

# PRIMARY ETALONNAGE OF NEGATIVE GAUGE PRESSURES USING PRESSURE BALANCES AT THE CZECH METROLOGY INSTITUTE

## PRIMARNE KALIBRACIJSKE METODE ZA NEGATIVNI RELATIVNI TLAK S TLAČNIMI TEHTNICAMI NA ČEŠKEM INŠTITUTU ZA METROLOGIJO

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This paper summarizes the methods of using the piston pressure balance principle for the etalonnage of negative gauge pressures, i.e., the classical piston pressure balance with an inversed piston cylinder or working under an evacuated bell jar, digital non-rotating piston pressure balances and an absolute pressure divider. It describes the principles of their use (focusing on their utilization at the Czech Metrology Institute), the methods of traceability and analyzes their achievable uncertainties.

Keywords: negative gauge pressure, pressure balance, traceability

V članku so predstavljene različne metode z batnimi tlačnimi tehtnicami za umerjanje merilnikov negativnega tlaka, tj. klasične batne tlačne tehtnice z obrnjenim sklopom bat-valj ali z evakuirano zvonasto posodo, digitalne batne tlačne tehtnice z nevrtečim se batom in z delilnikom absolutnega tlaka. Opisan je princip dela s poudarkom na njihovi rabi na Češkem inštitutu za metrologijo, načini sledljivosti in analiza dosegljivih merilnih negotovosti.

Ključne besede: merilnik negativnega pritiska, tlačna tehtnica, sledljivost

## 1 INTRODUCTION

The etalonnage of negative gauge pressures is a very much neglected branch of primary metrology (although very important in practice). This peripheral position is documented by the fact that no Key Comparison has ever been performed in this range. The only such comparisons known to the authors were organized at the national level, e.g. <sup>1,2</sup>. Simultaneously, the use of piston pressure balances as the primary standards of negative gauge pressure leads to very interesting constructions. The Laboratory of Pressure Metrology of the Czech Metrology Institute intends to ensure this pressure range via piston pressure balances.

In this paper the values of the negative gauge pressure  $P_{ng}$  will be positive and all the uncertainties will be unexpanded ( $k = 1$ ).

## 2 CLASSICAL PRESSURE BALANCES

Pressure balances can also be utilized for the primary etalonnage of negative low pressures <sup>3</sup>. There are three basic set-ups enabling this. Firstly, an **inverse piston-cylinder** design; secondly, the utilization of an **evacuated bell jar**; and thirdly, an **absolute pressure balance with a reference pressure monitor**, enabling the calculation of the negative gauge pressure from their pressure difference (not discussed further in this article).

The first solution is based on a piston cylinder that is vertically reversed (**Figure 1**). It is held in its floating position by the atmospheric pressure  $P_a$  acting upwards, balancing the residual pressure  $P$  acting downwards on the piston together with the force  $F$  that is the sum of the gravitational forces acting on the piston and the loaded masses. So, the generated negative gauge pressure is given by the following equation:

$$P_{ng} = P_a - P = \frac{M \left( 1 - \frac{\rho_a}{\rho_M} \right) g}{A_{ef}(p, T)} \quad (1)$$

where:

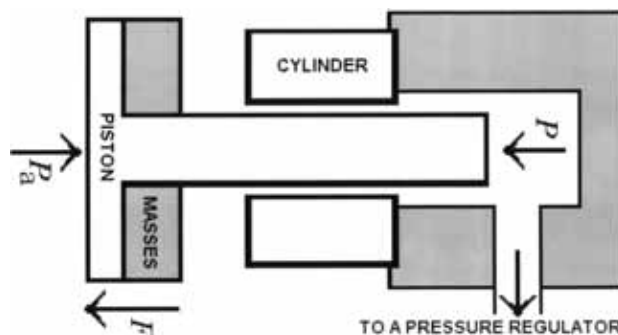


Figure 1: The inverse piston-cylinder design

Slika 1: Prikaz delovanja obrnjenega sklopa bat-valj

$A_{ef}(p,T)$  is the effective area of the piston cylinder that is generally dependent on the acting pressure  $p$  and its temperature  $T$ ,

$M$  is the total mass of the piston and the masses loaded on it,

$\rho_M$  is the average density of the piston and the masses,

$\rho_a$  is the density of the ambient air,

$g$  is the local acceleration due to gravity.

In practice, the piston cylinder is mounted on tubing, enabling it to be calibrated in the gauge mode in the normal position and then turned down and utilized for negative gauge pressures. This solution, however, brings about certain restrictions. For the range up to 100 kPa generated by a piston cylinder with a nominal effective area of 10 cm<sup>2</sup> (widely used in this range of gauge pressures) a total mass of 10 kg is needed, which introduces design complications. This is solved by inverse piston cylinders with a nominal effective area of 1 cm<sup>2</sup>, which, on the other hand, reduce the sensitivity.

The second solution is based on a classical piston-cylinder design working under a bell jar (Figure 2). This is normally used for working in the absolute mode when the space under the bell jar is evacuated (and the residual pressure is measured with a suitable vacuum meter). In this case, however, the ambient atmospheric pressure is applied under the piston, while under the bell jar such an absolute pressure is set in order to balance (together with the pressure defined by the piston cylinder) the atmospheric pressure. So the generated negative gauge pressure is given as:

$$P_{ng} = P_a - P = \frac{M \left( 1 - \frac{\rho_g}{\rho_M} \right) g}{A_{ef}(p, T)} \quad (2)$$

where  $\rho_g$  is the density of the gas pressure medium under the bell.

The second solution is very useful because it is possible to utilize a piston manometer in a reversed set-up of its absolute mode, only with the possibility to

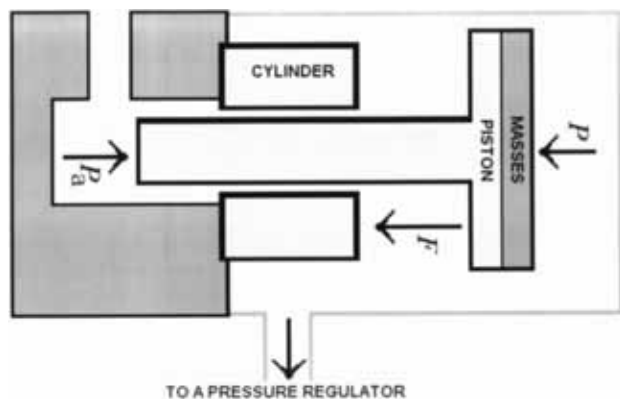


Figure 2: Principle of utilizing a bell to measure negative gauge pressures

Slika 2: Prikaz rabe zvonaste posode za merjenje negativnega pritiska

employ piston cylinders with large effective areas. However, there are also certain disadvantages. The space beneath the vacuum bell jar has a large volume that requires a very skilled operator. Another task is to accurately determine the density of the gas under the bell jar. Furthermore, for measuring the low negative gauge pressures, it is necessary to evacuate the bell jar first in order to reach an efficient sealing level, and only then is it possible to return to low negative gauge pressures.

The laboratory utilizes the classical DHI PG7601 piston gauge<sup>4,5</sup> with a piston cylinder 10 cm<sup>2</sup> in the mode described above. It is also equipped with an automated mass-handling system and automated pressure regulators. This system was developed for work in the absolute mode, but the laboratory also utilizes it in the negative gauge mode with very satisfactory results.

The simple free-deformation piston cylinders are utilized in this mode. Dadson and Sutton's model of an effective area of a piston cylinder also depends on the pressure distribution along the gap and the viscosity and density of the pressure medium used as functions of the pressure. It would therefore be necessary to evaluate the effective area not only for various working modes (absolute, gauge, negative gauge) but also for various generated pressure points. Numerical and experimental studies have proven that these changes never exceed 0.0002 %, which is negligible.

The calculation of the uncertainty of the negative gauge pressure generated by the above-mentioned piston cylinder is based on equation (2). The effective area value and its uncertainty  $u_{A_{ef}}$  were obtained from geometrical measurements using Dadson and Sutton's model<sup>3</sup> and were confirmed by the hydrostatic cross-floating method, both in absolute and gauge modes. The

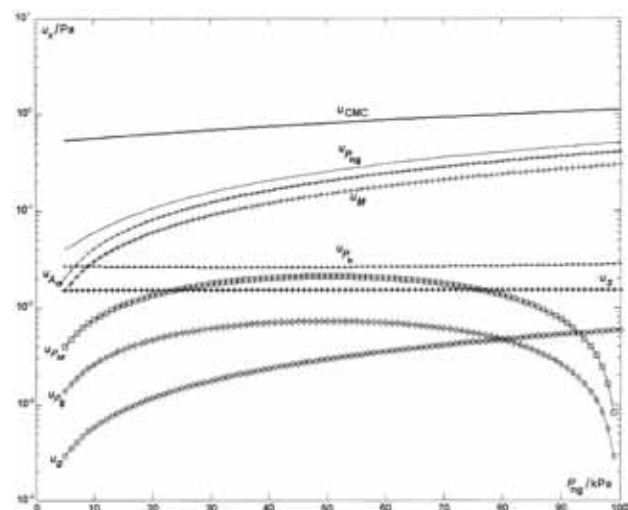


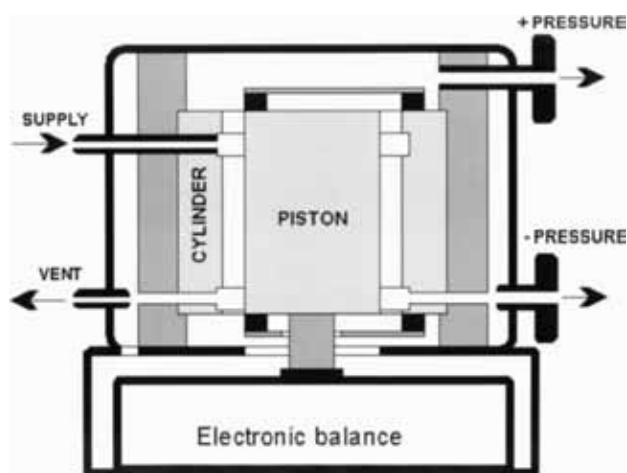
Figure 3: The influence of the uncertainties of all the input quantities on the total uncertainty of the negative gauge pressure generated by a classical pressure balance with an evacuated bell jar

Slika 3: Vpliv negotovosti vseh vstopnih veličin na skupno merilno negotovost negativnega pritiska, ki ga generiramo s klasično batno tlačno tehtnico z evakuirano zvonasto posodo

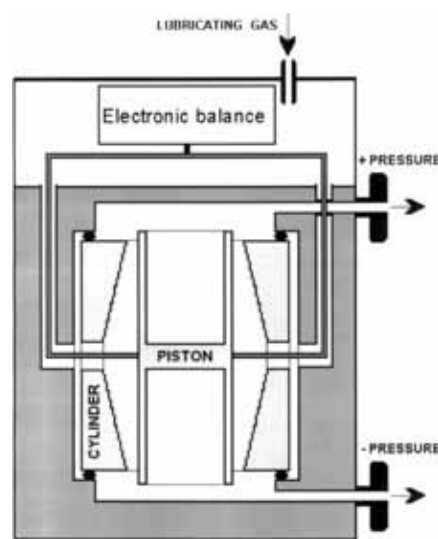
pressure-deformation coefficient of the piston cylinder can be omitted in this pressure range, but the influence of its thermal expansion must be taken into account. The second most important contribution to the total uncertainty is the uncertainty of the mass  $u_M$ . The influence of the uncertainty of the density of the gas under the vacuum bell jar  $u_{\rho_g}$  has been a source of relatively high uncertainty and so pure nitrogen has been introduced as the pressure medium. Its density values are tabulated in <sup>6</sup> with a precision 0.01 %. The influence of the head-pressure uncertainty  $u_{p_h}$  depends on the difference between the reference levels, which is usually very small, but an exaggerated value of 25 cm was used here. The correlations between  $u_{\rho_g}$  and the uncertainty of the acceleration due to gravity  $u_g$  that also appear in the calculation of  $u_{p_h}$  were neglected. The remaining sources of uncertainty are the uncertainties of the mass density  $u_{\rho_M}$  and the sensitivity  $u_s$ . The graph in **Figure 3** shows the influence of the uncertainties of all the input quantities on the total uncertainty of the generated negative gauge pressure  $u_{p_{ng}}$  and compares it with the internationally accepted measurement capability of the Czech Republic  $u_{CMC} = 0.5 \text{ Pa} + 0.0006 \%$  of the measured value in the range from 5 kPa to 100 kPa <sup>7</sup>.

### 3 DIGITAL PRESSURE BALANCES

The measurement of small pressure differences and low absolute pressures using a classical piston manometer with a rotating piston has two basic limitations. First, it is only possible to measure pressures that are high enough to balance the mass of the piston (a few kilopascals). Second, the periodic pressure fluctuations caused by rotation of the piston that have amplitudes of the order of tenths of a pascal are an important source of uncertainty in low-pressure measurements. A solution based on the connection of a non-rotating piston to an electronic dynamometer can measure even very small changes of the state of equilibrium of the piston. It also



**Figure 4:** Digital non-rotating piston manometer FRS 4 HR  
**Slika 4:** Digitalni nerotirajoči batni manometer FRS 4 HR



**Figure 5:** Digital non-rotating piston manometer DHI FPG 8601  
**Slika 5:** Digitalni nerotirajoči batni manometer DHI FPG 8601

offers the possibility to use a larger effective area of the piston cylinder.

The laboratory uses an improved, commercially available Furness Controls FRS 4 HR standard able to work in differential, gauge and (reversing the outputs) also negative gauge modes <sup>8</sup>. The nominal area of the piston is 100 cm<sup>2</sup>, and the width of the gap is approximately 30 μm. The electronic balance can be calibrated using an external mass before every measurement. The friction between the piston and the cylinder is prevented by a lever mechanism (**Figure 4**), allowing axial movement of the piston without any contact with the surface of the cylinder.

The flow of dry air enters the upper part of the cylinder during the measurement. This air then flows through the gap and exits through the outputs in the base. The system was highly sensitive to small instabilities of atmospheric pressure. It was therefore decided to use mass-flow controllers in the input and output instead of the flow-controlling unit in order to insulate the instrument's ports from the ambient and to stabilize and control the gas flow (i.e., the pressure difference).

This instrument has made it possible to ensure the etalonnage of the negative gauge pressure in the range from 1 Pa to 3.2 kPa. The internationally accepted measurement capability of the Czech Republic  $u_{CMC} = 0.01 \text{ Pa} + 0.004 \%$  of the measured value <sup>7</sup>.

**Figure 5** introduces the DHI FPG 8601 manometer <sup>9-12</sup>. It is based on a non-rotating tungsten-carbide piston 10 cm<sup>2</sup> placed in a tungsten-carbide cylinder, where the gap has a 1-to-6 μm conical profile. The piston is centred by means of a lubricating gas with a pressure that is about 40 kPa higher than the reference pressure and is balanced by a mass comparator (electronic balance). The pressure and the gas flow are regulated by a control unit equipped with two flowmeters (for coarse and fine adjustments), and also a PC. The attainable uncertainty

in the negative gauge mode in the range from 1 Pa to 15 kPa is  $u_{p_{ng}} = 0.01 \text{ Pa} + 0.0014 \%$  of the measured value.

This instrument was evaluated using primary methods and compared with a classical pressure balance, both in gauge (evaluating number  $E_n$  up to 0.4) and in absolute ( $E_n$  up to 0.3) modes. Both the digital non-rotating piston pressure balances were also compared mutually in the gauge mode ( $E_n$  up to 0.6).

#### 4 ABSOLUTE PRESSURE DIVIDER

The principle of this standard is the same as in the classical pressure divider<sup>13</sup>. It is based on three concentric pistons (A, B and C). The effective area of the piston B is 101-times larger than that of the pistons A and C (Figure 6). The pressure generated by an oil-piston manometer is connected to the base of piston C. Due to the ratio of the effective areas of the piston cylinders of the divider, the increase in the pressure above piston B is nominally 100-times lower than the increase in the hydraulic pressure under piston C. There is a vacuum pump connected via an insulating valve to the lower chamber beneath piston B. The device under test is also connected to the lower chamber.

The DH-Budenberg 1600 absolute pressure divider can be used to generate absolute, gauge, negative gauge and differential (at line pressures other than atmospheric) pressures. The work in the negative gauge mode is as follows. First, the spaces under and over the central piston B are opened to the atmosphere. Such masses must be put on the oil-piston manometer in order to generate an appropriate pressure with a magnitude of approximately 1 MPa, acting on the base of piston C and compensating for the gravity of the rotating piston system. After reaching an equilibrium state of the

oil-piston standard and the divider, the valve connecting the lower chamber to the atmosphere can be closed. Now, an additional mass must be placed onto the oil-piston manometer to generate an increase in the hydraulic pressure 100-times higher than is the demanded negative gauge pressure value. This will increase the pressure upwards acting on the base of piston C, i.e., on the piston system of the divider, and raise this system in the upper stroke. Then the lower chamber is evacuated until the demanded negative gauge pressure is reached. Consequently, the vacuum pump is disconnected by the valve and the hydraulic pressure is trimmed in order to ensure the middle floating position of the piston system of the divider.

Since the effective area of piston B is nominally 101-times larger than that of pistons A and C, the ratio of the hydraulic and the differential pressure (dividing ratio) is nominally:

$$R_D = \frac{\Delta p_o}{p_{ng}} = \frac{A_B - A_C}{A_C} = \frac{101-1}{1} = 100 \quad (3)$$

where:

$\Delta p_o$  is the change in hydraulic pressure

$A_C$  is the effective area of piston C

$A_B$  is the effective area of piston B

The hydraulic pressure at the time of the initial equilibrium is defined as:

$$p_o = \frac{m \left( 1 - \frac{\rho_{a0}}{\rho_m} \right) + \sigma c}{A_o(p_o, T_{o0})} + h \rho_o g \quad (4)$$

where:

$m$  is the total applied mass by initial equilibrium

$\rho_m$  is the mean density of the piston and loaded masses

$A_o$  is the effective area of the hydraulic pressure piston cylinder

$T_{o0}$  is the temperature of the hydraulic pressure piston cylinder during initial equilibrium

$\rho_{a0}$  is the density of ambient atmosphere during initial equilibrium

$c$  is the piston circumference at its exit from the oil

$\sigma$  is the surface-tension coefficient of the oil

$\rho_o$  is the density of the oil

$h$  is the height of the piston base above the reference level

The change in the hydraulic pressure (caused by the additional mass  $m_p$ ) can be written as:

$$\Delta p_o = \frac{(m_p + m) \left( 1 - \frac{\rho_a}{\rho_m} \right) g + \sigma c}{A_o(p_o, T_{o0} + \Delta p_o)} - \frac{m \left( 1 - \frac{\rho_{a0}}{\rho_m} \right) g + \sigma c}{A_o(p_o, T_{o0})} \quad (5)$$

where  $\rho_a$  is the density of the ambient atmosphere and  $T_o$  is the temperature upon generating the differential pressure.

The generated negative gauge pressure is:

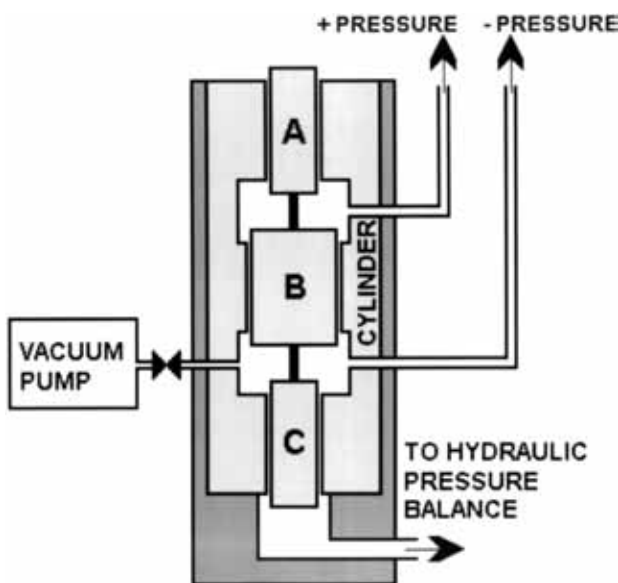


Figure 6: Principle of the absolute pressure divider  
Slika 6: Prikaz delovanja absolutnega delilnika tlaka



$$P_{ng} = \frac{1}{R_D} \left[ \frac{(m_p + m) \left(1 - \frac{\rho_a}{\rho_m}\right) g + \sigma c}{A_o(p_o, T_{o0} + \Delta p_o)} - \frac{m \left(1 - \frac{\rho_{a0}}{\rho_m}\right) g + \sigma c}{A_o(p_o, T_{o0})} \right] \quad (6)$$

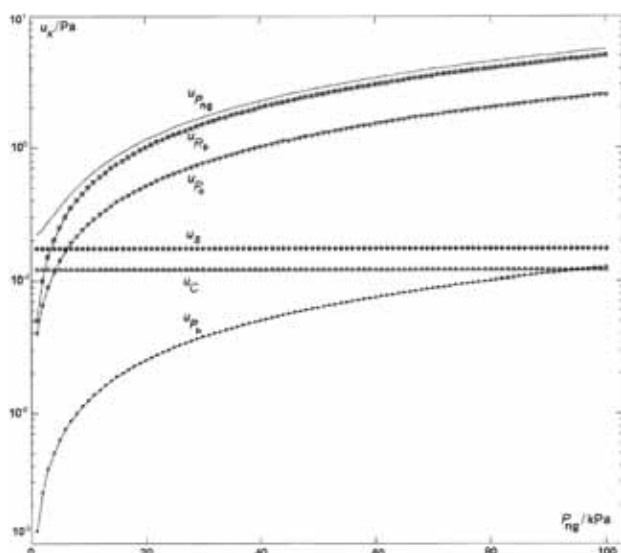
The following notation that simplifies the previous equation is used for the purpose of analysing the uncertainty:

$$P_{ng} = \frac{1}{R_D} [P_o - C] \quad (7)$$

The graph in **Figure 7** shows the influence of the uncertainties of all the input quantities on the total uncertainty of the generated negative gauge pressure  $u_{P_{ng}}$ . The major influences come from the uncertainty of the dividing ratio  $u_{R_D}$  and the component  $u_{R_o}$ , followed by the sensitivity  $u_s$  and the component  $u_c$ . The influence of the head-pressure uncertainty  $u_{p_h}$  depends on the difference between the reference levels (again, an exaggerated value of 25 cm was used). The correlations between  $u_{p_o}$ ,  $u_c$  and  $u_{p_h}$  were neglected. The resultant uncertainty (for negative gauge pressures higher than 1 kPa) can be approximated by  $u_{P_{ng}} = 0.15 \text{ Pa} + 0.0027\%$  of the measured value.

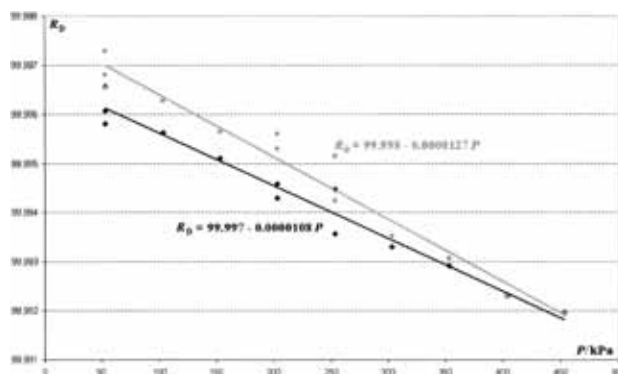
### 5 TRACEABILITY OF THE ABSOLUTE PRESSURE DIVIDER

The traceability of the pressure divider is composed of two items. First, it is the traceability of the hydraulic pressure balance, which is trivial. Second, it is the traceability of the dividing ratio that was performed with the national standard in the gauge pressure (up to



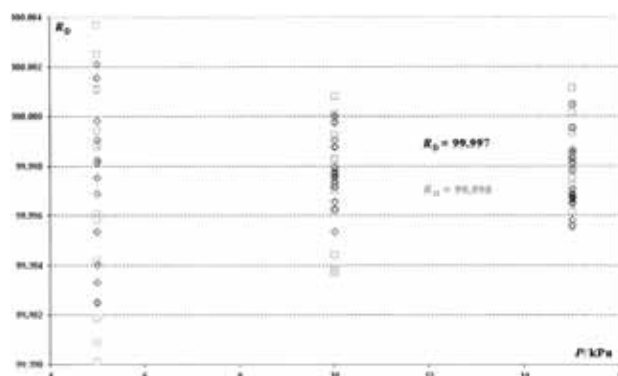
**Figure 7:** Influence of the uncertainties of all the input quantities on the total uncertainty of the negative gauge pressure generated by the absolute pressure divider

**Slika 7:** Vpliv negotovosti vseh vstopnih veličin na skupno merilno negotovost negativnega pritiska, ki ga generiramo z absolutnim delilnikom tlaka



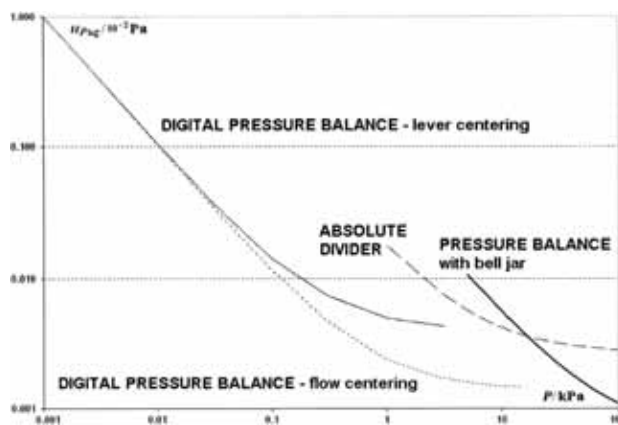
**Figure 8:** Results of the traceability of the dividing ratio in the gauge mode for the years 2007 (diamonds) and 2004 (squares)

**Slika 8:** Rezultati sledljivosti razmerja delitve za relativni tlak v letu 2007 (rombi) in 2004 (kvadrati)



**Figure 9:** Results of the traceability of the dividing ratio in the absolute mode for the years 2007 (diamonds) and 2004 (squares)

**Slika 9:** Rezultati sledljivosti razmerja delitve za absolutni tlak v letu 2007 (rombi) in 2004 (kvadrati)



**Figure 10:** Uncertainties attainable via different methods  
**Slika 10:** Primerjava merilne negotovosti različnih metod

450 kPa, see **Figure 8**). The dividing ratio is, in general, dependent on the pressure, but this can be neglected in the negative gauge mode. The obtained value  $R_D = (99.997 \pm 0.005)$  is in agreement with the value obtained three years before  $R_D = (99.998 \pm 0.005)$ .

In order to be sure that the dividing ratio is not influenced by the working mode its traceability in the absolute mode was also performed. The above-mentioned DHI FPG8601 digital non-rotating piston manometer served as a standard for this (**Figure 9**). The measured pressure points were (5, 10 and 15) kPa. In this case the result was  $R_D = (99.997 \pm 0.007)$ , and three years before it was also  $R_D = (99.998 \pm 0.007)$ .

Furthermore, the comparison in the negative gauge mode between the absolute divider and the classical piston manometer, the digital non-rotating piston manometer with flow centring and the digital non-rotating piston manometer with lever centring was performed. The instruments were separated by a zero indicator (MKS 1 torr Baratron). The comparisons with the digital non-rotating piston-pressure balances gave worse results at the lower ends of their ranges, where the noise is higher. The comparison with the classical piston-pressure balances also gave worse results at the lower end of its range (problems with the free rotation time of the piston), but also at the higher end (problems with the regulation of the pressure). However, the evaluation number was lower than one in each case.

## 6 CONCLUSION

Utilization of the classical piston gauge operating under a bell jar for the etalonnage of negative gauge pressures is a useful and cost-effective solution. **Figure 10** shows a comparison of the attainable uncertainties of all of the mentioned instruments. The Czech Metrology Institute seems to be the only case in the CMC tables covering the negative gauge pressure range using such a principle. The problems it brings have been solved to a satisfactory degree. On the other hand, the use of the absolute divider for this range is much faster, and with only an insignificant (for typical calibrations) decrease in the uncertainty. Moreover, it enables a comparison of

classical and digital pressure balances in the negative gauge range.

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