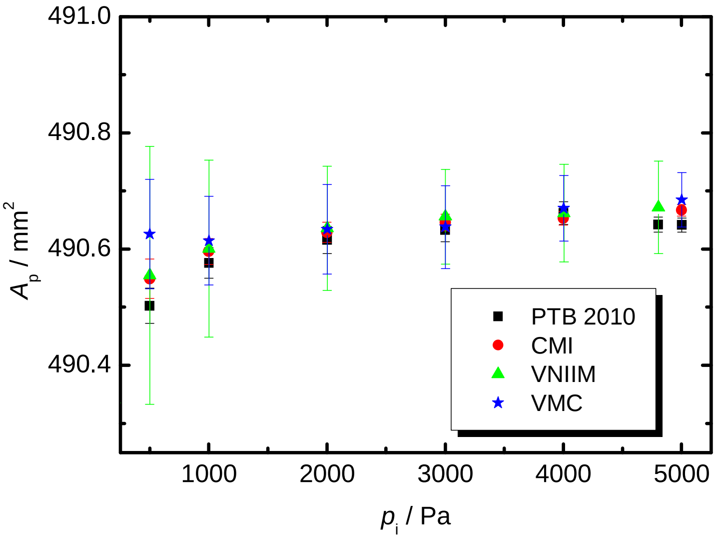

Figure 8. Mean effective areas and their uncertainties of TS with piston "L" determined by the participants

6.3 Reference value calculation

The key comparison reference value (KCRV), $A_{p,\text{ref}}$, was calculated at each pressure point of the KC as a weighted mean, see eq. (4), where N is the number of the independent participants, PTB, CMI and VNIIM, $N = 3$, $A_{p,i}$ and $u(A_{p,i})/A_{p,i}$ are the mean effective area and its uncertainty, respectively, of participant i at pressure p. This method is recommended in [6] and has been used for evaluation of numerous KCs. The uncertainty of $A_{p,\text{ref}}$, $u(A_{p,\text{ref}})$, was calculated by equation (5). The weighted mean method is applicable only when the results of the participants succeed the consistency check based on the chi-squared test. Results are considered as consistent if the observed chi-squared value χ^2_{obs} calculated by (6) is smaller than the value of the chi-square distribution calculated for degree of freedom $\nu = N - 1$ at probability Pr = 0.05, $\chi^2(\nu, \text{Pr})$.

$$A_{p,\text{ref}} = \sum_{i=1}^{N} \frac{A_{p,i}}{u^2(A_{p,i})} \Bigg/ \sum_{i=1}^{N} \frac{1}{u^2(A_{p,i})} \quad (4)$$

$$u(A_{p,\text{ref}}) = \left[\sum_{i=1}^{N} \frac{1}{u^2(A_{p,i})} \right]^{-0.5} \quad (5)$$

$$\chi^2_{\text{obs}} = \sum_{i=1}^{N} \frac{(A_{p,i} - A_{p,\text{ref}})^2}{u^2(A_{p,i})} \quad (6)$$

Note, that although four NMIs participated in this KC, VMC was excluded from the evaluation of KCRV since it is traceable to PTB (calibration certificate Nr. 0050 PTB 2009, see section 2.4) and, hence, is statistically not independent. Taking into account the instability of TS as defined by equation (3), the combined standard uncertainty of KCRV, $u^*(A_{p,\text{ref}})$, is defined as

$$u^*(A_{p,\text{ref}}) = [u^2(A_{p,\text{ref}}) + u^2_{\text{instab}}(A_p)]^{0.5}. \quad (7)$$

Results of the KCRV evaluation are summarised in Table 14.

Table 14. Key comparison reference values ($A_{p,\text{ref}}$), their relative standard uncertainties ($u(A_{p,\text{ref}})/A_{p,\text{ref}}$) and uncertainties combined with the TS instability ($u^*(A_{p,\text{ref}})/A_{p,\text{ref}}$) as well as parameters of the chi-squared test χ^2_{obs} and $\chi^2(\nu, 0.05)$

Nominal pressure in Pa	$A_{p,\text{ref}}$ / mm^2	$u(A_{p,\text{ref}})$ / $A_{p,\text{ref}} \times 10^6$	$u^*(A_{p,\text{ref}})$ / $A_{p,\text{ref}} \times 10^6$	χ^2_{obs}	$\chi^2(\nu, 0.05)$, $\nu = N$-1
100	490.337	145	199	0.12	5.99
200	490.444	91	157	0.18	5.99
500	490.535	55	68	0.67	5.99
1000	490.591	37	43	0.25	5.99
2000	490.626	29	43	0.20	5.99
3000	490.643	23	31	0.35	5.99
4000	490.656	20	22	0.16	5.99
5000	490.659	16	25	2.42	5.99

[1] This value of $A_{p,\mathrm{ref}}$ was obtained by combining the PTB and CMI results obtained at 5 kPa with the result of VNIIM obtained at 4.8 kPa. Such a combination is justified by the measurements of the pilot laboratory performed at pressures of 4.8 and 5 kPa which showed no difference in the effective area.

The consistency check supports the choice of the KCRV calculation as a weighted mean.

6.4 Degree of equivalence

The degrees of equivalence of the participants in relation to KCRV considered at each pressure are expressed in terms of relative deviations of the participants' results from KCRV ($\Delta A_{p,i} / A_{p,\mathrm{ref}}$) and relative expanded uncertainties of these deviations ($U(\Delta A_{p,i}) / A_{p,\mathrm{ref}}$), the latter being calculated as:

$$U\left(\Delta A_{p,i}\right)/A_{p,\mathrm{ref}} = 2 \cdot \left[u^2\left(A_{p,i}\right) - u^2\left(A_{p,\mathrm{ref}}\right) + u^2_{\mathrm{instab}}\left(A_p\right)\right]^{0.5} / A_{p,\mathrm{ref}} \ . \tag{8}$$

Numerical data for the deviations and the uncertainties at all pressures are listed in Table 15, a graphical presentation is given in Appendix A.

Table 15. Relative deviations of the participants' results from the reference values ($\Delta A_p/A_p$) and their expanded uncertainties ($U(\Delta A_p/A_p)$)

Nominal pressure (Pa)	PTB		VNIIM		CMI		VMC	
	$\Delta A_p/A_p \times 10^6$	$U(\Delta A_p/A_p) \times 10^6$	$\Delta A_p/A_p \times 10^6$	$U(\Delta A_p/A_p) \times 10^6$	$\Delta A_p/A_p \times 10^6$	$U(\Delta A_p/A_p) \times 10^6$	$\Delta A_p/A_p \times 10^6$	$U(\Delta A_p/A_p) \times 10^6$
100	-174	1046	71	3270	14	287	-121	1864
200	-81	549	-210	1690	14	267	-46	897
500	-67	182	41	447	29	117	186	376
1000	-30	127	21	304	11	67	49	305
2000	-20	114	20	220	6	77	17	316
3000	-21	87	26	164	6	53	-11	290
4000	13	78	13	167	-5	31	30	227
5000	-34	59	27	164	17	47	54	193

The degrees of equivalence between the laboratories are expressed by relative differences between them (d_{ij}) and relative expanded uncertainties of these differences ($U(d_{ij})$) calculated as:

$$d_{ij} = \left(A_{p,i} - A_{p,j}\right)/A_{p,\mathrm{ref}} \ , \tag{9}$$

$$U\left(d_{ij}\right) = 2\left[u^2\left(A_{p,i}\right) + u^2\left(A_{p,j}\right) + u^2_{\mathrm{instab}}\left(A_p\right)\right]^{1/2} / A_{p,\mathrm{ref}} \ , \tag{10}$$

which are presented in the tables in Appendix B.

7. DISCUSSION

From Table 13, the appropriate performance of TS can be deduced. At the highest pressure of 5 kPa, the typical relative standard deviations of A_p of the participants range from 5 ppm to 80 ppm. A comparison of $s(A_p)$ with $u(A_p)$ indicates that for most NMIs in this KC a major contribution to the uncertainty of A_p was from the uncertainty of the laboratory standards. Comparison of typical $s(A_p)/A_p$ values from laboratory to laboratory clearly show different performance of pressure measurements of the participating NMIs. Among the laboratories, all values of the reported A_p agree within their expanded uncertainties ($k = 2$). All NMIs agree with the reference value of KC within their expanded uncertainties ($k = 2$), (Table 15 and Appendix A). The most results agree with the reference value of the KC even within their standard uncertainties ($k = 1$) except for PTB at 5 kPa. A comparison in pairs demonstrates that the results of all participants agree with each other within their expanded uncertainties ($k = 2$) at all pressures (Appendix B). Within 48 compared pairs of results, in no case there is a disagreement on the level of the expanded uncertainties ($k = 2$). Only in two cases there are differences between the laboratories which are bigger than the standard uncertainties of these differences.

8. CONCLUSIONS

The transfer standard was stable within 0.025 Pa + $2 \cdot 10^{-5} p$ in terms of pressure in the period of the KC. For all laboratories all the results are equivalent with the key comparison reference values within the expanded uncertainties ($k = 2$), all but one results even within the standard uncertainties. For the NMIs' results compared in pairs, all of totally 48 pairs show agreement within the expanded ($k = 2$) and 46 pairs within the standard uncertainties.

With the results of this comparison PTB, VNIIM, CMI and VMC have supported their measurement capabilities stated in the KCDB of BIPM.

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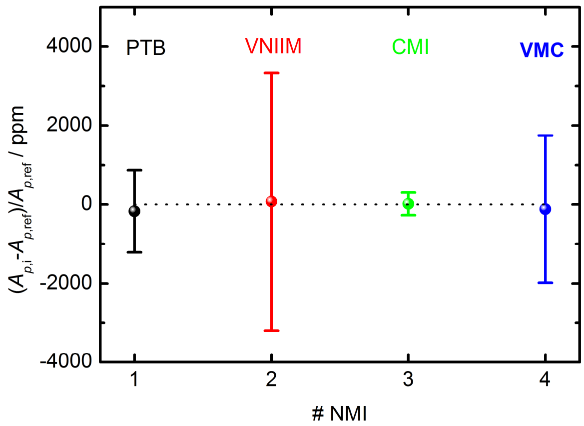
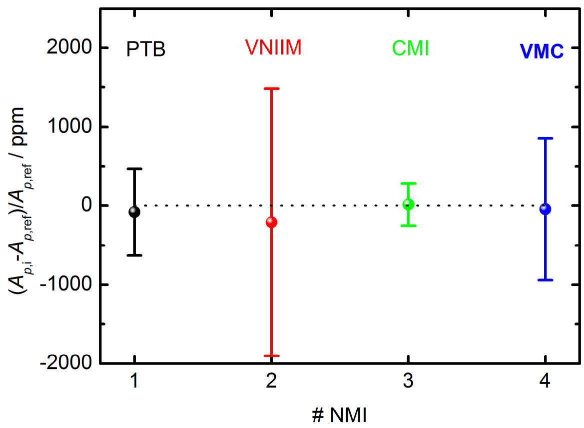
APPENDIX A. Relative deviations of the participants results' from the reference value $((A_{p,i} - A_{p,\mathrm{ref}})/A_{p,\mathrm{ref}})$ with the expanded uncertainties (k = 2) of these deviations at pressures 100 Pa to 5 kPa

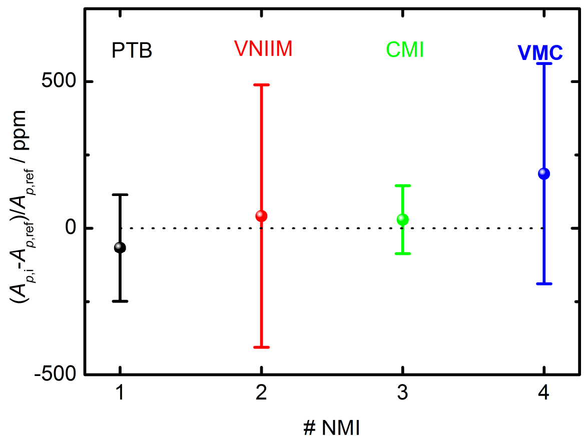
100 Pa:



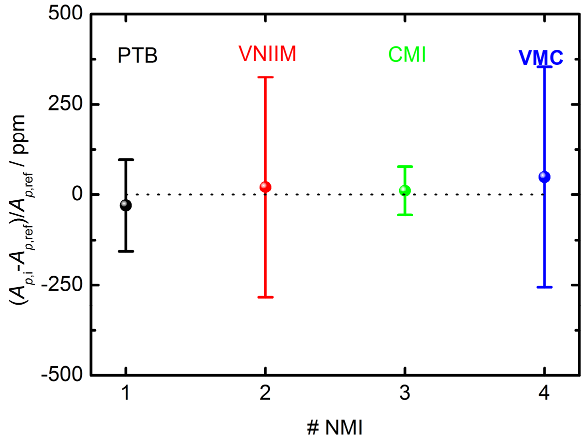
200 Pa:



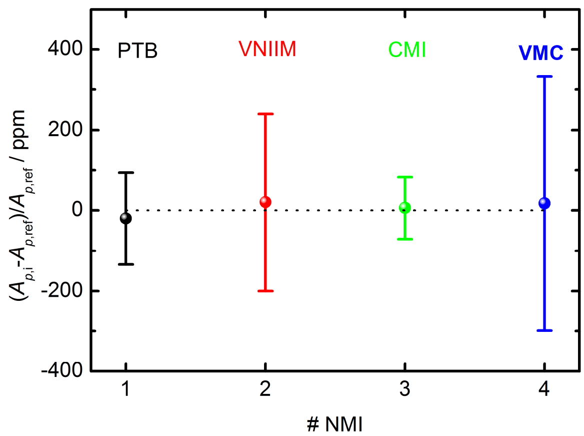
500 Pa:



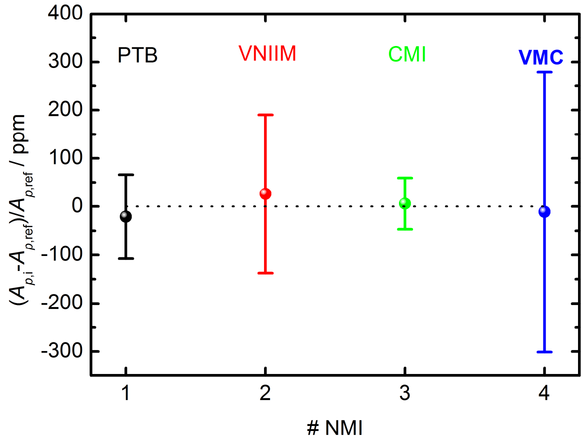
1000 Pa:



2000 Pa:



3000 Pa:



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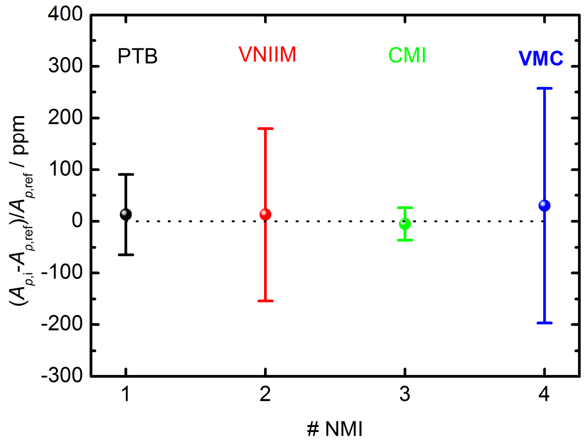
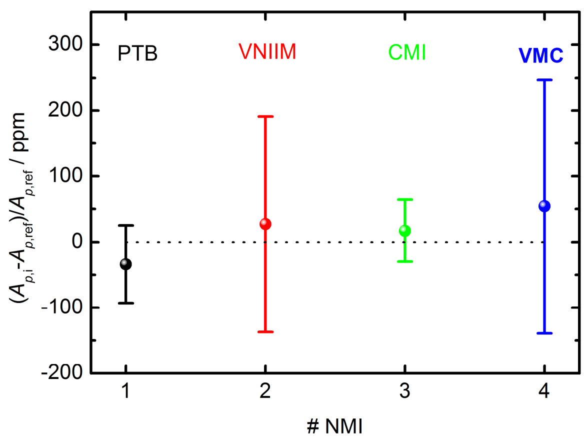


5000 Pa:



APPENDIX B. Relative differences between the participants' results (d_{ij}) and their expanded uncertainties ($U(d_{ij})$) (k = 2) at pressures 100 Pa to 5 kPa

j

	p = 100 Pa	CMI		VNIIM		PTB		VMC	
		$d_{ij} \times 10^6$	$U(d_{ij}) \times 10^6$	$d_{ij} \times 10^6$	$U(d_{ij}) \times 10^6$	$d_{ij} \times 10^6$	$U(d_{ij}) \times 10^6$	$d_{ij} \times 10^6$	$U(d_{ij}) \times 10^6$
i	CMI			245	6573	188	1092	53	2142
	VNIIM	-245	6573			-57	6495	-192	6752
	PTB	-188	1092	57	6495			-135	1891
	VMC	-53	2142	192	6752	135	1891		

j

	p = 200 Pa	CMI		VNIIM		PTB		VMC	
		$d_{ij} \times 10^6$	$U(d_{ij}) \times 10^6$	$d_{ij} \times 10^6$	$U(d_{ij}) \times 10^6$	$d_{ij} \times 10^6$	$U(d_{ij}) \times 10^6$	$d_{ij} \times 10^6$	$U(d_{ij}) \times 10^6$
i	CMI			-224	1711	-96	610	-61	936
	VNIIM	224	1711			128	1777	164	1914
	PTB	96	610	-128	1777			35	1051
	VMC	61	936	-164	1914	-35	1051		

j

	p = 500 Pa	CMI		VNIIM		PTB		VMC	
		$d_{ij} \times 10^6$	$U(d_{ij}) \times 10^6$	$d_{ij} \times 10^6$	$U(d_{ij}) \times 10^6$	$d_{ij} \times 10^6$	$U(d_{ij}) \times 10^6$	$d_{ij} \times 10^6$	$U(d_{ij}) \times 10^6$
i	CMI			12	481	-96	253	157	415
	VNIIM	-12	481			-108	500	145	599
	PTB	96	253	108	500			253	438
	VMC	-157	415	-145	599	-253	438		

j

	p = 1 kPa	CMI		VNIIM		PTB		VMC	
		$d_{ij} \times 10^6$	$U(d_{ij}) \times 10^6$	$d_{ij} \times 10^6$	$U(d_{ij}) \times 10^6$	$d_{ij} \times 10^6$	$U(d_{ij}) \times 10^6$	$d_{ij} \times 10^6$	$U(d_{ij}) \times 10^6$
i	CMI			10	326	-41	174	38	327
	VNIIM	-10	326			-51	343	28	441
	PTB	41	174	51	343			79	345
	VMC	-38	327	-28	441	-79	345		

j

	p = 2 kPa	CMI		VNIIM		PTB		VMC	
		$d_{ij} \times 10^6$	$U(d_{ij}) \times 10^6$	$d_{ij} \times 10^6$	$U(d_{ij}) \times 10^6$	$d_{ij} \times 10^6$	$U(d_{ij}) \times 10^6$	$d_{ij} \times 10^6$	$U(d_{ij}) \times 10^6$
i	CMI			14	238	-26	146	11	329
	VNIIM	-14	238			-41	253	-3	388
	PTB	26	146	41	253			37	340
	VMC	-11	329	3	388	-37	340		

j

	p =3 kPa	CMI		VNIIM		PTB		VMC	
		$d_{ij} \times 10^6$	$U(d_{ij}) \times 10^6$	$d_{ij} \times 10^6$	$U(d_{ij}) \times 10^6$	$d_{ij} \times 10^6$	$U(d_{ij}) \times 10^6$	$d_{ij} \times 10^6$	$U(d_{ij}) \times 10^6$
i	CMI			20	181	-26	115	-16	300
	VNIIM	-20	181			-47	193	-37	338
	PTB	26	115	47	193			10	307
	VMC	16	300	37	338	-10	307		

j

	p = 4 kPa	CMI		VNIIM		PTB		VMC	
		$d_{ij} \times 10^6$	$U(d_{ij}) \times 10^6$	$d_{ij} \times 10^6$	$U(d_{ij}) \times 10^6$	$d_{ij} \times 10^6$	$U(d_{ij}) \times 10^6$	$d_{ij} \times 10^6$	$U(d_{ij}) \times 10^6$
i	CMI			18	178	18	100	35	236
	VNIIM	-18	178			0	192	17	287
	PTB	-18	100	0	192			17	246
	VMC	-35	236	-17	287	-17	246		

j

	p = 5 kPa	CMI		VNIIM		PTB		VMC	
		$d_{ij} \times 10^6$	$U(d_{ij}) \times 10^6$	$d_{ij} \times 10^6$	$U(d_{ij}) \times 10^6$	$d_{ij} \times 10^6$	$U(d_{ij}) \times 10^6$	$d_{ij} \times 10^6$	$U(d_{ij}) \times 10^6$
i	CMI		47	10	172	-51	78	37	200
	VNIIM	-10	172			-61	175	26	254
	PTB	51	78	61	175			88	203
	VMC	-37	200	-26	254	-88	203		

APPENDIX C. Stability of TS

The following discussion focuses – by chance – on the nominal pressure point of 3000 Pa as a typical data point, compare to the whole dataset of TS measurements at the pilot laboratory in Fig. 12. It could be shown that a linear fit without slope containing all measurements without weighing procedure yielded a satisfying result indicating the stability of TS. Performing a weighted linear fit $y = ax + b$ the following values for a and b were obtained $b = 90.636 \pm 0.026\ \mathrm{mm}^2$ and $a = (2.36 \pm 3.57)\cdot 10^{-5}\ \mathrm{mm}^2/\mathrm{day}$, see Figure C1.

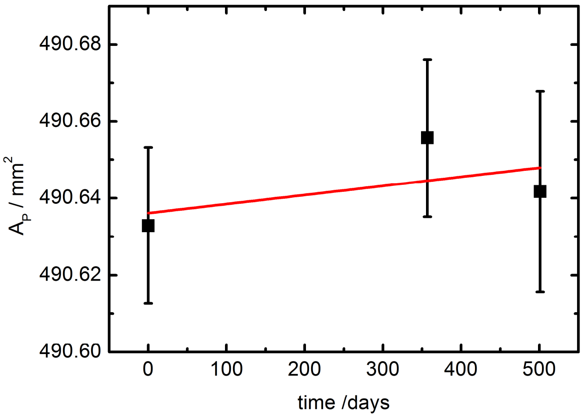


Figure C1. Weighted linear fit of the mean effective areas with corresponding combined uncertainties at p = 3000 Pa measured in March 2010 (0 days), March 2009 and November 2008, assuming a time drift.

Due to the uncertainties of the fit, a constant – zero – slope was deduced as a consequence, i.e. it was assumed that basically the TS had no systematic drift during the time of the comparison.

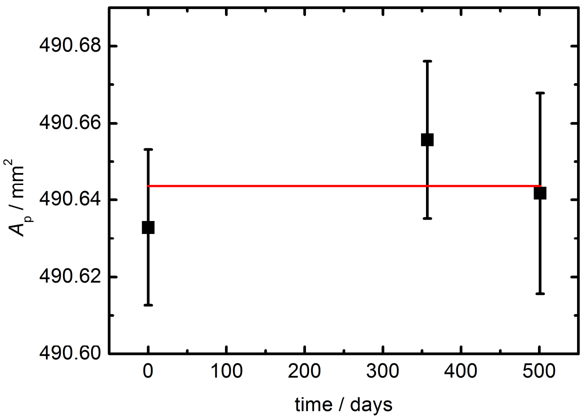


Figure C2. Weighted linear fit of the mean effective areas with corresponding combined uncertainties at p = 3000 Pa measured in March 2010 (0 days), March 2009 and November 2008, assuming no time drift.

On the other hand, the TS is associated with a time-constant uncertainty to be evaluated. Performing a weighted linear fit $y = ax + b$ assuming $a = 0$ we obtained then $b = 490.643 \pm 0.014$ mm^2 (Figure C2). This analysis was compared with a non-weighted linear fit with zero-slope, see Figure C3.

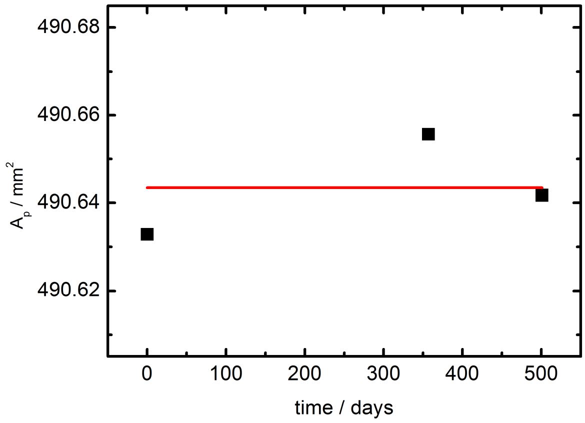


Figure C3. Non-weighted linear fit of the mean effective areas with corresponding combined uncertainties at *p* = 3000 Pa measured in March 2010 (0 days), March 2009 and November 2008, assuming no time drift.

We gained $b = 490.643 \pm 0.013$ mm^2. Finally, we analysed all data points available, i.e. we considered the single pressure-value measurements in November 2009 for a non-weighted linear fit with zero-slope, see Figure C4.

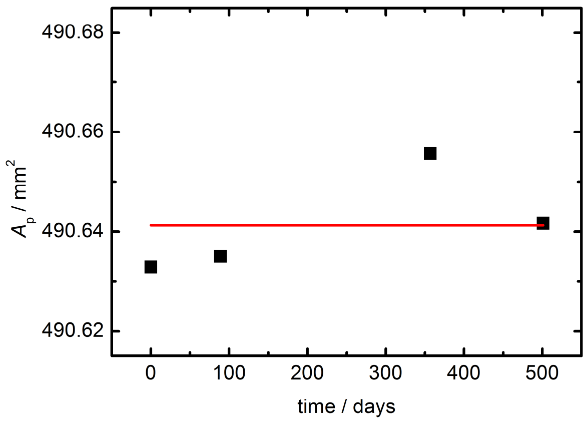


Figure C4. Non-weighted linear fit of the mean effective areas with corresponding combined uncertainties at *p* = 3000 Pa measured in March 2010 (0 days), November 2009, March 2009 and November 2008, assuming no time drift.

The result is then $b = 490.641 \pm 0.010$ mm^2. Hence, we realized that the different analyses with zero-slope basically yielded similar results. Consequently, we performed non-weighted linear fits with the complete data set to estimate the uncertainty of TS due to its instability.