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Calibration methods for negative gauge pressure down to -100 kPa

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Abstract

The measurement of negative gauge pressure is a field of increasing importance in science and industry. One of the main aims of the EMPIR Joint Research Project 14IND 06 pres2vac is to develop guidelines for it. We present a comparison of three different methods for measuring negative gauge pressure: (i) using two absolute pressure measuring instruments, (ii) generating a negative gauge pressure in the bell jar of the pressure balance, and (iii) using a ‘hanging piston’ pressure balance. The advantages and drawbacks are discussed and some preliminary results presented.

Keywords: negative gauge pressure, piston pressure gauge, differential pressure, pressure calibration, barometer, manometer calibration


(Some figures may appear in colour only in the online journal)

Introduction

The measurement of negative gauge pressure is an area of increasing importance for many scientific and industrial applications (clean room technology, petrochemical and pharmaceutical production, storage of nuclear and toxic waste). As demands on accuracy increase, the calibration of measuring instruments needs to be treated more carefully. Methods for absolute and gauge pressure measurement are well established and their uncertainty budgets well understood. However, when the same methods are applied to negative gauge pressure measurement, atmospheric pressure fluctuations can affect the calibration significantly, and the associated uncertainties are often underestimated. One of the principal aims of the EMPIR Joint Research Project 14IND06 pres2vac is to address this issue. Indeed, one of the deliverables of the project is a calibration guide for this measurement mode. Here we present a comparison of several methods for weather-independent

negative gauge pressure measurement based on the use of pressure balances (as primary standards) and digital manometers (as secondary standards). Three variations of the pressure balance method have been studied and their advantages and drawbacks highlighted: (i) using two absolute pressure measuring instruments (Method 1), (ii) generating a negative pressure in the bell jar of the pressure balance (Method 2), and (iii) using a ‘hanging piston’ pressure balance (Method 3). Method 1 is accurate but requires two instruments and so is more expensive. Method 2 requires only a single piston gauge, but the stabilization time for the balance can be up to several minutes for each pressure measurement point. Method 3 requires a purpose-built instrument.

In the simplest way of implementing these methods, the lowest attainable negative pressure is limited either by the ambient atmospheric pressure (decreasing with altitude) or by the limited measuring range of the reference instrument itself. To circumvent the constraint of low atmospheric pressure, it is often advantageous to replace the real atmospheric pressure by a pressure close to it generated and stabilised in an auxiliary volume. For the best uncertainty, this closed volume is thermally insulated. In this way, the uncertainty obtained is

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typically $0.1 \text{ Pa} + 1.10^{-5} \times |P|$. For secondary laboratories, it is sufficient to use a digital manometer, providing one follows strictly a protocol whereby ambient conditions are well specified. In such cases, it is possible to achieve an uncertainty of $2 \text{ Pa} + 1.10^{-4} \times |P|$ without recourse to a pressure balance. This fact should have an impact on the widespread calibration of gauges at this level of accuracy. Indeed, so far it was thought that two gauges were required to achieve such a level of accuracy. When only a single gauge is used, measurement cycles can be performed far more quickly and easily, saving time and costs. Results for all three types of negative gauge pressure measurements are presented below.

1. Methods for accurate and weather-independent calibration of negative gauge pressure

Contrary to absolute pressure calibration for which the reference pressure is vacuum, gauge pressure is referred to ambient atmospheric pressure. This difference has an impact on the method used to perform calibration. In the case of absolute pressure measurement, the vacuum is stable and no specific requirement is needed while gauge pressure measurement, on the other hand, is sensitive to fluctuations of ambient conditions. Most instruments used in gauge pressure measurements have the possibility to compensate atmospheric pressure change by means of a reference port. However, during calibration it is necessary to ensure that both standard and device under test have the same reference pressure and are subjected to the same variation. When instruments have reference ports, one obtains the best results by connecting these reference ports directly.

In addition, the possibility to measure negative gauge pressure (P_{neg}) also depends on the value of atmospheric pressure value due to the fact that, by definition, the maximum negative gauge pressure applicable cannot be higher than atmospheric pressure and with most instruments it is difficult to perform measurements beyond -95 kPa . When atmospheric pressure is less than 100 kPa or if the laboratory is located at a high altitude, calibration becomes impossible. To reduce these constraints, one of the activities of the Joint Research Project pres2vac consists in the development of methods that are independent of the value of atmospheric pressure and its fluctuations. Two ways have been studied. The first was by using a variable volume in which the pressure can be adjusted. This volume is connected to the reference ports of the standard and instrument to be calibrated. The second method shown in figure 1 was developed by the Czech metrology institute (CMI) and consists in the use of a hermetic chamber large enough to enclose both the standard and device under test.

This chamber can be used when one of the devices has no reference port and cannot therefore be connected to a reference volume. In addition, for uncertainties below $2.10^{-5} \times |P_{\text{neg}}|$, thermal insulation can be added to reduce ambient pressure fluctuations due to temperature changes.

Studies of pressure stability have been carried out using volumes with capacities of 0.5 litre , 1 litre and 2 litres . Results have shown that variations of pressure inside these



Figure 1. Hermetic chamber developed by CMI in which the standard instrument and device under test can be enclosed. A handle allows one to clamp the closing cover. Leaktight feedthroughs are used for pressure and electrical connections.

volumes are below 0.1 kPa when the laboratory temperature is regulated to within $\pm 1 \text{ }^\circ\text{C}$.

2. Description of methods for negative gauge pressure calibration

In practice, in pressure metrology, the use of a pressure balance is required to achieve calibration uncertainties around $10^{-5} \times |P_{\text{neg}}|$. In the case of negative pressure, only ‘hanging piston’ pressure balances are able to measure these pressures directly in gauge mode. Although this type of instrument is set up for negative gauge pressure measurement, it does not allow one to achieve the same uncertainty level as with pressure balances used for positive gauge pressure. The main reason for this is the deterioration of its performance with increase of the negative gauge pressure. In order to ensure the continuity of uncertainties between positive and negative pressure measurements, national metrology institutes have developed alternative methods using pressure balances in absolute mode, one by using two absolute pressure instruments, the second by generating a negative pressure in the bell jar of the pressure balance. All three methods are described below.

2.1. Method 1 calibration by using two absolute pressure measuring instruments

This method uses a pressure balance in absolute mode together with a barometer [1]. The negative pressure P_{neg} is calculated by the difference between the pressure balance reading P_{abs} and the indication of the barometer which is corrected for its accuracy error E_0 :

$$P_{\text{neg}} = P_{\text{abs}} - P_{\text{atm}} \quad (1)$$

where

$$P_{atm} = P_m + E_0. \quad (1a)$$

Atmospheric pressure P_{atm} is given by the indication of the barometer P_m corrected for the measurement error E_0 . This correction is determined around atmospheric pressure P_{m0} using the same absolute pressure balance:

$$E_0 = P_{abs0} - P_{m0} \quad (2)$$

where P_{abs0} is the absolute pressure delivered by the balance at the time the error is determined.

This error must be determined before and after calibration in order to check the short-term stability of the barometer. For the best barometers, the stability between these two determinations is typically around 0.1 Pa.

The negative gauge pressure is then given by the combination of equations (1) and (2):

$$P_{neg} = P_{abs} - P_m - P_{abs0} + P_{m0}. \quad (3)$$

The absolute pressure delivered by the balance at the reference level of the device under test (DUT) is given by [2]

$$P_{abs} = \frac{\sum_i [m_i \cdot g]}{A_0 [1 + (\alpha_p + \alpha_c) \cdot (t - t_{ref})] [1 + \lambda \cdot P]} + \mu + \rho_f \cdot g \cdot \Delta h \quad (4)$$

where the variables are defined as follows:

- m_i : True masses of the piston and the piston loads in kg
- g : Acceleration due to gravity in the laboratory in $m \cdot s^{-2}$
- A_0 : Effective area of piston-cylinder assembly at zero pressure and temperature t_{ref} in m^2
- α_p : Linear thermal expansion coefficient of the piston in $^{\circ}C^{-1}$
- α_c : Linear thermal expansion coefficient of the cylinder in $^{\circ}C^{-1}$
- t : Temperature of piston-cylinder assembly in $^{\circ}C$
- t_{ref} : Reference temperature of piston-cylinder assembly in $^{\circ}C$, usually $20^{\circ}C$
- λ : Deformation coefficient of piston-cylinder assembly in Pa^{-1}
- P : Nominal value of measured pressure P_{abs} in Pa
- μ : Residual pressure in the bell jar in Pa
- ρ_f : Density of the fluid at the measured pressure in $kg \cdot m^{-3}$
- Δh : Height difference between the pressure balance and the DUT in m.

When the measurement error of the barometer is determined, the pressure P_{abs0} delivered by the pressure balance is

$$P_{abs0} = \frac{\sum_i [m_{i0} \cdot g]}{A_0 [1 + (\alpha_p + \alpha_c) \cdot (t_0 - t_{ref})] [1 + \lambda \cdot P_0]} + \mu_0 + \rho_{f0} \cdot g \cdot \Delta h \quad (5)$$

where m_{i0} , t_0 , P_0 , μ_0 and ρ_{f0} have the same meaning as the symbols without index '0' but refer to the time point of the barometer calibration.

When the barometer is installed at the same level as the device under test, the negative pressure is given by a combination of equations (3)–(5):

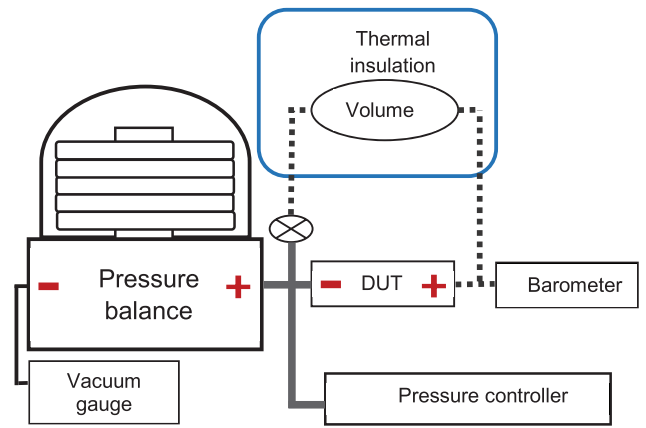


Figure 2. Set-up for negative gauge pressure calibration using two absolute pressure measuring instruments: the negative pressure to be measured is applied to the ‘-’ port of the DUT and to the ‘+’ port of the pressure balance. To measure the residual pressure in the bell jar, the vacuum gauge is connected to the ‘-’ port. The barometer is connected to either the ‘+’ port (reference port) of the DUT or to ambient pressure.

$$P_{neg} = \frac{\sum_i [m_i \cdot g]}{A_0 [1 + (\alpha_p + \alpha_c) \cdot (t - t_{ref})] [1 + \lambda \cdot P]} - \frac{\sum_i [m_{i0} \cdot g]}{A_0 [1 + (\alpha_p + \alpha_c) \cdot (t_0 - t_{ref})] [1 + \lambda \cdot P_0]} + \mu - \mu_0 - P_m + P_{m0} + (\rho_f - \rho_{f0}) \cdot g \cdot \Delta h. \quad (6)$$

At constant temperature, this method offers the advantage of eliminating the error due to the vacuum gauge and low mass load.

Figure 2 below shows the set-up necessary to calibrate a DUT in negative gauge pressure mode. The DUT ‘-’ port and a pressure controller are connected, while a vacuum gauge is connected to the ‘-’ port of the pressure balance. To read the ambient pressure, a barometer is connected to the ‘+’ port of the DUT. This barometer must be located at the same height level as the DUT to avoid one’s having to apply a supplementary head level correction. By adding a volume, whose temperature can be regulated, one can control the atmospheric pressure. This is particularly advantageous in the case of low atmospheric pressure (below 95 kPa) encountered in laboratories at altitude. When using a piston-cylinder assembly of area 10 cm^2 , one should apply a pressure of 105 kPa to perform the measurement point at -95 kPa because the minimum absolute pressure point is 10 kPa. This volume must be connected directly to the barometer and the ‘+’ port of the DUT as well as to the pressure controller using a valve.

To obtain the best measurement uncertainties, one must respect different steps which are necessary to eliminate the contribution of additional parameters due to the method in the uncertainty budget (vacuum and pressure measurements, low mass load and head level).

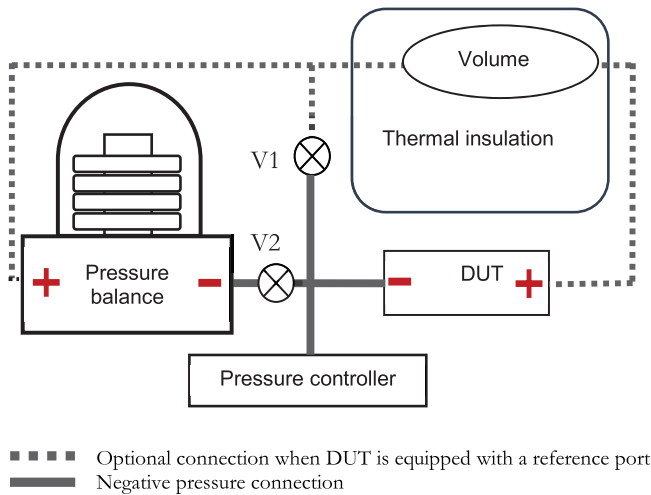


Figure 3. Set-up for negative gauge pressure calibration by applying sub-atmospheric pressure to the bell jar. Negative pressure to be measured is applied to the ‘-’ port of the DUT and to the ‘-’ port of the pressure balance which is connected to the bell jar. The ‘+’ ports of the DUT (reference port) and of the pressure balance are connected together to a volume or to ambient pressure.

The first step consists of determining the measurement error E_0 of the barometer and performing zeroing of the DUT at the reference, almost atmospheric pressure. For this, the valve between volume and the pressure controller is opened and a pressure of 105 kPa is applied using the pressure balance. After stabilization, knowing the temperature of piston-cylinder assembly, residual pressure μ_0 and mass applied on the pressure balance one calculates the pressure P_{abs0} according to (5), the indication of barometer is noted and its measurement error E_0 is calculated using equation (2). The zeroing of the device under test has to be performed at this moment, before the valve is closed.

From this time onward, the valve must remain closed until the end of the calibration unless another zero check of the DUT has to be performed between two measurement cycles. In this case, the reference pressure delivered by the balance has to be stabilized before opening the valve and performing the zero check. The reference pressure inside the volume, close to atmospheric pressure, should vary by no more than 0.1 kPa.

For each measurement point, the absolute pressure corresponding to the negative pressure to be applied is given by the relation:

$$P_{abs} = P_{neg} + P_{atm}. \quad (7)$$

At the end of calibration, the last step consists in checking the short-term stability of the barometer. For this, the reference pressure is generated by the balance and after stabilization the valve can be opened to connect the pressure balance to the volume. As in the first step, the measurement error of the barometer is calculated. The final measurement error E_0 is calculated from the mean of the two determinations. For the best instruments, the deviation between the two values is typically about 0.1 Pa.

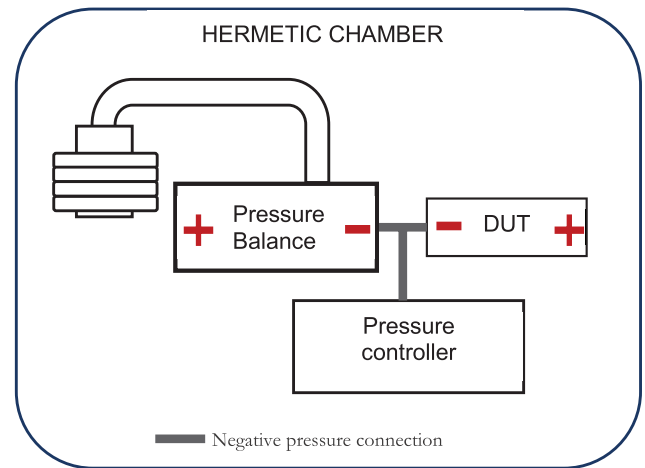


Figure 4. Set-up for calibration using a piston-cylinder assembly mounted upside-down. The negative pressure to be measured is applied to the ‘-’ port of the DUT and to the ‘-’ port of the pressure balance. The ‘+’ ports of the DUT (reference port) and of the pressure balance are both at ambient pressure.

2.2. Method 2 calibration by applying negative pressure to the bell jar of the balance

This method is based on the use of an absolute pressure balance [3] whose measurement port is open to atmosphere and the pressure in the bell jar represents the negative gauge pressure. Unlike the usual mode of operation, here the DUT ‘-’ port is connected to the ‘-’ port of the pressure balance, which is connected to the bell jar volume where the negative pressure is generated. As shown in figure 3, the ‘+’ port of the balance and the ‘+’ port of the DUT are connected to be independent of ambient pressure fluctuations. If desired, a thermally insulated volume can be used to provide a better stability of the reference pressure than that of the bare laboratory.

Regardless of which mode the pressure balance is used, the pressure P_{bal} generated at the ‘+’ port is calculated by the relation:

$$P_{bal} = \frac{\sum_i \left[m_i \left(1 - \frac{\rho_f}{\rho_{m_i}} \right) \cdot g \right]}{A_0 [1 + (\alpha_p + \alpha_c) \cdot (t - t_{ref})] [1 + \lambda \cdot P]} + \mu + (\rho_a - \rho_f) \cdot g \cdot \Delta h \quad (8)$$

where ρ_{m_i} is density of the mass placed on the piston, in $\text{kg} \cdot \text{m}^{-3}$, ρ_a is air density of in $\text{kg} \cdot \text{m}^{-3}$, and all other symbols as defined above.

In normal use, the ‘-’ port of the pressure balance corresponds to the residual pressure in absolute mode or atmospheric pressure in gauge mode. In negative gauge pressure measurement using this method, the two ports are inverted as well as the flow direction in the piston-cylinder gap. The standard negative pressure is measured in the bell jar. Taking into account that $P_{bal} = P_{atm}$ and $P_{neg} = P_{atm} - \mu$, from equation (7), one obtains

$$P_{neg} = - \frac{\sum_i \left[m_i \left(1 - \frac{\rho_f}{\rho_{m_i}} \right) \cdot g \right]}{A_0 [1 + (\alpha_p + \alpha_c) \cdot (t - t_{ref})] [1 + \lambda \cdot P]} + (\rho_f - \rho_a) \cdot g \cdot \Delta h. \quad (9)$$

Table 1. Uncertainty budget for negative pressure measurement using an absolute pressure balance and a barometer.

Parameter	Estimation method	Uncertainty of quantity x_i	Standard uncertainty $u(x_i)$	Sensitivity coefficient $c_i = \frac{\partial P_{\text{neg}}}{\partial x_i}$	Standard uncertainty $u_i(P_{\text{neg}}) = c_i \cdot u(x_i)$
Repeatability	Five determinations				0.080 Pa
Mass. including stability	Calibration	7.5 mg/5 kg	$0.75 \cdot 10^{-6} \cdot m$	$P_{\text{neg}} / (m - m_0)$	$0.75 \cdot 10^{-6} \cdot P_{\text{neg}} $
Effective area	Dimensional measurements	$0.1 \mu\text{m}$	$2.8 \cdot 10^{-6} \cdot A_0$	$-P_{\text{neg}} / A_0$	$2.8 \cdot 10^{-6} \cdot P_{\text{neg}} $
Effective area stability	30 year history		$3.0 \cdot 10^{-6} \cdot A_0$	$-P_{\text{neg}} / A_0$	$3.0 \cdot 10^{-6} \cdot P_{\text{neg}} $
Distortion coefficient	Calculation	10%	$0.04 \cdot \lambda$	$-P_{\text{neg}} \cdot (P_{\text{neg}} + 2P_{\text{abs},0})$	$2.1 \cdot 10^{-8} \cdot P_{\text{neg}} $
Temperature	Pt100 probe + ohmmeter	$0.05 \text{ }^\circ\text{C}$	$0.05 \text{ }^\circ\text{C}$	$-(\alpha_p + \alpha_c) \cdot P_{\text{neg}}$	$0.45 \cdot 10^{-6} \cdot P_{\text{neg}} $
Temperature coefficient	Negligible				
Residual pressure	Measured	0.005 Pa	0.005 Pa	1	0.005 Pa
Verticality	Estimation (spirit level)	0.00029 rad	0.00017 rad	$\tan\theta \cdot P_{\text{neg}}$	$0.028 \cdot 10^{-6} \cdot P_{\text{neg}} $
Reference level including piston position	Ruler	3 mm	1.0 mm	$\rho_{\text{N}_2} \cdot g$	$0.09 \cdot 10^{-6} \cdot P_{\text{neg}} $
Gravity	BRGM measurement (Bureau de Recherches Géologiques et Minières)		$5 \cdot 10^{-8} \cdot g$	P_{neg} / g	$0.05 \cdot 10^{-6} \cdot P_{\text{neg}} $
Atmospheric variation	Measured	0.05 Pa	0.05 Pa	1	0.05 Pa
Barometer resolution		0.1 Pa	0.029 Pa	1	0.029 Pa
Expanded uncertainty ($k = 2$)					$0.20 \text{ Pa} + 6.6 \cdot 10^{-6} P_{\text{neg}}$
Expanded uncertainty ($k = 2$) Method 2					$0.10 \text{ Pa} + 7.0 \cdot 10^{-6} P_{\text{neg}} $
Expanded uncertainty ($k = 2$) Method 3					$0.16 \text{ Pa} + 1.0 \cdot 10^{-5} P_{\text{neg}} $

Note that the calculation for apparent mass depends on the density of the air in contact with masses. The correction for apparent mass involves the density of the fluid and not that of air.

As shown in figure 3, a volume can be added to adjust the atmospheric pressure. For this, two valves have to be installed: one between the pressure controller and the volume, the second one placed at the ‘-’ port of the pressure balance. Before starting a calibration, one must adjust the reference atmospheric pressure in the volume by closing valve V2 and opening valve V1. The zero of device under test is performed at this time. Valve V1 is closed and valve V2 is opened to begin a measurement cycle.

For each measurement point of a cycle, negative pressure is applied to the bell jar; the volume to be pumped is large and difficulties to achieve the desired pressure can appear from -50 kPa onwards. An additional pump can be used with a bypass valve in order to approach the target pressure more quickly before adjusting it with a pressure control. Where negative pressure is close to atmospheric pressure, the bell jar can sometimes leak around the seal. To prevent this, a mass can be placed on top of the jar.

2.3. Method 3 negative pressure calibration using piston-cylinder mounted upside-down

The so-called ‘hanging piston’ method is used in some laboratories. For this one needs to have a pressure balance specially built for negative pressure mode. As shown in figure 4, an inverted cane is mounted.

To achieve equilibrium of the piston, masses have to be raised by suction, i.e. a negative pressure. In contrast to the previous method, the pressure balance is used in gauge

mode, and its reference pressure as well as the piston with the loading mass pieces are subject to atmospheric pressure. In this set-up, the ‘-’ ports of the DUT and of the pressure balance are connected to the pressure controller, while the ‘+’ ports are at the reference atmospheric pressure. The control of atmospheric pressure cannot be performed using a volume but it is possible to place this balance and the DUT under test in a hermetic chamber.

When the equilibrium of the pressure balance is achieved, the standard negative pressure is given by

$$P_{\text{neg}} = - \frac{\sum_i \left[m_i \left(1 - \frac{\rho_a}{\rho_{m_i}} \right) \cdot g \right]}{A_0 [1 + (\alpha_p + \alpha_c) \cdot (t - t_{\text{ref}})] [1 + \lambda \cdot P]} + (\rho_f - \rho_a) \cdot g \cdot \Delta h. \quad (10)$$

It is important to note the differences between equations (9) and (10) concerning the density of air. Here, the mass correction due to air buoyancy is calculated with the density of air at ambient pressure.

3. Uncertainties related to negative pressure measurements

These three methods for negative pressure measurements are based on the use of pressure balances and their uncertainty budgets are subject to the same parameters. If, for most of them, the contributions to the uncertainty budget are the same, a number of them depend on the method used. Table 1 shows all the parameters with estimation methods [4], uncertainty of the quantity, standard uncertainty and sensitivity coefficient.

Here values correspond to the best attainable uncertainty at LNE/LCM for the method using absolute pressure balance and barometer [5]. Parameters specific to this method are detailed below.

3.1. Uncertainty due to mass

In normal use of a pressure balance, the uncertainty due the mass corresponds to that of all the masses loaded, including the piston mass and that of the mass carrying bell. In this case, regarding equation (6), this is the mass difference $m - m_0$ between the total mass loaded at measurement point and the masses loaded at the time the measurement error E_0 is determined. In this way, mass contributions that are taken into account in the uncertainty budget have the same value as for the usual methods but their decomposition is different. For example, when a negative pressure of -20 kPa is applied using a pressure balance equipped with a piston-cylinder assembly of 10 cm^2 area, 2 kg are loaded with the usual method whereas 8 kg are loaded in the pressure balance and barometer method. In the uncertainty calculation, these 8 kg are deducted from the 10 kg loaded at the time the accuracy error of the barometer is calculated (atmospheric pressure at 100 kPa). Note that, using this method, errors due to low mass load are eliminated and only the highest masses, for which the relative uncertainties are lower, contribute to the uncertainty.

3.2. Uncertainty due to piston density

The masses used with pressure balances have a density close to $8000\text{ kg} \cdot \text{m}^{-3}$ so as to limit corrections when apparent masses are calculated. However, materials such as tungsten carbide for the piston and aluminium for the bell jar are preferred due to the fact that pistons need suitable physical properties (low thermal expansion coefficient, high hardness) while low-density materials for bells are a good way to minimize the minimum applicable pressure. When the traditional method is used, an uncertainty contribution of $1 \cdot 10^{-3} \times \rho_{mi}$ is taken into account. This contribution is eliminated when one uses the method with an absolute pressure balance and a barometer.

3.3. Uncertainty in the distortion coefficient

The distortion coefficient λ of the $10\text{ kPa} \cdot \text{kg}^{-1}$ piston-cylinder for free deformation is $4.2 \cdot 10^{-6} \cdot \text{MPa}^{-1}$. The uncertainty of this coefficient is $\pm 10\%$ with a coverage factor of 2 [6]. For the maximum pressure, which is 0.1 MPa , the standard uncertainty in pressure is estimated to be $2.1 \cdot 10^{-8} \cdot |P_{\text{neg}}|$.

3.4. Uncertainty due to the temperature

The standard uncertainty in the temperature measurement is estimated to be $0.1\text{ }^\circ\text{C}$. According to equation (6), the contribution to the temperature measurements is approximated by the uncertainty on the difference $t - t_{\text{ref}}$, which is conservatively estimated to be $0.05\text{ }^\circ\text{C}$. In addition, as the remaining temperature uncertainty is the difference $t - t_{\text{ref}}$, the uncertainty of the thermal expansion is negligible.

3.5. Uncertainty due to residual pressure

If residual pressure does not contribute to the uncertainty budget in gauge mode, it is taken account in the absolute

pressure measurement. This pressure is measured with a capacitive gauge (MKS-Baratron⁵) for which the standard uncertainty is equal to 0.1 Pa . Regarding equation (6), with this method, the term $\mu - \mu_0$ allows one to eliminate the error due to the residual pressure measurement. However, the difference between μ and μ_0 has to be considered; this value never exceeds 1.0 Pa during the calibration. The uncertainty for this difference is estimated to 0.005 Pa and contributes to the uncertainty budget of negative pressure measurement.

3.6. Uncertainty due to atmospheric pressure measurement

The uncertainty of this difference is function of the resolution (0.1 Pa) and the short-term stability of the precise barometer.

With the use of a volume, which can be regulated in temperature, typical pressure difference $P_m - P_{m0}$ during one pressure cycle is less than 100 Pa . The standard uncertainty of this parameter (short-term stability) is estimated to be 0.1 Pa .

3.7. Combined uncertainty

For the method using an absolute pressure balance and a barometer, when the components of each parameter are combined quadratically, the expanded uncertainty ($k = 2$) is expressed as $0.20\text{ Pa} + 6.6 \cdot 10^{-6} \cdot |P_{\text{neg}}|$. If the method by applying negative pressure in the bell jar is chosen using the same balance, the expanded uncertainty ($k = 2$) will be $0.10\text{ Pa} + 7.0 \cdot 10^{-6} \cdot |P_{\text{neg}}|$.

We can note a difference of the offset due to the contribution of the barometer. In use at LNE-LCM these expressions are rounded respectively to $0.20\text{ Pa} + 1 \cdot 10^{-5} \cdot |P_{\text{neg}}|$ and $0.10\text{ Pa} + 1 \cdot 10^{-5} \cdot |P_{\text{neg}}|$. Concerning the method using a 'hanging piston', LNE-LCM does not possess this type of balance but the best expanded uncertainty provided by another European national metrology institute is $0.16\text{ Pa} + 1 \cdot 10^{-5} \cdot |P_{\text{neg}}|$. Similarly to the uncertainty budgets for the methods using an absolute pressure balance with a barometer and measuring the pressure in the bell jar, as also in the method with a 'hanging piston', the pressure-proportional uncertainty contribution is defined by the uncertainty of the effective area and of the masses. From this point of view all three methods should have comparable type-B uncertainty budgets. However, there is a series of experimental, method-specific effects which can significant impact on the combined uncertainty, which are discussed in the following.

4. Comparison of the methods

To validate the capabilities of national metrology institutes to measure negative pressure, several comparisons were organized as a part of Euramet projects [7–9]. In the last one, seven laboratories took part in the comparison and the three methods detailed in this paper were used; four laboratories applied negative pressure in the bell jar, two used the method with an absolute pressure balance and one used the 'hanging piston' method. Even if results show that participants master

⁵ Identification of commercially available instruments in this paper does not imply recommendation or endorsement.

the different calibration techniques, differences in measurement capabilities between laboratories do not allow one to isolate their advantages and drawbacks in terms of the quality of results obtained.

4.1. Implementation of the three methods

As a part of EMPIR project pres2vac, these three methods have been implemented in the Pressure Laboratory at TUBITAK UME, the National Metrology Institute of Turkey for calibration of two types of digital manometers: the first one with high accuracy and the other one principally used in industry. For each instrument, calibration was performed in three cycles of six points.

As the high-accuracy digital manometer a Fluke RPM4⁵ was used whereas the industrial type digital manometer was represented by a G.E. Druck DPI615⁵. Both instruments had a resolution of 0.01 hPa during the measurement cycles and the same pressure balance was used as means of supplying the reference pressure.

In the first two measurement methods proposed, a closed thermally insulated volume had to be used. In order to connect the volume, the device under test should have '+' and '-' port inputs. While the aforementioned pressure connection port is available on the RPM4, an industrial type digital manometer such as the DPI615 has no such ports. There is only one pressure connection port on it, which then breaks the assumed measurement set-up and the reference atmospheric pressure follows the ambient pressure, not the pressure created in the thermally insulated volume. The use of a hermetic chamber as represented in this paper would be a way of circumventing this problem.

In the first method shown in figure 2, a barometer has to be used so a DH Budenberg DPM1⁵ digital pressure monitor was incorporated into the measurement system to record the current absolute reference pressure inside the thermally insulated volume and '+' port of the DUT. In the case of the DPI615 calibration, however, the barometer could not be connected to the DUT since no '+' port is available on it. In this case, the thermally insulated volume became idle and the barometer measured the ambient pressure as the indication (P_m) to be used in equation (1a). The stabilisation time including the evacuation time of the bell jar ranged from 2 to 4 min between successive measurement points in the application of the method where two absolute pressure measuring instruments were used.

The biggest challenge presented itself during the application of the second method proposed for negative gauge pressure calibration by applying sub-atmospheric pressure to the bell jar whose set up is given in figure 3. In the application of this method for RPM4, the exact experimental set-up as depicted in figure 3 was employed, the only addition being the secondary pressure controller and a vacuum pump connected between the negative port of the PG (pressure balance) and valve 2 given by V2 in figure 3. Such a modification was made to decrease the time needed to reach the applied pressure to the set calibration point. The thermally insulated volume was closed to the laboratory atmospheric conditions and the zeroing of the DUT was performed at this pressure.



Figure 5. Set-up for operating piston-cylinder assemblies mounted upside-down.

In the calibration of DPI 615, however, the measurement set-up shown in figure 3 had to be broken due to the fact that DPI 615 has no '+' port that can be connected to the right-hand side of the thermally insulated volume. Therefore, the right-hand side of the volume is blinded at the beginning of the calibrations at the laboratory atmospheric conditions. The results after the calibrations, however, indicated that DUT had errors of up to 1.27 kPa as compared with the reference pressure, which is an anomaly because the device shows no such erroneous behaviour in the calibrations performed by the other methods. A search for the source of this apparent systematic error was made first by repeating the experiment many times, looking for all possible leaks, errors in the calculated pressures etc. The exact reason became clear, however, when a barometer was connected to the right-hand side of the thermally insulated volume in place of the blank flange. Such a replacement of the blank flange with a barometer was a way of detecting any change in the pressure that was in the volume, and by its connection, in the positive port of the PG. It was observed after this replacement of the blank flange with the barometer that the pressure inside the volume and the positive port of the PG indeed decreases with such an amount as to account for the observed systematic anomaly in the pressures measured by the DUT. The reason for this behaviour was suspected to be caused by a flow of air from the positive side below the piston to the bell jar. In the case of the RPM4, the DUT does not suffer from the aforementioned effect possibly due to the fact that both the positive and negative ports of the DUT were communicating with the negative and positive ports of the PG and responding only to the exact difference. In the case of DPI615, however, the DUT always feels the ambient atmospheric pressure on the negative side but the corresponding positive side of the PG suffers from a reduced pressure which cannot be communicated with the DUT. In this method where the negative pressure was applied to the bell jar, stabilisation time rose to 6 min.

The third method implemented at UME was the one similar to the method shown in figure 4, set up for calibration using a piston-cylinder assembly mounted upside-down. The calibrations, however, were performed in laboratory conditions instead of with a hermetic chamber. The reference instrument which

Table 2. Drawbacks and advantages for negative pressure measurements using pressure balances.

	Method 1	Method 2	Method 3 (Commercial instrument)
Time needed to perform a calibration (1 cycle of 12 points)	40 min in manual operation 30 min in automatic operation	60 min	30 min
Facility of implementation	A specific protocol has to be respected	Difficulty to stabilise pressure	Easy
Cost	Very expensive: absolute pressure balance high accuracy barometer	Expensive: absolute pressure balance additional pump	Cheap: only negative gauge pressure balance
Control of ambient conditions	Volume	Volume	Hermetic chamber
Automatization	With commercial software	With customized software	Not applicable
Level of uncertainty	National metrology institute	National metrology institute	Calibration laboratory

is a pressure balance is used in gauge mode. Since the piston is hanging upside-down, the piston-cylinder loaded with the masses provides negative pressure to the DUT. In the set-up used for the reference instrument, there are no ‘+’ and ‘-’ ports available simultaneously. Only a single test port is available as with many industrial-type of references. As a result of such manufacturer construction, reference pressures were applied through a single pressure connection port to both RPM4 and DPI615.

At the German national metrology institute (PTB), a system was built for the realisation of negative gauge pressure measurements using piston-cylinders operated in an upside-down orientation (figure 5). This system equipped with a series of adapters allows almost any of the commercially available piston-cylinder assemblies used for positive gauge or absolute pressure measurements to be calibrated in negative gauge pressure mode too. The system is particularly useful when the same piston-cylinder assembly needs to be calibrated in both positive and negative pressure modes.

4.2. Advantages and drawbacks of the methods

Each method has its advantages and disadvantages that can influence the choice for one laboratory to use one method to the detriment of another according to several criteria.

Indeed, the method using an absolute pressure balance equipped with an automated mass handling system and associated with a barometer (Method 1) requires a greater investment in instrumentation than the other methods but also more rigour when carrying out a calibration. However, it offers various advantages such as the possibility of carrying out fully automated calibrations when the scale so allows. Furthermore, this method allows one to carry out calibrations in negative and positive pressure without having to change the calibration standard or the configuration of the balance. One further important advantage of this method is its capability to realise the reference pressure independently of the ambient pressure. Herewith a calibration over full negative gauge pressure down to -100 kPa is possible even for laboratories located high above the sea level where the ambient pressure is essentially lower than 100 kPa. Moreover, choosing the reference pressure at 105 kPa and operating the pressure balance over the range (5 – 105 kPa) of absolute pressure, in which its performance is appropriate, calibration over the full negative gauge pressure range from 0 to -100 kPa becomes achievable. An important aspect of this method is that the metrological

properties of the pressure balance such as its zero-pressure effective area and the pressure distortion coefficient defined for absolute pressure mode are valid when measuring negative pressures, which appropriately addresses the question of traceability. However, along with high instrumental requirements and costs, one of the drawbacks of this method is its applicability to calibration of electromechanical manometers only. Calibration of another pressure balance operated in the negative gauge pressure mode, such as a balance with a ‘hanging piston’, is hardly possible.

Regarding the method of applying the negative pressure in the bell jar (Method 2), we have seen that it provides quite low uncertainties but has many disadvantages. Indeed, at pressures below -50 kPa, an additional vacuum pump with a bypass valve is necessary to reach the desired pressure, because of the high volume subjected to suction. Moreover, at pressures close to atmospheric pressure, the equilibrium of the balance often becomes difficult because of leaks that may appear at the seal of the bell. These difficulties will be more significant if the instrument to be calibrated is devoid of a reference port which can allow the stabilization of the atmospheric pressure. As for traceability, the zero-pressure effective area of the reference pressure balance corresponds to that of the gauge mode. Concerning the pressure distortion coefficient, even though its effect is rather smaller, its value in the negative gauge pressure measurement is not the same as in the positive gauge or absolute mode. This is related to the fact that the load of the cylinder outside changes with pressure when operated in the negative gauge mode, whereas in the positive gauge and absolute mode it stays constant. As long as the distortion coefficient is used in the pressure equation—it might be omitted because the effect of the distortion coefficient is small—the change of the coefficient between the operation modes might need to be considered. Using a reference volume with a controlled reference, this method can also be applied by laboratories in elevated regions. The highest negative gauge pressure of this method is limited to about -5 kPa, which is one of its drawbacks. Also the lowest theoretical negative gauge pressure of -100 kPa cannot be reached, a fact related to the rapidly increasing piston fall rate when approaching zero absolute pressure in the bell jar. Indeed, the piston fall rate (v_f) is given by the equation

$$v_f = -\frac{Q_m}{\pi r^2 \rho(P)} \quad (11)$$

in which Q_m is the fluid mass flow rate through the piston-cylinder gap, r the piston radius and $\rho(P)$ the fluid density at pressure P , which is to be taken for the volume which has no fluid exchange with the outside. For gauge and absolute operation modes, this is the volume and absolute pressure P below the piston. For negative gauge pressure operation, the volume above the piston is relevant, and P is absolute pressure in the bell jar. At a negative gauge pressure equal to -100 kPa, the absolute pressure in the bell jar is zero and the piston fall rate must be infinite. At a negative gauge pressure of -95 kPa, the piston fall rate is about 20 times higher than those when measuring an absolute pressure of 100 kPa in absolute operation mode. This shows the problems one meets when measuring low negative pressure in the bell jar. With increasing piston fall rate, first, a viscous force between the piston and cylinder has impact on piston load and, consequently, pressure, and, second, the piston quickly leaves its working position which requires its re-adjustment. The problem could be reduced by removing the gas flowing into the bell jar by means of an appropriate pumping system, but this would require additional instrumentation and could have impact on the pressure homogeneity between the pressure balance and the DUT. Like Method 1, Method 2 is also suitable for calibration of electro-mechanical manometers but can hardly be used for calibration of negative gauge pressure piston gauges.

The method with a 'hanging piston', Method 3, is by far the easiest to implement and the fastest for calibration. Along with this, another advantage of this method over Methods 1 and 2 is its capability to calibrate another 'hanging piston' pressure balance in negative gauge mode directly. For instance, in Germany, there are several laboratories accredited for negative gauge pressure which use the 'hanging piston' gauge as a reference. According to legal regulations, all these reference standards must be calibrated in negative gauge mode which, based on pressure balances, is possible only by Method 3. As for the characteristics of the piston-cylinder assembly, temperature stability, masses loaded on the piston, etc. Their uncertainty contributions are principally the same as for Methods 1 and 2. However, as in these methods, there are features specific to Method 3 which can have impact on the measurement uncertainty. Traceability of the zero-pressure effective area of the piston gauge operated in negative gauge mode is realised through a calibration in positive gauge mode. As pressure balance theory shows [2], the zero-pressure effective area stays the same when the piston-cylinder is inverted. As for the pressure distortion coefficient, its value in the normal and inverted orientation is not the same because of different boundary conditions and also a possible asymmetry of the piston-cylinder gap profile over the middle of the piston-cylinder sealing length. However, as the effect of the pressure distortion coefficient in this pressure range is negligibly small, a variation of this coefficient has no notable effect on the uncertainty. Similarly to Method 2, measurement of very low negative pressure by this method is affected by increasing piston fall rate as explained above, see equation (11). Also, the lowest negative pressure is, as

in Method 2, limited by the minimum mass of the piston and its load at which the piston gauge operates properly. One of the method's drawbacks is related to the fact that it is hardly possible to provide a reference pressure other than an atmospheric one and stabilised, which can have impact when calibrating indicating instruments. An isolation of the reference piston gauge and the DUT from the ambient pressure would in principle be possible using a sufficiently large hermetic chamber like those shown in figure 1, but this would require implementing a sophisticated, remotely-operated piston loading system, which appears impractical.

The main drawbacks and advantages of each method are summarised in table 2.

5. Conclusions

This paper has presented three different methods for negative gauge pressure calibration, all three based on pressure balances as the reference. Even if these methods allow uncertainties to be reached that allow the connection of secondary standards which can ensure traceability to industry, they differ greatly in terms of cost, difficulty of implementation, time required to carry out a calibration, calibration ranges, requirements to ambient conditions and types of instruments that can be calibrated. We have also shown that, with two of the methods, it is possible to carry out calibrations whatever the ambient conditions by circumventing the constraints related to the instability of the ambient atmospheric pressure by means of either a closed volume or a hermetic chamber. While all three methods can be used to calibrate mechanical and electronic negative gauge pressure measuring instruments, only one of them is suitable for calibration of piston gauges in negative gauge mode. Along with the cost, factors such as types of instruments to be calibrated, calibration ranges and laboratory altitude should also be taken into account when deciding about which one of the three methods to implement.

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