Final report

Final report on EURAMET.M.P-S12 – Bilateral Supplementary Comparison of the National Pressure Standards of CMI and INRIM in the Range 300 Pa to 15 kPa of Negative Gauge Pressure

Zdeněk Krajíček[1], *Mercede Bergoglio*[2], *Dominik Pražák*[1], *Stefano Pasqualin*[2]

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1. Czech Metrology Institute (CMI), Okruřní 31, 63800 Brno, Czech Republic
2. Istituto Nazionale della Ricerca Metrologica (INRIM), Strada delle Cacce 91, 10135 Torino, Italy

Abstract

This report describes a EURAMET bilateral supplementary comparison between Czech CMI and Italian INRIM in low negative gauge pressure in gas (nitrogen), denoted as EURAMET.M.P-S12.

The digital non-rotating pressure balance FPG8601 manufactured by Fluke/DH-Instruments, USA is normally used for gauge and absolute pressures in the range from 1 Pa to 15 kPa, but with some modifications it can be used also for the negative gauge pressures in the same range.

During the preparation of the visit of INRIM at CMI for the last comparison within the framework of EURAMET.M.P-K4.2010, it was agreed to perform also an additional comparison in the range from 300 Pa to 15 kPa of negative gauge pressure. The measurements were performed in October 2012.

Both institutes successfully proved their equivalence in all the tested points in the range from 300 Pa to 15 kPa of negative gauge pressure in a comparison which had been unique so far.

Content

1. Introduction

The digital non-rotating pressure balance FPG8601 manufactured by Fluke/DH-Instruments is based on a 10 cm^2 non-rotating tungsten-carbide piston-cylinder with a conical gap [1]. It is normally used for gauge and absolute pressures in the range from 1 Pa to 15 kPa, but with some modifications it can be used also for the negative gauge pressures in the same range.

However, measurement of the negative gauge pressures has been a neglected branch of the primary metrology with the lack of the international interlaboratory comparisons [2]. Recently an intercomparison (EURAMET.M.P-S8) was performed [3] and two others are being in progress (EURAMET.M.P-S9, SIM.M.P-S5), but none of these covers the negative gauge pressures lower than 10 kPa.

In the year 2011, CMI got experience with utilizing FPG 8601 in the negative gauge pressure mode. During the preparation of the visit of INRIM at CMI for the last comparison within the framework of EURAMET.M.P-K4.2010, it was agreed to perform also an additional comparison in the range from 300 Pa to 15 kPa of negative gauge pressure. It was registered as a supplementary comparison (EURAMET.M.P-S12) and it is aimed to verify the equivalence of the national low negative gauge pressure standards of Czech Republic and Italy. The staff of INRIM volunteered to transport their FPG8601 to CMI, Brno, where the measurements were performed from October 4 - 5, 2012.

Each standard was evaluated in its own institute, so that they were considered independent. The fact that both standards met in one laboratory gave the possibility to compare the two systems without any transfer standard. A capacitance diaphragm gauge (CDG), however, had to be used to separate them. In this case a digital pressure controller Fluke/DH-Instruments PPC3-200K was used for pressure regulation of both FPGs instead of Fluke/DH-Instruments Very Low Pressure Controllers (VLPCs) which is otherwise normally utilized.

The Technical Protocol of this comparison was prepared on the basis of Technical Protocol of EURAMET.M.P-K4.2010 and in accordance with the Guidelines for CIPM Key Comparisons. The nominal negative gauge pressure points were 300 Pa, 1 kPa, 3 kPa, 10 kPa and 15 kPa. Measurements were performed in two cycles in two different days. Nitrogen was used as pressure transmitting medium.

2. Standard of CMI

The standard of CMI was a digital non-rotating piston gauge FPG8601, identified by serial number 107, see [4]. The effective area was evaluated by the measurement of the piston-cylinder geometry and validated by the cross-floating techniques. An intercomparison with the Slovak SMU was performed in 2002, with the Finnish MIKES in 2003, EUROMET.M.P-S2 in 2006 and EURAMET.M.P-K4.2010 is still running. The uncertainty (for k = 1) of this standard equals $u_C = 0.01\ \text{Pa} + 1.4\cdot10^{-5}\cdot p$, where p is in pascal.

In this case a DH-Instruments PPC3-200K, identified by serial number 225, was used as a pressure regulator instead of a VLPC. (When measuring in negative mode, the VLPCs of both FPGs were connected to their FPGs only electrically but pneumatically disconnected.)

A CDG (MKS Baratron of type 698A01TRA, identified by serial number 000043657, with control unit of type 270, identified by serial number 000042869) with a set of valves served as a zero indicator and as a separator between both standards. This instrument is capable of reading via PC with installed FPG TOOLS. It was provided by CMI with a calibration for both plus and minus indications with an emphasis on the range around zero. Its multiplicative correction factor C_{CDG} = (1.010 ±0.009), for k = 1 (consisting of the calibration uncertainty and the long-term stability). However, during the measurements the CDG indication was kept as near to zero as possible.

3. Standard of INRIM

The standard of INRIM was a digital non-rotating piston gauge FPG8601 manufactured by DH-Instruments, identified by serial number 132. Its uncertainty (for k = 1) was evaluated equal to $u_I = 0.01\ \text{Pa} + 1.5\cdot10^{-5}\cdot p$, where p is in pascal.

The effective area was evaluated by the measurement of the piston-cylinder geometry and validated by the cross-floating techniques in gauge mode. Both values agreed within the uncertainties.

4. Procedure of the comparison

The nominal negative gauge pressure points p_n were 300 Pa, 1 kPa, 3 kPa, 10 kPa and 15 kPa. Measurements were made in two cycles. Each cycle was performed at a different day. The pressure transmitting medium was dry nitrogen (dry is the gas entering FPG stand, however the FPG adjusts humidity of the gas to approximately 50 % via its internal reservoir of water).

Both standards were located close to each other to keep the pressure line between the two instruments as short as possible. There was no height difference between the reference levels of both standards within an uncertainty of about 1 mm. (Pressure uncertainty of small pressure head is included in the declared uncertainty of the FPG of the CMI.) Both reference ports (but in the negative gauge mode the upper port serves as the reference port) of both standards were fully open to atmosphere. The VLPCs of both FPGs were connected to their FPGs only electrically but disconnected pneumatically and the setting “maximum range” was chosen in the software menus of both FPGs.

The comparison measurements were performed using 1 torr CDG as a zero indicator. A bypass line with a valve connected the both sides of the zero pressure indicator to control its zero pressure reading. The CDG was not heated during the measurements.

The CDG was connected to both standards via tubings (bellows) that were as similar to each other as much as possible concerning their diameters and volumes. The by-pass closing valve of the CDG did not induce large changes of pressure.

Before the start of the comparison measurements both standards were zeroed and then calibrated internally. (Check of the internal calibration was repeated every four hours.) Then the by-passes of the FPGs were closed by a software command and the regulation by PPC3 (connected to the low pressure port) was activated.

Then both instruments were zeroed again and the zero was checked and read. Then the isolation valve between both standards was closed (but with CDG by-pass valve remaining open). Only after this, the target nominal pressure was set by an FPG that was not connected to the CDG at the moment. Then the generated target pressure was set by the other FPG (filling also CDG). After stabilization, the zero of the CDG was read by the open by-pass valve. Then the by-pass valve was closed and the isolating valve open. After stabilization of reading, 5 successive readings were taken by averaging outputs of FPGs and CDG during at least 1 min. After measuring a point, a check of the CDG zero drift (if sufficiently stable this checking needs not to be performed after every point) and check of the zero drifts of both standards were done. The results were corrected for these drifts.

5. Evaluation of the comparison

The pressure defined from the FPG of CMI (corrected for its zero drift) in combination with the CDG reading was used to predict the reading inside the FPG of INRIM. This predicted value $(p_{\mathrm{I}})_{\mathrm{predicted}}$ was compared to the value p_{I} evaluated from the FPG of INRIM itself (also corrected for its zero drift).

If p_{C} denotes the pressure as determined by the standard of CMI and p_{CDG} the pressure reading of the CDG, the predicted pressure in the standard of INRIM is given by:

$$(p_{\mathrm{I}})_{\mathrm{predicted}} = p_{\mathrm{C}} - \left(p_{\mathrm{CDG}} - (p_0 + p_0')/2\right) \cdot C_{\mathrm{CDG}}, \quad (1)$$

where

p_0 zero reading of the CDG before the measurement,

p_0' zero reading of the CDG after the measurement,

C_{CDG} calibration factor of the CDG.

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 is calculated as:

$$d_{ij} = p_{\mathrm{I}ij} - \left(p_{\mathrm{I}ij}\right)_{\mathrm{predicted}}. \quad (2)$$

For each nominal pressure a single value of *d* is calculated by taking the mean of all measurements of the two days:

$$d = \frac{1}{10} \cdot \sum_{j=1}^{2} \sum_{i=1}^{5} d_{ij} \;. \quad (3)$$

The uncertainty u_d of *d* is determined by the uncertainties of p_{C}, p_{I}, p_{CDG}, p_0, p_0', C_{CDG}. The uncertainties of p_{C}, p_{I} and p_{CDG} (denoted as u_{C}, u_{I} and $u_{C_{\mathrm{CDG}}}$) were already listed in Sections 2 and 3. Since the sensitivity coefficient for $u_{C_{\mathrm{CDG}}}$ varied significantly at a pressure point, we took the maximum value. Because the CDG was calibrated by the FPG of CMI, we assume a full correlation between u_{C} and $u_{C_{\mathrm{CDG}}}$. The uncertainties of p_{CDG}, p_0, p_0', are inaccurate by the scatter and short term instabilities 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*. Since *n* = 10 measurements were taken with an effective degree of freedom of 9, the square of the standard deviation of the mean of the repeated measurements $s_{d_{ij}}$ was multiplied by $(n-1)/(n-3)$, as suggested by Kacker and Jones [5].

Hence the total uncertainty u_d of d for each nominal pressure is then given by:

$$u_d = \sqrt{u_\mathrm{I}^2 + \left(u_\mathrm{C} + \left|p_\mathrm{CDG} - \frac{p_0 + p_0'}{2}\right| u_{C_\mathrm{CDG}}\right)^2 + \frac{n-1}{n-3}\left(s_{d_{ij}}\right)^2} \,. \tag{4}$$

6. Results of the comparison

Tab. 1 gives the summarized results for d for each nominal pressure p_n, the experimental standard deviation of the mean of the d_{ij}, the remaining sources of uncertainty and the total uncertainty u_d of d.

p_n	d	s_d	u_{C_CDG}	u_C	u_I	u_d
Pa	Pa	Pa	Pa	Pa	Pa	Pa
300	-0,037	0,008	0,0003	0,014	0,015	0,022
1000	-0,010	0,004	0,0003	0,024	0,025	0,035
3000	0,045	0,001	0,0003	0,052	0,055	0,076
10000	0,275	0,006	0,0003	0,150	0,160	0,220
15000	0,432	0,009	0,0003	0,220	0,235	0,322

Tab. 1: Results of the comparison and uncertainty budget.

Tab. 2 gives the overview of d for each nominal pressure p_n, the uncertainties declared by both participants, the total expanded (k = 2) uncertainty U_d of d and the degrees of equivalence. Fig. 1 a 2 give a graphical illustration of this. It can be seen from Tab. 2 that all E_n (normalized error) values were always within ±1, so that the full equivalence of both standards was proved.

p_n	d	U_d	U_C	U_I	E_n
Pa	Pa	Pa	Pa	Pa	-
300	-0,037	0,044	0,028	0,029	-0,84
1000	-0,010	0,070	0,048	0,050	-0,14
3000	0,045	0,152	0,104	0,110	0,30
10000	0,275	0,439	0,300	0,320	0,63
15000	0,432	0,645	0,440	0,470	0,67

Tab. 2: Results of the comparison, relevant uncertainties and equivalence.

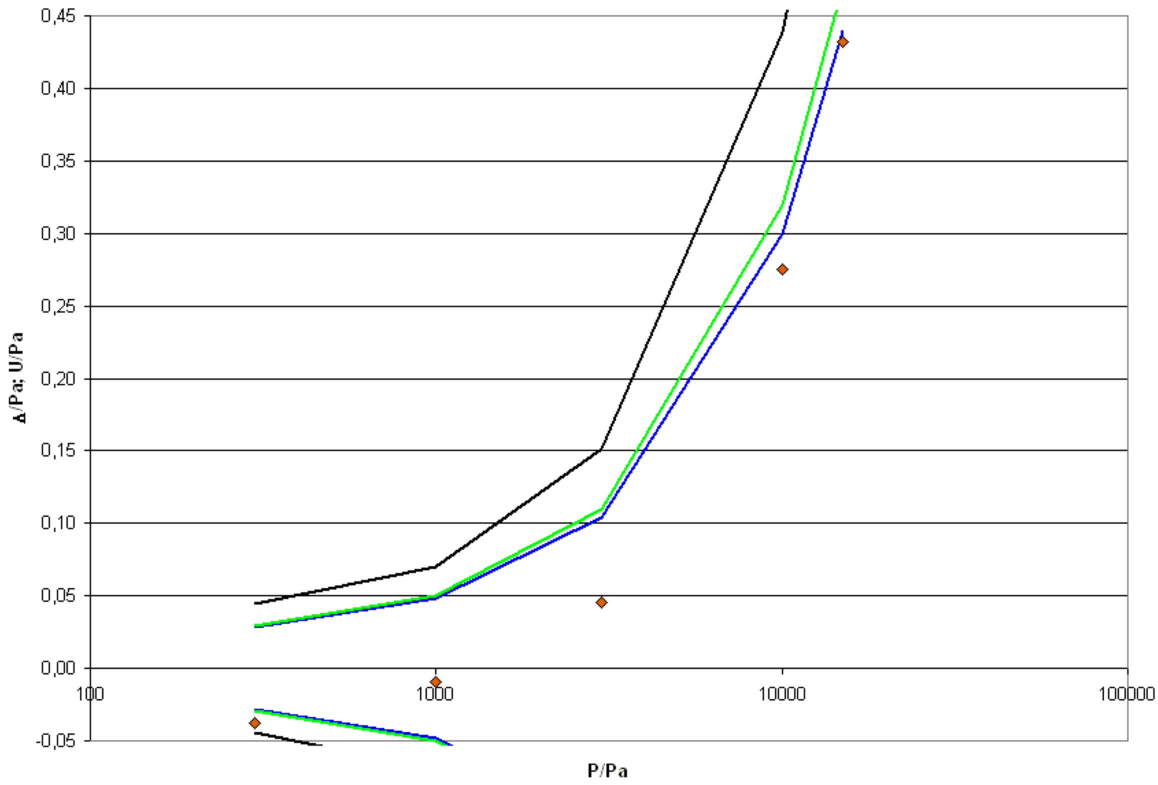

Fig. 1: Results of the comparison and relevant uncertainties. d – diamonds, U_C – blue line, U_I – green line, U_d – black line.

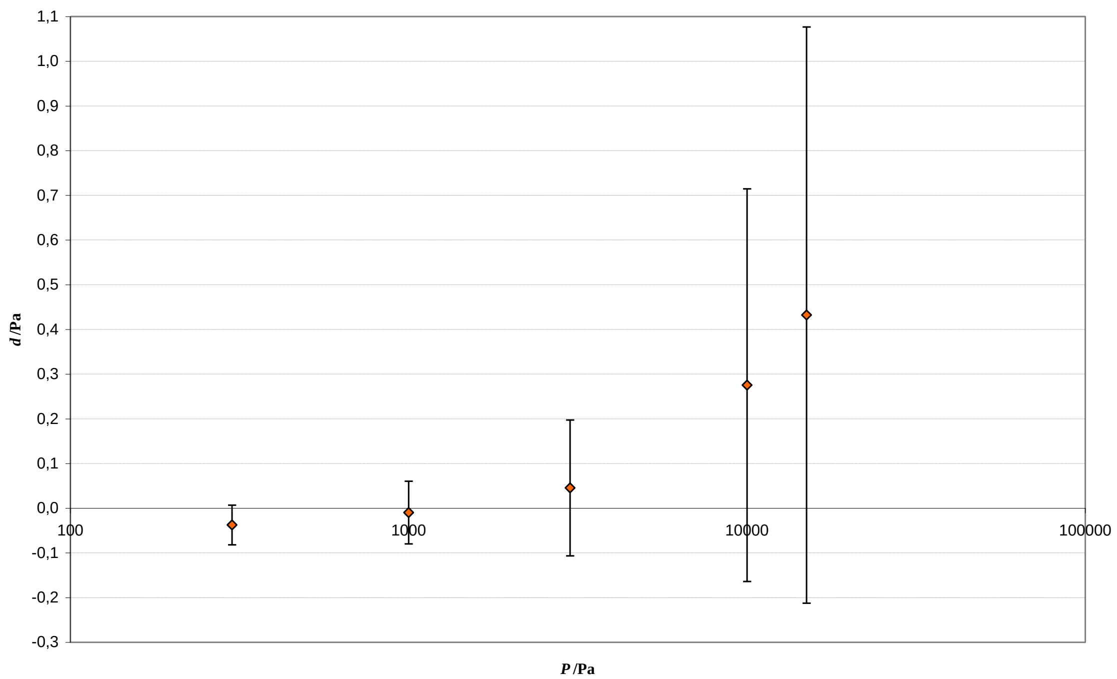

Fig. 2: Results of the comparison and relevant uncertainties. d – diamonds, U_d – error bars.

7. Conclusions

Both institutes successfully proved their equivalence in the range from 300 Pa to 15 kPa of negative gauge pressure in a comparison which had been unique so far.

8. References

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