Final report on the supplementary comparison Euromet.M.P-S2 (bilateral comparison) in the pressure range from 30 Pa to 7000 Pa

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Content

1. Introduction

During the last decade new types of pressure balances became available for vacuum metrology [1], [2], [3]. These instruments can measure absolute pressures, gauge pressures and pressure differences. They may serve as primary standards in National Metrological Institutes or in calibration services and replace rotating piston gauges or static expansion systems in part of their respective ranges. The measurement uncertainties that can be achieved with these pressure balances are usually considerably smaller than the ones obtained with static expansion systems or secondary standards of the capacitance diaphragm type.

Both the Czech Metrological Institute (CMI) and the Physikalisch-Technische Bundesanstalt (PTB) established such new pressure balances in their calibration schemes. In order to validate these pressure balances for absolute pressures with their respective measurement uncertainties the CMI and PTB decided to perform a bilateral comparison. The CMI acquired the force balanced piston gauge FPG by DH Instruments, the PTB the FRS5 by the Furness Company. Both of these pressure balances use a non-rotating piston-cylinder assembly and a balance as force meter, the centering of the piston, however, is performed quite differently. Also, the balance of the FPG operates in an intermediate rough vacuum of about 20 kPa between test and reference side of the piston, while the balance of the FRS5 is located in high vacuum on the reference side. These two different lay-outs make it rather improbable that there are common systematic errors in the two gauges for which reason the comparison is more useful than with two identical systems. In addition, either system was evaluated in its own institute, so that the two pressure balances are being considered completely independent.

The fact that both instruments can be transported gave the possibility to compare the two systems without any transfer standard. A zero indicator, however, had to be used to separate the two systems. The CMI system was transported to PTB, Berlin, where the measurements were performed from May 29 to June 2, 2006.

Target pressures p_t were chosen at 30 Pa, 50 Pa, 100 Pa, 300 Pa, 1 kPa, 3 kPa, 5kPa, and 7 kPa.

2. The CMI system

The physical principle of the FPG has been described in [2], the commercial set-up is described in [3]. The range of the instrument in both gauge and absolute mode is 1 Pa to 15 kPa.

As a modification of the commercial set-up a turbo-molecular pump was added to the pumping system of the reference side with the former rotary vane pump serving now only for the backing of the turbo pump. This was done in order to reduce the pressure on the reference side and the uncertainty of the measurement of the reference pressure. The lowest residual pressure attainable now is about 0.05 Pa. As a consequence, the capacitance diaphragm gauge provided by the manufacturer was replaced by an ionization vacuum gauge for the measurement of the reference pressure.

The system acquired by CMI had been carefully evaluated as described in [4]. The effective area was evaluated both by the measurement of the piston-cylinder geometry and by cross-floating techniques with classical and digital pressure balances. These comparisons were performed with standards of CMI during the year 2002 (from 5 to 15 kPa both in gauge and absolute pressure mode with PG7601 and from 1 Pa to 3.2 kPa gauge pressure with FRS4 HR). An intercomparison with the Slovak Metrological Instititute (SMU) was performed in December 2002 (from 2 to 15 kPa in both gauge and absolute pressure mode with Bell and Howell pressure balance). Another intercomparison with the Finnish national Metrological 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, both with another DHI FPG 8601).

3. The PTB system

The physical principle of the FRS5 has been described in some detail in 1999 [1]. The range of the instrument in both gauge and absolute mode is 1 Pa to 11 kPa. Some improvements in the commercial instrument have been made since then: A so called “zero” setting allows the user to disconnect the piston from the balance and to put an internal mass artefact (1 kg) on the same. This allows to record any drift of the balance during the measurements. Also, an additional turbomolecular pump was added on the test side in order to reach the base pressure more rapidly.

At PTB some more dosing valves were added to the commercial instrument in order to get more stable gas flows into the system and therefore more stable pressures.

The effective cross sectional area of the piston was determined by comparison with the U-tube mercury manometer primary standard of PTB in the range from 1 kPa up to 10 kPa both in absolute mode as well in the gauge mode. Both values agreed within the uncertainties. In absolute mode, helium and nitrogen were used to determine the effective area in order to check, if there would be any dependence of the effective area on the mean free path of the atom’s respective molecules, which was not the case. Also, there was no significant dependence of the effective cross section area are on pressure.

In addition, the effective cross section area determined by comparison with the liquid column agreed well within the uncertainties with the geometrical data obtained from measurements of piston and cylinder by a UKAS accredited laboratory.

For these reasons, it was concluded that within the standard uncertainty the effective piston area does not depend on the flow around it, respectively the test pressure.

4. Uncertainties of the two systems

The total standard uncertainty ($k = 1$) of pressure p of the FPG was estimated by the CMI by the following equation,

$$u_{\mathrm{FPG}}(p/\mathrm{Pa}) = 0.01 + 1.4 \cdot 10^{-5}\, p/\mathrm{Pa}\,, \tag{1}$$

and of the FRS5 by the PTB by

$$u_{\mathrm{FRS}}(p/\mathrm{Pa}) = \sqrt{1.73 \cdot 10^{-4} + 1.08 \cdot 10^{-8} \cdot p/\mathrm{Pa} + 5.54 \cdot 10^{-10}\, p^2/\mathrm{Pa}^2}\ . \tag{2}$$

Table 1 presents the relative standard uncertainties for the two primary standards for the target pressures in this comparison. Only Type B uncertainties according to the GUM [5] were considered, since Type A uncertainties will be revealed during the repeated measurements in this comparison, which will be considered separately.

Table 1 Relative standard uncertainties (k = 1, Type B) of generated pressures as reported by the participants at the target pressures p_t. Type A uncertainties contributing to the scatter of repeat measurements are not considered in this table.

p_t in Pa	FPG (CMI) u_{FPG}/p_t	FRS5(PTB) u_{FRS}/p_t
30	3.5E-04	4.4E-04
50	2.1E-04	2.6E-04
100	1.1E-04	1.3E-04
300	4.7E-05	5.0E-05
1000	2.4E-05	2.7E-05
3000	1.7E-05	2.4E-05
5000	1.6E-05	2.4E-05
7000	1.5E-05	2.4E-05

5. Procedure of comparison and value determined by the comparison

The aim of the comparison was to compare the two pressure values determined from the readings of the two pressure balances. Since each pressure balance has its own vacuum system and needs its own special gas flow rate to establish a certain pressure and keep it constant over time, it was not possible to directly connect the two pressure balances to have the same pressure in both systems at the same time. No stable pressure could exist in this case. Therefore the two pressure balances were connected to each other with a differential capacitance diaphragm gauge (CDG) of 1 Torr full scale in between. The set-up is shown in Fig. 1. The by-pass valve V2 bypassing the reference and test side of the CDG was used to check the CDG zero reading when V2 was open. Via the valves V1 and V3 either the FRS5 or the FPG could be disconnected from the differential CDG.

When the two systems are separated, there is no way to be sure that exactly the same pressure exists at the two places of the piston-cylinder assemblies. Although a CDG was used to detect any pressure difference p_{CDG} between the FPG and FRS5, even for an apparent zero signal we could not be sure that pressures were exactly equal. The reason is two-fold: Both systems establish a pressure by a continuous flow controlled by inlet valves and pump valves with some relaxation time for control. Even for synchronous readings it is not assured that the same homogeneous and static pressure exists at the two piston-cylinder assemblies when the differential pressure across the CDG is zero. In addition, the two temperatures inside the pressure balance systems were different (typically T_{FPG} = 296.5 K and T_{FRS} = 298.5 K), so that for the target pressures of 30 Pa and 50 Pa the effect of thermal transpiration [6] caused differently sized effects in the heated CDG gauge head (about 318 K).

For this reason we performed the comparison in the following manner: The pressure p_{FPG} evaluated from the FPG in combination with the CDG reading and the measured temperatures was used to predict the reading inside the FRS5. This predicted value was compared to the value p_{FRS} evaluated from the FRS5 itself.

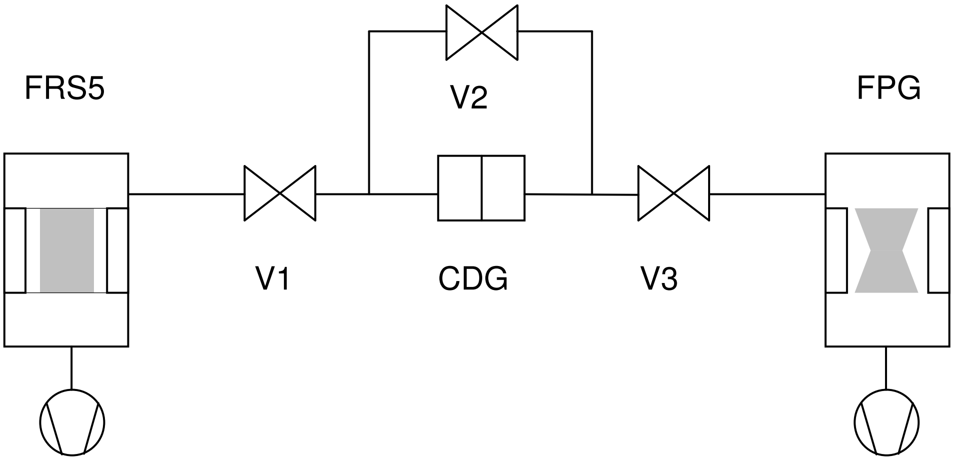

Figure 1 Experimental set-up to compare the two pressures balances FRS5 and FPG. CDG: capacitance diaphragm gauge. V1 to V3: valves.

The comparison procedure for each pressure target point was the following: In the beginning, at residual pressure conditions, V1 was closed, while V2 and V3 were open. The zero reading p_0 of the CDG was taken by the control software of the FPG. Both pressure balances were zeroed. Then the desired target pressure was adjusted by the flow controller of FPG. At the same time equal pressure was established in the FRS5. Then, V2 was closed and V1 opened. The two pressures were so close that the CDG never was near full scale. In a fine adjustment the flow rate in the FRS5 was adjusted such that the CDG showed a minimum deviation from the previous zero reading. Normally, the FRS5 pressure reading was within 0.02% of the FPG reading. At this time five successive signals of the two pressure balances and the CDG were taken. Valve V1 was closed again and V2 opened, before control of the FPG pumped the system down to base pressure. The FRS5 was evacuated as well to base pressure. Reaching this, another zero value p_0' of the CDG was taken as for each pressure balance.

If p_{FPG} denotes the pressure as determined by the FPG and p_{CDG} the pressure reading of the CDG, for target pressure p_{t} = 100 Pa and higher, where no thermal transpiration effect exists, the predicted pressure in the FRS5 is given by:

$$(p_{\mathrm{FRS}})_{\mathrm{predicted}} = p_{\mathrm{FPG}} - \left(p_{\mathrm{CDG}} - (p_0 + p_0')/2\right) \cdot CF \qquad (3)$$

Herein *CF* denotes the correction factor as determined by a calibration of the CDG before and immediately after the comparison. The zero corrected signal $\left(p_{\mathrm{CDG}} - (p_0 + p_0')/2\right) \cdot CF$ of the CDG had to be subtracted from p_{FPG} to predict the reading p_{FRS}, since the test side of the CDG was connected with the FPG. Therefore a positive signal indicated that the pressure on the FPG side was higher than on the FRS5 side (the reference side of the CDG).

For lower p_{t}, p_{FRS} can be predicted in the following manner: If T_{FPG}, T_{FRS}, and T_{CDG} are the absolute temperatures inside the FPG, FRS5, and the CDG respectively, $p_{\mathrm{CDG,ref}}$ and $p_{\mathrm{CDG,test}}$ denote the absolute pressures on the reference respectively test side of the CDG, and p_{CDG} the differential pressure reading of the CDG at target pressure, the following equations hold:

$$\frac{p_{\mathrm{CDG,\,ref}}}{p_{\mathrm{FRS}}} = 1 + f(p_t) \cdot \left(\sqrt{\frac{T_{\mathrm{CDG}}}{T_{\mathrm{FRS}}}} - 1 \right) \tag{4}$$

$$\frac{p_{\mathrm{CDG,\,test}}}{p_{\mathrm{FPG}}} = 1 + f(p_t) \cdot \left(\sqrt{\frac{T_{\mathrm{CDG}}}{T_{\mathrm{FPG}}}} - 1 \right), \tag{5}$$

$$\left(p_{\mathrm{CDG}} - (p_0 + p_0')/2\right) \cdot CF = p_{\mathrm{CDG,test}} - p_{\mathrm{CDG,\,ref}} \quad . \tag{6}$$

where $f(p_{\mathrm{t}})$ denotes a factor (0…1) which describes to what extent the thermal transpiration effect can develop at the target pressure p_{t}. From the calibrations of the CDG $f(p_{\mathrm{t}})$ is estimated to 0.07 at 30 Pa and 0.03 at 50 Pa and 0.00 for all other pressures. From these equations one obtains

$$\left(p_{\mathrm{FRS}}\right)_{\mathrm{predicted}} = \frac{p_{\mathrm{FPG}} \left[1 + f(p_{\mathrm{t}}) \left(\sqrt{\frac{T_{\mathrm{CDG}}}{\mathrm{T}_{\mathrm{FPG}}}} - 1 \right) \right] - \left(p_{\mathrm{CDG}} - (p_0 + p_0')/2\right) \cdot CF}{1 + f(p_{\mathrm{t}}) \left(\sqrt{\frac{T_{\mathrm{CDG}}}{\mathrm{T}_{\mathrm{FRS}}}} - 1 \right)} \tag{7}$$

For $f(p_{\mathrm{t}}) = 0$, Eq. (7) is identical to Eq.(3). The temperatures were T_{FPG} = 296.5K , T_{FRS} = 298.5 K, and T_{CDG} = 318 K.

These predicted values had to be compared to the value determined by FRS5.

For each measurement i (i = 1…5) on day j (j = 1,2) at the defined target pressure the difference d_{ij} between the two systems was obtained by:

$$d_{ij} = p_{\mathrm{FRS}} - \left[p_{\mathrm{FPG}} - \left(p_{\mathrm{CDG}} - (p_0 + p_0')/2\right) \cdot CF\right] \quad p_{\mathrm{t}} \geq 100\ \mathrm{Pa} \tag{8}$$

$$d_{ij} = p_{\mathrm{FRS}} - \frac{p_{\mathrm{FPG}}\left[1 + f(p_{\mathrm{t}})\left(\sqrt{\frac{T_{\mathrm{CDG}}}{\mathrm{T}_{\mathrm{FPG}}}} - 1\right)\right] - \left(p_{\mathrm{CDG}} - (p_0 + p_0')/2\right)\cdot CF}{1 + f(p_{\mathrm{t}})\left(\sqrt{\frac{T_{\mathrm{CDG}}}{\mathrm{T}_{\mathrm{FRS}}}} - 1\right)} \qquad p_{\mathrm{t}} < 100\ \mathrm{Pa}$$

On a single measurement day, the target pressures were raised from the lowest to the highest point. As already mentioned, the measurement series was repeated on a separate day.

In order to be able to assess the equivalence of the two systems, for each target pressure a single value of *d* was calculated by taking the mean of all measurements of the two days:

$$d = \frac{1}{10}\cdot\sum_{j=1}^{2}\sum_{i=1}^{5} d_{ij}\ . \tag{9}$$

6. Uncertainties

The model of the determined value, *d*, has been formulated in Eq.(9). The uncertainty u_d of *d* is determined by the uncertainties of p_{FPG}, p_{FRS}, p_{CDG}, p_0, p_0', *CF*, and the terms $f(p_{\mathrm{t}})\left(\sqrt{\frac{T_{\mathrm{CDG}}}{\mathrm{T}_{\mathrm{FRS}}}} - 1\right)$ and $f(p_{\mathrm{t}})\left(\sqrt{\frac{T_{\mathrm{CDG}}}{\mathrm{T}_{\mathrm{FPG}}}} - 1\right)$.

The uncertainties of p_{FPG} and p_{FRS} were already listed in Section 4.

p_{CDG}, p_0, and p_0' are inaccurate by the scatter of reading, the resolution scatter, offset instability and short term instabilities all of which are revealed in the scatter of repeat calibrations. Therefore these uncertainties are being considered in the experimental standard deviation of the mean of *d*.

The correction factor *CF*, however, was determined before and after the comparison and is according to the GUM [5] a Type B uncertainty. It must be noted that the CDG was operated at the target pressure (30 Pa to 7000 Pa) as line pressure and only the small pressure differences between FRS5 and FPG gave a signal on the CDG. This difference was typically $2\cdot10^{-4}$ of the target pressure, in the maximum, at 30 Pa, $3\cdot10^{-3}$. This means that a relative uncertainty of 1 % of the reading of the CDG would cause relative uncertainties between $2\cdot10^{-6}$ and $3\cdot10^{-5}$ of the target pressure only. In fact, the total relative expanded (k = 2) uncertainty $2\cdot u_{CF}/CF$ of the CDG is estimated to 0.32%, calculated from the calibration uncertainty (0.15%), the long-term instability (0.2%), and an uncertainty (0.2%) as a possible difference between the differential (or line) pressure and absolute pressure mode, since the CDG was calibrated in the absolute mode only. It is well known that the difference between the differential pressure mode and absolute mode is little beyond the thermal transpiration regime [7].

The total uncertainty u_d of d for each target pressure ≥ 100 Pa is given by:

$$u_d = \sqrt{u_{\mathrm{FRS}}^2 + u_{\mathrm{FPG}}^2 + \left(p_{\mathrm{CDG}} - (p_0 + p_0')/2\right)^2 \cdot u_{CF}^2 + \frac{n-1}{n-3} s_{d_{ij}}^2} \quad p_{\mathrm{t}} \geq 100\ \mathrm{Pa}\,, \tag{10}$$

where $s_{d_{ij}}^2$ is the square of the standard deviation of the mean of the repeated measurements d_{ij}. Since n = 10 measurements were taken with an effective degree of freedom of 9, $s_{d_{ij}}$ was multiplied by $\sqrt{(n-1)/(n-3)}$, i.e. 1.13, as suggested by Kacker and Jones [8].

Since the sensitivity coefficient related to u_{CF} changed with each d_{ij} we took the mean of the variance $\left(p_{\mathrm{CDG}} - (p_0 + p_0')/2\right)^2 \cdot u_{CF}^2$ of the 10 measurements for the evaluation of u_d in Eq. (10).

The terms *corr1* = $f(p_{\mathrm{t}})\left(\sqrt{\frac{T_{\mathrm{CDG}}}{\mathrm{T}_{\mathrm{FRS}}}} - 1\right)$ and *corr2* $= f(p_{\mathrm{t}})\left(\sqrt{\frac{T_{\mathrm{CDG}}}{\mathrm{T}_{\mathrm{FPG}}}} - 1\right)$ are rather small (< 0.003) and are estimated as Type B uncertainty with a relative standard uncertainty of 10%. This covers both the uncertainty of $f(p_{\mathrm{t}})$ and the measured temperatures (T_{CDG} is known only from the manufacturer) as well as the fact that the gas temperatures might be different from the measured temperatures of the pressure balance bodies.

$$u_d = \sqrt{\begin{array}{l} u_{\mathrm{FRS}}^2 + \left(\frac{1+corr1}{1+corr2}\right)^2 u_{\mathrm{FPG}}^2 + \left(\frac{\left(p_{\mathrm{CDG}} - (p_0 + p_0')/2\right)}{1+corr2}\right)^2 \cdot u_{CF}^2 + \\ \left(\frac{p_{\mathrm{FPG}}}{1+corr2}\right)^2 \cdot u_{corr1}^2 + \left(\frac{p_{\mathrm{FPG}}(1+corr1))}{(1+corr2)^2}\right)^2 \cdot u_{corr2}^2 + \frac{n-1}{n-3} s_{d_{ij}}^2 \end{array}} \quad p_{\mathrm{t}} < 100\ \mathrm{Pa}\,, \tag{11}$$

7. Results

The results for the d_{ij} on the two measurement days agreed within their uncertainties which justified the procedure to take the mean of all data. Table 2 gives the summarized results for d for each target pressure p_{t}, the experimental standard deviation of the mean of the d_{ij} and the absolute and relative total expanded (k = 2) uncertainty U_d of d.

Table 2 The results of the comparison. p_t is the target pressure, d is defined by Eqs. (9) and (8), $u_A(d)$ is the experimental standard deviation of the mean, $U_d = 2 \cdot u_d$ with u_d defined in Eqs. (10) and (11), E_n is defined in Eq. (14).

p_t	d	d/p_t	$u_A(d)$	$u_A(d)/p_t$	U_d	U_d/p_t	E_n
Pa	**Pa**	**rel.**	**Pa**	**rel.**	**Pa**	**rel.**	
30	-0.0026	-8.6E-05	0.0014	4.6E-05	0.0342	1.1E-03	0.08
50	0.0094	1.9E-04	0.0057	1.1E-04	0.0363	7.3E-04	0.26
100	0.0126	1.3E-04	0.0015	1.5E-05	0.0359	3.6E-04	0.35
300	0.0203	6.8E-05	0.0046	1.5E-05	0.0432	1.4E-04	0.47
1000	0.0088	8.8E-06	0.0057	5.7E-06	0.0746	7.5E-05	0.12
3000	0.0126	4.2E-06	0.0049	1.6E-06	0.1815	6.1E-05	0.07
5000	0.0037	7.4E-07	0.0118	2.4E-06	0.2898	5.8E-05	0.01
7000	0.0518	7.4E-06	0.0227	3.2E-06	0.4087	5.8E-05	0.13

Equivalence is generally assumed, if

$$d \le 2u_d = U_d \tag{12}$$

or

$$E_n \le 1 \tag{13}$$

with

$$E_n = \frac{|d|}{U(d)} \quad . \tag{14}$$

Figure 2 gives a graphical illustration of the relative difference of the two pressure balances in dependence of pressure.

8. Discussion and conclusions

It can be seen from Table 2 that all E_n values were < 0.5, so that full equivalence of the two pressure balances could be shown. All relative differences d/p_t from 30 Pa to 7000 Pa were smaller than 0.002% demonstrating the close agreement of the two systems.

It was also apparent, however, that $d > 0$ for any but the lowest target pressure. This means that the pressure evaluated from the FRS5 was systematically, though only slightly, higher than the one predicted from the FPG. Therefore it is highly probable that at least one factor common to all target pressures in at least one system, e.g. the effective piston area or the calibration of the balance, caused this albeit insignificant effect.

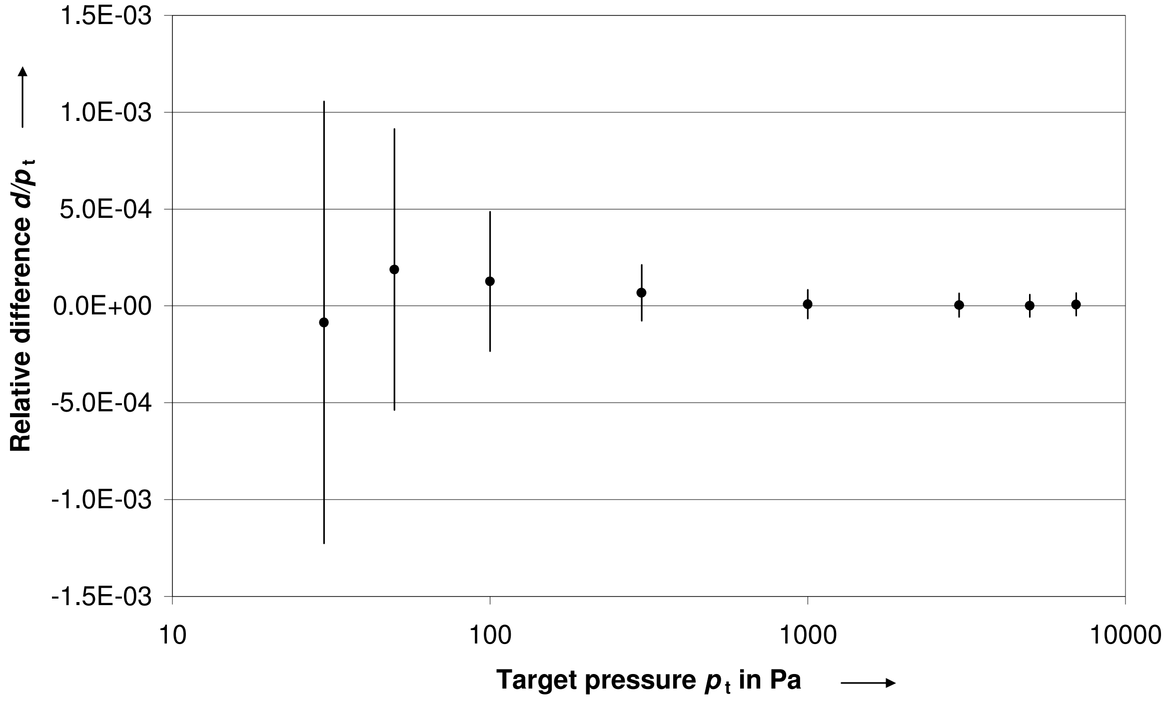

Figure 2 Graphical representation of the relative difference d/p_t between the pressures evaluated from the PTB and CMI pressure balances. d was calculated according to Eq. (9). Uncertainty bars are given as ± $2u_d/p_t$. When value plus or minus the uncertainty bar overlap with zero, the two systems are considered equivalent.

The close agreement indicates that the pressure control within the pressure balances does not significantly affect the homogeneity of pressure in the connected volumes to the pressure balances.

It is also evident from Table 2 that there is no pressure dependence in the sense that lower or higher pressures give significantly larger deviations of the two systems. This justifies the assumption made for both pressure balances that the type of flow has no influence on the effective area within the uncertainties of the respective measurements. Since the shape and the clearances of the two pistons are different as well as the gas flow rates and the type of gas (humidified nitrogen in the case of the FPG, pure nitrogen in the case of the FRS5), it is highly improbable that any flow effect on the effective area would exactly cancel out each other at any pressure.

At PTB the FRS5 was established to replace the static expansion system from 30 Pa to 1000 Pa as primary standard. By this comparison the validation was completed and the FRS5 can be used to improve the realisation of the pressure scale and the calibration service.

This was the first time that the CMI system was compared to a pressure balance traceable to a mercury liquid column while previous comparisons of this system were only with devices traceable to geometrical measurements. The success of this independent comparison gives additional confidence in the performance and the evaluated uncertainty of the instrument.

9. References

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