Final Report on Comparison EURAMET.M.P-S6 of PTB and INTI Pressure Standards for the Range 1.5 kPa to 300 kPa of Absolute Gas Pressure

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August, 2008

ABSTRACT

This report presents results of a bilateral comparison between PTB and INTI in the range 1.5 kPa to 300 kPa of absolute gas pressure. The comparison was carried out in April-May 2008. The pilot laboratory was PTB. The pressure standards of the participants were pressure balances of the same type. The transfer standard was an electronic pressure gauge. Deviations of the transfer standard's pressures from the reference pressures generated by the laboratories' standards at specified pressures were reported by the participants and used for calculating degrees of equivalence between the laboratories. The results of the comparison presented in the Draft A report have induced INTI to revise their uncertainty by an increase in the uncertainty of the residual pressure. After this revision, the results presented in the actual report demonstrate equivalence within the expended uncertainties (k=2) reported by the laboratories at all pressures of the comparison. The degrees of equivalence stated in this comparison will provide a supporting basis for a new pressure CMC table of INTI.

CONTENTS

1. INTRODUCTION

This bilateral comparison was proposed by INTI to support their new pressure CMC table planed for a submission to the BIPM KCDB. PTB, which has well-established measurement capabilities in the actual pressure range supported by several former key comparisons, e.g. CCM.P-K2 [1], was chosen by INTI as a partner in this comparison because of a scheduled peer-review visit of a PTB specialist to INTI, which simplified the transportation of the transfer standard to and from INTI. A pressure measuring instrument suitable as a transfer standard was available at PTB.
PTB was agreed to be a pilot laboratory in this comparison.

2. COMPARISON SCHEDULE

The comparison schedule was as follows:

1 – 4 April 2008: initial investigation of TS at PTB,
9 – 11 April 2008: measurements at INTI
5 – 9 May 2008: final investigation of TS at PTB.

The transfer standard (TS) was transported to and from INTI by hand.

3. LABORATORY STANDARDS AND MEASUREMENT METHODS OF THE PARTICIPANTS

Both laboratories used pressure balances as laboratory standards (LS).

3.1 PTB pressure balance

PTB used a Ruska pressure-balance Model 2465 with two different piston-cylinder assemblies. It can be used for measurement of gauge and absolute pressures (p_e and p_{abs}). The properties of these pressure standards and ambient conditions are given in Table 1.

Table 1. PTB standards and measurement conditions

Manufacturer	Ruska	
Identification number of piston-cylinder assembly	TL-1568	C-362
Measurement range in kPa	1.4 to 190, p_e and p_{abs}	10 to 700, p_e and p_{abs}
Material of piston	steel	tungsten carbide
Material of cylinder	tungsten carbide	tungsten carbide
Zero-pressure effective area (A_0) at reference temperature in cm^2	3.3566056	0.8408019
Relative standard uncertainty of A_0	$6\cdot10^{-6}$	$6\cdot10^{-6}$
Relative standard uncertainty of mass pieces	$3\cdot10^{-6}$	$3\cdot10^{-6}$
Linear thermal expansion coefficient of piston-cylinder assembly(α_p+α_c) in °C^{-1}	$1.5\cdot10^{-5}$	$9.1\cdot10^{-6}$
Reference temperature (t_0) in °C	20	
Local gravity (g) in m/s^2	9.812533	
Relative standard uncertainty of g	$5\cdot10^{-7}$	
Height difference between LS and TS (h) in mm	10	
Residual pressure in bell jar and its standard uncert., Pa	(1.5 to 2) ± 0.5	

This pressure balance equipped with the two piston-cylinder assemblies is a secondary pressure standard.
The effective areas of both piston-cylinder assemblies are traceable to the primary mercury manometer [2] and primary pressure balances characterised by dimensional measurements [3].
The primary mercury manometer participated in key comparisons (KC) CCM.P-K2, CCM.P-K6 and APMP.M.P-K6, the primary pressure balances took part in comparisons CCM.P-K1.b, CCM.P-K1.c and several RMOs KCs.
Piston-cylinder assembly TL-1568 was used in the pressure range (1.4 to 150) kPa, piston-cylinder assembly C-362 was applied at pressures between (180 and 300) kPa.
Nitrogen was used as a pressure-transmitting medium.

3.2 INTI pressure balance

INTI used a Ruska pressure-balance Model 2465 with two different piston-cylinder assemblies. It can be used for measurement of gauge and absolute pressures (p_e and p_{abs}). The properties of these pressure standards and ambient conditions are given in Table 2

Table 2. INTI standards and measurement conditions

Manufacturer	Ruska	
Identification number of piston-cylinder assembly	TL-1476	C-573
Measurement range in kPa	1.5 to 190, p_e and p_{abs}	12 to 700, p_e and p_{abs}
Material of piston	steel	tungsten carbide
Material of cylinder	tungsten carbide	tungsten carbide
Zero-pressure effective area (A_0) at reference temperature in cm^2	3.35715	0.839476
Relative standard uncertainty of A_0	$10\cdot10^{-6}$	$10\cdot10^{-6}$
Relative standard uncertainty of mass pieces	$5\cdot10^{-6}$	$5\cdot10^{-6}$
Linear thermal expansion coefficient of piston-cylinder assembly($\alpha_p+\alpha_c$) in °C^{-1}	$1.5\cdot10^{-5}$	$9.1\cdot10^{-6}$
Reference temperature (t_0) in °C	20	
Local gravity (g) in m/s^2	9.79688	
Relative standard uncertainty of g	$5\cdot10^{-6}$	
Height difference between LS and TS (h) in mm	70	
Residual pressure in bell jar and its standard uncert., Pa	(16 to 18) ± 1	

This pressure balance equipped with the two piston-cylinder assemblies is a secondary pressure standard.
The effective areas of both piston-cylinder assemblies are traceable to PTB Pressure Laboratory.
Piston-cylinder assembly TL-1476 was used in the pressure range (1.5 to 180) kPa, piston-cylinder assembly C-573 was applied at pressures between (210 and 300) kPa.
Nitrogen was used as a pressure-transmitting medium.

The measurements were performed by a direct connection of TS to LS.

4. TRANSFER STANDARD

The transfer standard was a Paroscientific digital absolute pressure gauge model 760 - 45A serial no. 65655 with a resolution of 0.1 Pa and the manufacturer-declared rms conformance better than 0.002% of full scale as well as a total error of less than 0.008%. This pressure standard is in useat PTB since 1998 and has been calibrated several times.
The results of the calibrations in 2003 and in 2008 are shown in Fig. 1 in form of zero-pressure corrected deviations of the pressure gauge from a reference standard. For measurements with increasing pressures, the deviations agree within about 5 Pa, the same is valid for measurements with decreasing pressures above 100 kPa. Herewith this pressure gauge appeared suitable for the purpose of this comparison. The stability of TS during the comparison was controlled by the PTB measurements at the beginning and the end of the comparison, and is discussed in section 6.1.

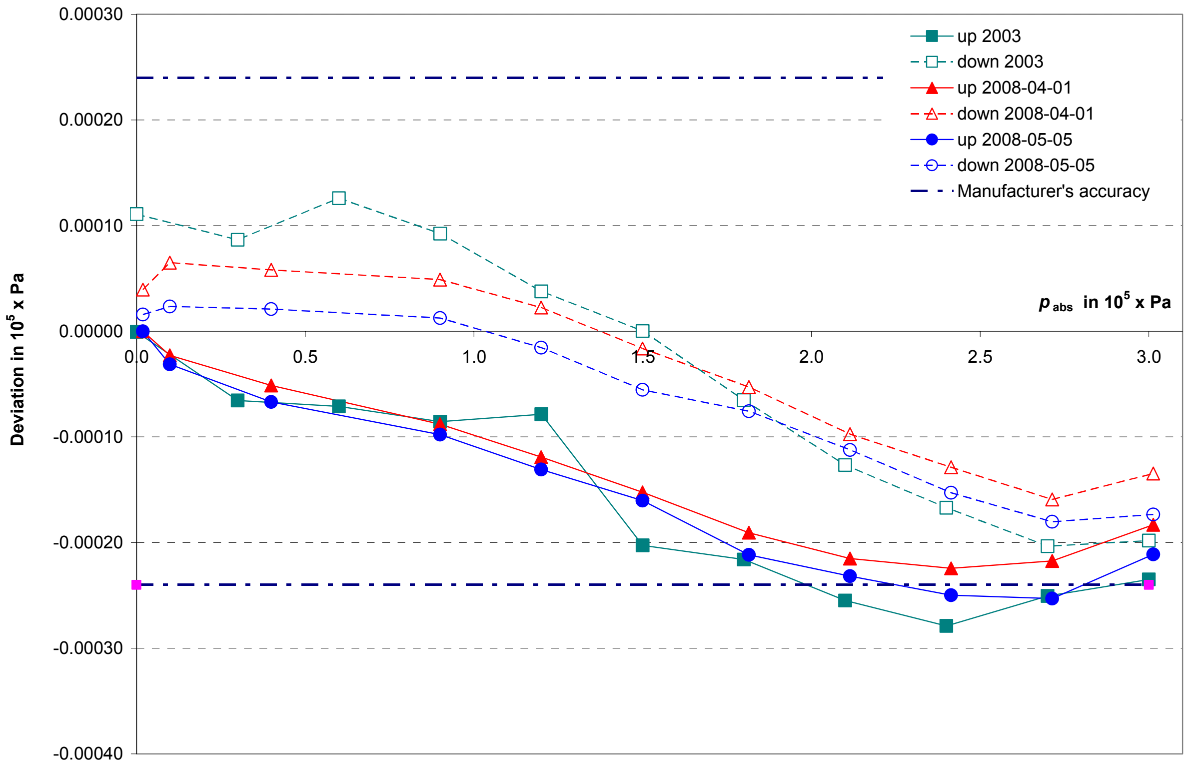

Fig. 1. Stability of the transfer standard. Measurements at PTB in 2003 and 2008

5. MEASUREMENT PROCEDURE

It was agreed to perform measurements at the following pressure points (1.88, 9.89, 39.9, 89.94, 119.92, 149.94, 181.47, 211.43, 241,39, 271.35, 301.31) kPa, with two complete measurement cycles each including measurement series at increasing and decreasing pressures. The measurements were carried out by a direct comparison with the laboratory standards. No adjustment of TS during the comparison was performed and no zero point correction was done when evaluating the results of the measurements.
For the measurements, the time between measurement points was specified to be always 4 min with exception of pressure points 149.94 Pa and 181.47 kPa, when the piston-cylinder unit of LS had to be changed. Between these pressure points, the time difference was 5 min. With this measurement program a complete measurement cycle was possible on one day.
Each laboratory had to report the nominal pressure and the pressure difference between the pressure measured with TS and the pressure generated by LS, for each measurement point. In addition, mean pressure differences between TS and LS had to be reported for separate upwards and downwards series, and uncertainty budgets had to be reported for these results. The uncertainty budget had to include uncertainty of the reference standard, of height difference between LS and TS, resolution of TS, repeatability and hysteresis.
The pressure generated by LS at the TS reference level was calculated by the well-known equation:

$$p_{\text{abs}} = \frac{\sum_i m_i g}{A_0\left[1+\left(\alpha_{\text{p}}+\alpha_{\text{c}}\right)\left(t-t_0\right)\right]} + p_{\text{res}} + \rho_{\text{N}_2} g h, \quad \text{where} \qquad (1)$$

m_i are true masses of the piston, the weight carrier and the mass pieces placed on the weight carrier of LS;
t is temperature of LS;
p_{res} is residual pressure measured in the bell jar of LS,

ρ_{N2} nitrogen density at pressure p_{abs},
and all other quantities as defined before.
In total, INTI performed 3 measurement cycles instead of agreed minimum of 2 cycles. PTB measured 2 cycles at the beginning and 2 cycles at the end of the comparison. Finally, for evaluation of the comparison results, all 3 INTI cycles and all 4 PTB cycles were taken. The use of all 4 PTB cycles was justified by the fact that no systematic difference had been observed between the PTB results at the beginning and the end of the comparison.

6. RESULTS

6.1 Stability of the transfer standard

On stability of TS during the comparison it can be concluded from the PTB results obtained at the beginning and the end of the comparison. The difference between the mean values is mostly smaller than 5 Pa.

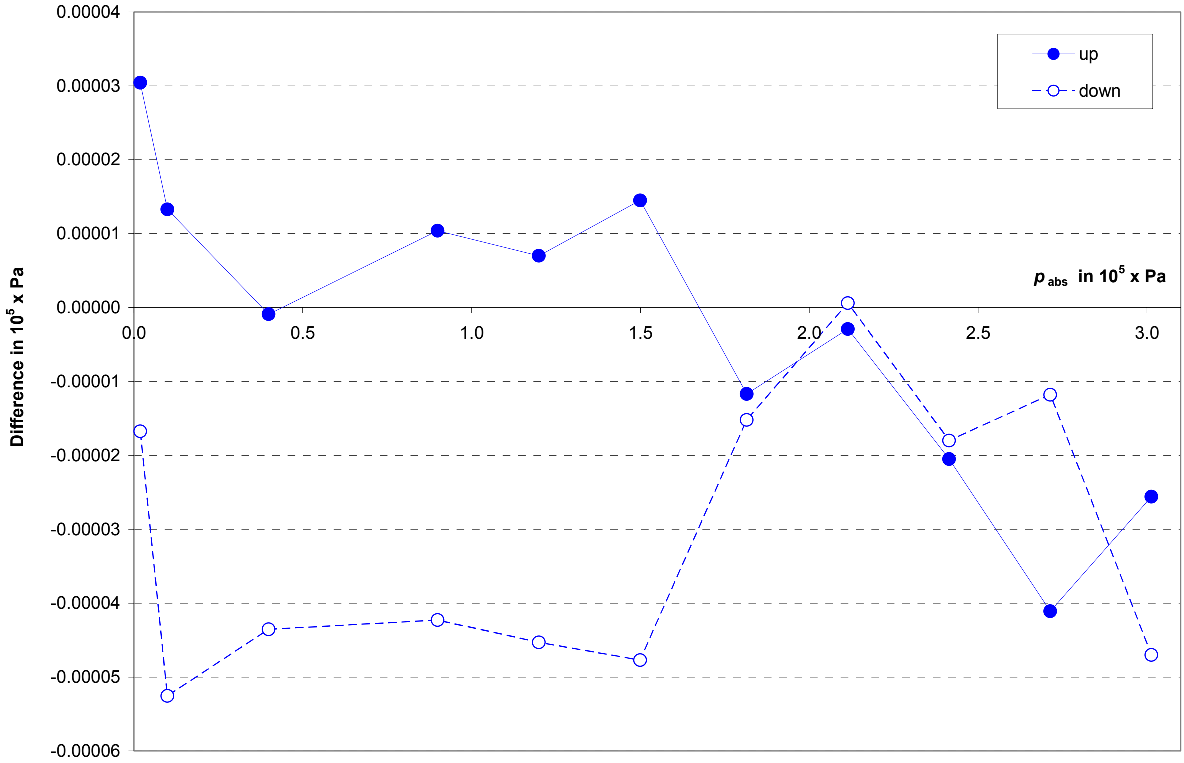

Fig. 2. Stability of the transfer standard. Measurements at PTB at the beginning and the end of the comparison

6.2 Results of the participants

Deviations of TS from the reference standards as measured in PTB and INTI at increasing and decreasing pressures are shown in Fig. 3. The difference between PTB and INTI is smaller than the hysteresis of TS. Therefore it appeared reasonable to compare the laboratories' results for separate "up" and "down" measurements.
The results of the comparison presented in the Draft A report have induced INTI to revise their uncertainty by an increase in the uncertainty of the residual pressure. This change has effected the uncertainty of the reference pressure in the range (2 to 120) kPa. The actual report presents the revised uncertainties in this pressure range. All other data and, in particular, the

values of the reference pressure, the indication of the transfer standard and the uncertainties at pressures above 120 kPa remained unchanged and identical with those in the Draft A report.

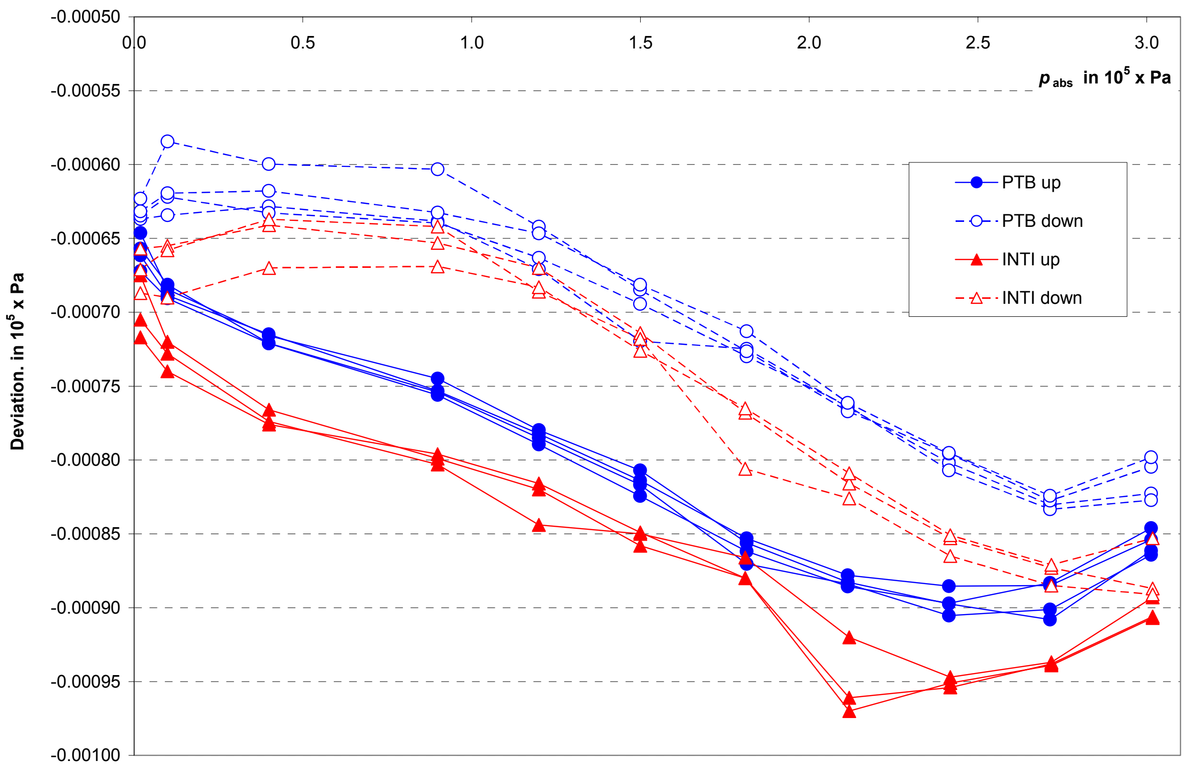

Fig. 3. Deviations of TS from PTB and INTI standards for all individual measurements

The average TS deviations and their standard uncertainties are reported in Table 3. These results are shown graphically in Fig. 4, where vertical bars present expanded (k=2) uncertainties. The pressures measured at INTI are systematically lower than the PTB pressures.

Table 3. Standard uncertainty of the reference pressure ($u(p_{ref})$), deviation of the TS pressure from the reference pressure (δp) and standard uncertainty of this deviation ($u(\delta p)$) for measurements at increasing (up) and decreasing (down) pressures at PTB and INTI

$p_{nom.}$ in kPa	PTB					INTI				
	$u(p_{ref})$ in Pa	up		down		$u(p_{ref})$ in Pa	up		down	
		δp in Pa	$u(\delta p)$ in Pa	δp in Pa	$u(\delta p)$ in Pa		δp in Pa	$u(\delta p)$ in Pa	δp in Pa	$u(\delta p)$ in Pa
2	0.6	-65.9	1.6	-63.2	1.5	1.4	-70.0	2.8	-67.6	2.0
10	0.6	-68.6	1.5	-61.5	2.0	1.6	-73.0	2.1	-67.1	2.2
40	0.6	-71.8	1.5	-62.0	1.7	1.8	-77.0	2.1	-65.2	1.9
90	0.8	-75.2	1.6	-62.8	1.8	1.8	-79.4	2.0	-66.0	2.1
120	0.9	-78.4	1.6	-65.6	1.8	2.1	-82.0	2.2	-69.0	2.5
150	1.0	-81.6	1.7	-69.5	2.0	2.5	-85.0	3.0	-73.0	3.0
181	1.2	-86.0	1.8	-72.3	1.8	3.0	-87.0	3.5	-78.0	3.5
211	1.3	-88.3	1.9	-76.4	1.9	3.0	-95.0	4.0	-82.0	4.0
241	1.5	-89.6	2.0	-80.0	2.0	3.5	-95.0	5.0	-85.0	5.0
271	1.6	-89.4	2.2	-82.9	2.1	3.8	-94.0	5.5	-88.0	5.5
301	1.8	-85.6	2.3	-81.3	2.4	4.1	-90.0	6.0	-87.0	6.0

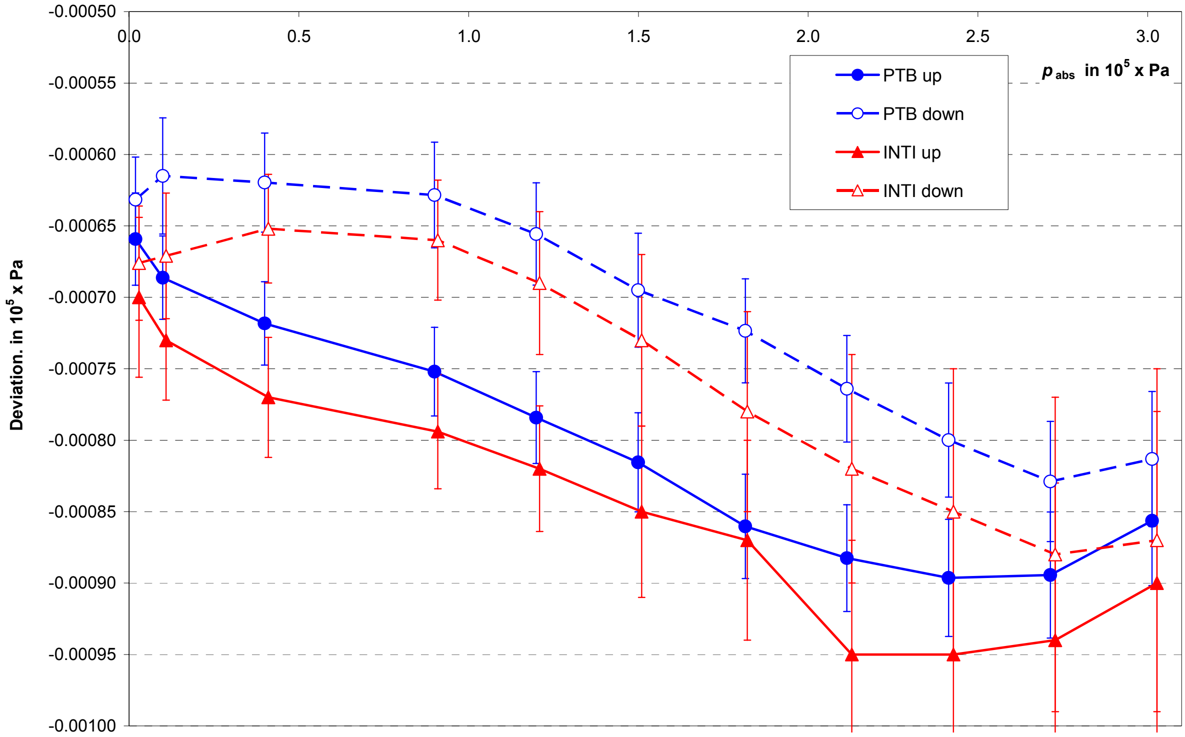

Fig. 4. Deviations of TS from reference pressures and expanded (k=2) uncertainties of these deviations for measurements with increasing and decreasing pressures

6.3 Degree of equivalence

Degrees of equivalence between the laboratories are expressed by the differences of their results, δp_{INTI} - δp_{PTB}, and the expanded uncertainties of these differences, $U(\delta p_{\mathrm{INTI}} - \delta p_{\mathrm{PTB}})$, calculated with

$$U(\delta p_{\mathrm{INTI}} - \delta p_{\mathrm{PTB}}) = \left[U^2(\delta p_{\mathrm{INTI}}) + U^2(\delta p_{\mathrm{PTB}})\right]^{0.5}, \quad \text{where} \tag{2}$$

$U(\delta p_{\mathrm{INTI}})$ and $U(\delta p_{\mathrm{PTB}})$ are expanded uncertainties reported by the institutes. Equation (2) assumes that the uncertainties of PTB and INTI are not correlated. In fact, due to traceability of INTI to PTB the results of the two laboratories are correlated to some extent so that uncertainties $U(\delta p_{\mathrm{INTI}} - \delta p_{\mathrm{PTB}})$ should be smaller than values given by (2).
The degrees of equivalence and the normalised errors calculated with

$$E_{\mathrm{n}} = \frac{\left|\delta p_{\mathrm{INTI}} - \delta p_{\mathrm{PTB}}\right|}{\left[U^2(\delta p_{\mathrm{INTI}}) + U^2(\delta p_{\mathrm{PTB}})\right]^{0.5}} \tag{3}$$

are presented in Table 4. The difference s and their uncertainties are shown in figure 5. According to this data, the results of the two institutes are equivalent at all pressures of the comparison.

Table 4. Differences between INTI and PTB results ($\delta p_{INTI} - \delta p_{PTB}$), expanded uncertainties (k=2) of these differences ($U(\delta p_{INTI} - \delta p_{PTB})$) and normalised errors (E_n) for measurements at increasing (up) and decreasing (down) pressures

$p_{nom.}$ in kPa	up			down		
	$\delta p_{INTI} - \delta p_{PTB}$ in Pa	$U(\delta p_{INTI} - \delta p_{PTB})$ in Pa	E_n	$\delta p_{INTI} - \delta p_{PTB}$ in Pa	$U(\delta p_{INTI} - \delta p_{PTB})$ in Pa	E_n
2	-4.1	6.5	0.6	-4.4	5.0	0.9
10	-4.4	5.1	0.9	-5.6	6.0	0.9
40	-5.2	5.1	1.0	-3.2	5.1	0.6
90	-4.2	5.1	0.8	-3.2	5.6	0.6
120	-3.6	5.4	0.7	-3.4	6.1	0.6
150	-3.4	6.9	0.5	-3.5	7.2	0.5
181	-1.0	7.9	0.1	-5.7	7.9	0.7
211	-6.7	8.8	0.8	-5.6	8.8	0.6
241	-5.4	10.8	0.5	-5.0	10.8	0.5
271	-4.6	11.9	0.4	-5.1	11.8	0.4
301	-4.4	12.8	0.3	-5.7	12.9	0.4

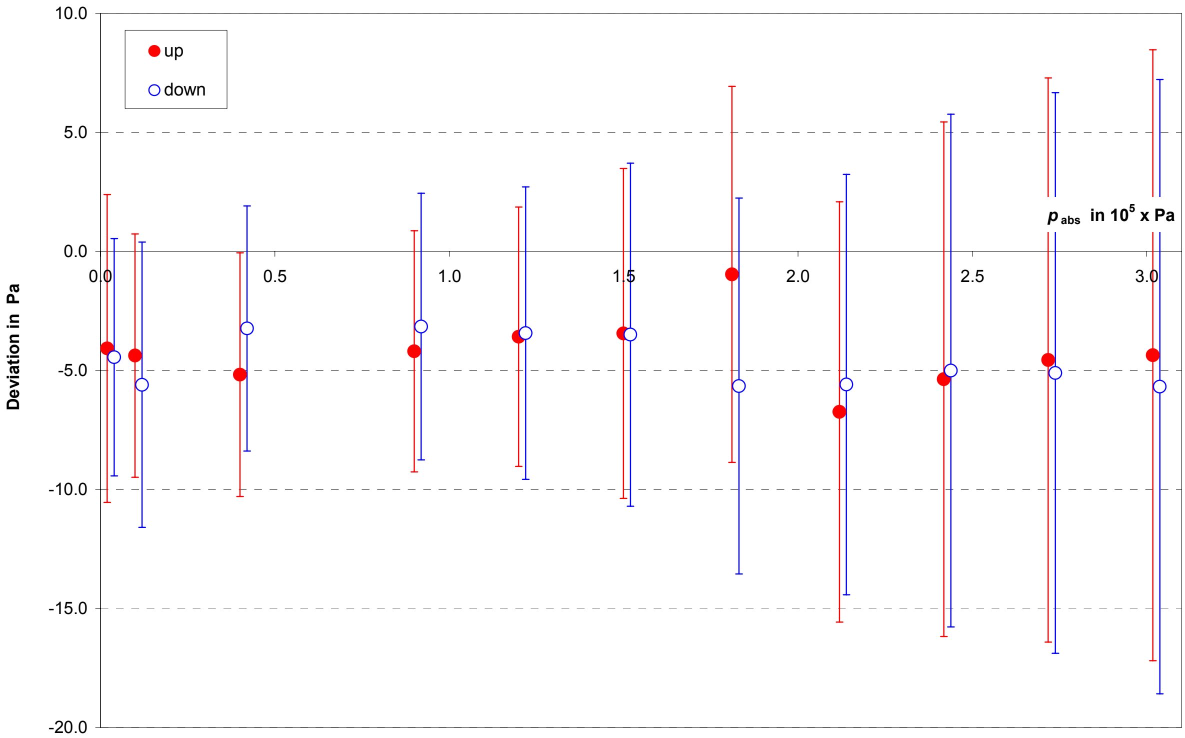

Fig. 5. Differences between INTI and PTB results and expanded uncertainties (k=2) of these differences for measurements at increasing (up) and decreasing (down) pressures

7. CONCLUSIONS

The uncertainties of INTI claimed for their capability of measuring absolute pressures are supported by this comparison. The results demonstrate a systematic nearly constant difference between the results over the whole pressure range which however agree with the uncertainties claimed by the laboratories. This difference can improbably be caused by properties of the transfer standard, which was found to be sufficiently stable during the period of the comparison. Problems with tightness of the pressure system at INTI, which could cause a pressure gradient between the INTI and the transfer standard, might be a possible reason for the systematic pressure differences. A careful check of the pressure system there is required. In addition, the uncertainty of the residual pressure measurement an INTI, while the residual pressure at INTI was about 10 times as high as that at PTB, might have been underestimated. This comparison provides a supporting basis for the INTI pressure CMC table being in preparation now.

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