Investigation of the effects of valve closing in a static expansion system

Th. Bock\textsuperscript{a,*}, M. Bernien\textsuperscript{a}, Ch. Buchmann\textsuperscript{a}, T. Rubin\textsuperscript{a}, K. Jousten\textsuperscript{a}

\textsuperscript{a}Physikalisch–Technische Bundesanstalt, Abbestraße 2–12, 10587 Berlin, Germany

Abstract

The precise measurement of the pressure of the gas enclosed in the starting volume of a static expansion system is important for achieving low measurement uncertainties. This work focuses on the valve closing process and the resulting pressure difference. The flow conservation law is used to set up a model and an experimental procedure has been developed to investigate the pressure drop across the moving valve plate. Various parameters are evaluated and compared with experimental data.

Keywords: Vacuum, static expansion system, metrology, instrumentation, valve, volume, conductance

1. Introduction

To this day static expansion systems are one of the most accurate realizations of the pressure scale in the range from $1 \times 10^{-2}$ Pa to 100 Pa and are used as primary standards in many metrology institutes [1]. Many contributions to the uncertainty have been investigated in great detail, e.g., the impact of the temperature during expansion and real gas effects [2, 3]. In this study we focus on the dynamic effects during the closing process of the valve that encloses the gas to be expanded. These effects become increasingly important when working with small volumes.

The PTB vacuum group currently evaluates the new static expansion primary standard SE3. The entire calibration pressure range will be covered by three different single stage expansions. This is beneficial since multiple consecutive expansions lead to increased uncertainties. The principle of static expansion relies on the transfer of a fixed amount of gas from a small volume into a larger volume. If the initial pressure and the ratio of the two volumes is known precisely, a well-known lower pressure is generated. In order to generate a calibration pressure $p_{\text{cal}}$ of $1 \times 10^{-2}$ Pa an expansion ratio $f = V_s/(V_s + V_e)$ of...
1 \times 10^{-4} \text{ and a filling pressure } p_{\text{fill}} \text{ of } 100 \text{ Pa is used. } V_s = 0.021 \text{ is the smallest starting volume of SE3. } V_e \text{ is the } 2001 \text{ volume of the calibration vessel.}

In static expansion systems the precise definition of gas amount \( N \) enclosed in the starting volume \( V_s \) is crucial. \( N \) is defined by

\[
N = (p_{\text{fill}} + \Delta p) \frac{V_s}{k_B T_{\text{before}}}
\]  

(1)

where \( k_B \) is the Boltzmann constant and \( T_{\text{before}} \) is the temperature of the enclosed gas. \( p_{\text{fill}} \) can only be measured outside the starting volume since a gauge introduces heat and additional volume. \( \Delta p \) denotes a potential pressure difference between the position of the gauge and inside of the closed starting volume. This pressure difference is the main topic of this work. After presenting an initial investigation, the layout and the design of the start volumes as well as the associated valves are described in the following. Then the pressure development during the closing phase is modeled. The derived dependencies are compared to experimental results. The experiments were performed with two different valves of the same type. Expansion ratio measurement with nitrogen, helium and argon are presented. A correction factor and an additional measurement uncertainty are introduced.

2. Initial investigation

To check for pressure differences due to valve closing of the starting volume, initial investigations with a modified pneumatic DN16CF all-metal valve were conducted. A differential capacitance diaphragm gauge (CDG) was used to measure \( \Delta p \) after closing the valve while varying the filling pressure, closing speed and gas species (see Figure 5).

Figure 1 shows the results of these investigations. Relative differences up to \( 1 \times 10^{-3} \) occur at high closing speeds. Pressure variations cause relative differences up to \( 7 \times 10^{-4} \), depending on the gas species. It became obvious that these deviations would significantly increase the measurement uncertainty of the system. To gain a better understanding, time resolved experiments were planned and carried out as described below. The use of servomotors allows for a precise speed and force control of the valve closing process.

3. Valves of the starting volume

Each of the starting volumes of SE3 consists of an aluminum cylinder and two DN16CF valves (inlet and outlet valves). The aluminum cylinders are designed and build to ensure an easily measurable volume. With a nominal volume of 5 cm\(^3\) the valves contribute with 50\% to the value of \( V_s \). The inlet and the outlet valves used for \( V_s \) are DN16CF all-metal corner valves of the manufacturer VAT (Type 571 24-GE02).
Figure 1: Results of initial investigations with a pneumatic DN16CF corner valve and an experimental setup as shown in Figure 4. The valve is modified: it has a reduced travel path length of 2 mm instead of 10 mm. The relative pressure differences across the valve plate after the closing process are plotted as a function of closing time for two different filling pressures of helium (left) and as a function of filling pressure for helium, argon and nitrogen with a closing time of 1 min (right).

Figure 2: Scans along the inlet valve body (blue) and the valve plate (red). Until the valve is closed, the volumes $V_1$ and $V_2$ are connected via the conductance $C(t)$. In the diagram the valve plate is positioned $\approx 1$ mm in front of the closed position. The direction of movement and the speed of the valve plate during the closing process are indicated by an arrow. The factor $g_{spring}$ is equal to 1 as long as there are no forces in $z$ direction. Furthermore, $F_{plate}$, the position of the sealing point at the valve plate and at the valve body is annotated.
3.1. Geometrical dimensions

For the purpose of the geometric determination of the contributing volume three valves were characterized by means of a 3D coordinate measuring machine. The dimensions of the inner volume were measured with up to 10 \((x,y,z)\)-line-scans per valve. These scans were used to create the schema in Figure 2. From these measurements a model of the valve with mean coordinates and uncertainties is derived. The sealing point of this model valve is determined to be at a radius of \((8.46 \pm 0.01)\) mm and the slope of the valve seat is \(m_{\text{seat}} = -(0.267 \pm 0.002)\) mm/mm. The radius of the piston is about 8 mm, its length almost 25 mm. The valve seat is 3 mm long.

3.2. Valves closing mechanism

Servomotors are used to open and close the valves. They provide a more precise control of the valve plate position, velocity and closing torque compared to a pneumatic setup. The motors are coupled to a gear with a ratio of \(R = 1/169\). The spindles of the valves have a pitch of \(s = 1.4\) mm per revolution. The linear velocity of the piston of the valve is given by

\[
v = \omega R s
\]

(2)

where \(\omega\) is the number of revolutions per minute. The relevant rotational speed range lies between \(\omega = 20\) rpm and \(\omega = 100\) rpm. With Equation (2) one gets velocities \(v\) between \(2.8 \times 10^{-6}\) m/s and \(14 \times 10^{-6}\) m/s.

The regulation circuit of the servomotor control is used to force a constant rotation velocity. To keep \(\omega\) constant, the servomotor current \(i_{\text{servo}}\) increases with increasing torque. The motor is stopped when the spring socket reaches its stopper and specified torque for closing the valve is reached.

3.3. Valve spring

The valve’s closing mechanism includes an arrangement of plate springs. If a force \(F_{\text{plate}}\) is exerted on the valve plate, the spring structure is deformed according to Hook’s law by \(\Delta z = \frac{F_{\text{plate}}}{D}\), where \(D\) is the spring constant and \(\Delta z\) the length difference due to deformation. The plate springs have a spring constant of 2400 N/mm and are pre-loaded with a force of about 600 N corresponding to a deformation of about 0.25 mm. Once the valve plate reaches the sealing point, the force on the valve plate increases. Once the force exceeds the pre-load, the deformation of the plate springs increases according to \(\Delta \dot{z} = \Delta v = \frac{F_{\text{plate}}}{D}\). Consequently, the relation between \(\omega\) and \(v\) as given in Equation (2) is modified. For \(\dot{F}_{\text{plate}} > 0\), the velocity of the valve plate \(v_{\text{plate}} = v - \Delta v\) is reduced. This is accounted for by a factor \(g_{\text{spring}} = 1 - \frac{\Delta v}{v}\), hence

\[
v_{\text{plate}} = \omega R s g_{\text{spring}}.
\]
Figure 3: Schematic drawing of the valve closing mechanism.
4. Model of gas flow during valve closing

The pressure and flow conditions during the closing process can be modeled by means of two coupled differential equations

\[ V_1 \frac{dp_1}{dt} = -p_1 \frac{dV_1}{dt} + C(t)(p_2 - p_1) \]  \hspace{1cm} (4)

and

\[ V_2 \frac{dp_2}{dt} = -p_2 \frac{dV_2}{dt} - C(t)(p_2 - p_1) \]  \hspace{1cm} (5)

based on the flow conservation law and the definition of flow conductance. Here, \( C(t) \) is the time-dependent conductance formed by the gap between valve plate and valve body. \( V_1 \) and \( p_1 \) denote the volume and the pressure behind the valve plate (left in Figures 2 and 3). \( V_2 \) and \( p_2 \) are the starting volume and the pressure of the enclosed gas (in front of the valve plate, right in the Figures 2 and 3). The pressure \( p_1 \) is measured by means of a Quartz Bourdon Spiral (during the initial experiments) or by means of a group of capacitance diaphragm gauges (SE3 group standard). \( p_1 \) corresponds to the filling pressure \( p_{\text{fill}} \) introduced in Equation 1.

During the experiments and during a calibration the inlet valve is closed and afterwards \( p_{\text{fill}} \) is measured. The metrologically relevant but inaccessible quantity is the pressure \( p_2 = p_{\text{fill}} + \Delta p \). Equation 5 can be rewritten as

\[ \dot{p}_2 = \frac{1}{V_2} \left( -p_2 \dot{V}_2 - C(t) \Delta p(t) \right). \]  \hspace{1cm} (6)

The following sections present information on \( p_2 \dot{V}_2 \) called the volume term and \( C(t) \Delta p(t) \) named conductance term.

4.1. Volume term

The term \( p_2 \dot{V}_2 \) corresponds to a flow caused by the motion of the valve plate along the \( z \) axis at a pressure \( p_2 \). With an area of \( A_{\text{plate}} = \pi r_2^2 \) the volume term can be expressed by the equation

\[ p_2 \dot{V}_2 = p_2 \frac{A_{\text{plate}}dz}{dt} = p_2 A_{\text{plate}} v_{\text{spring}}. \]

Table 1 gives an overview for the case \( g_{\text{spring}} = 1 \) and a volume of \( V_2 = 0.021 \).

| \( \omega \) in rpm | \( -\dot{V}_2 \) in \( \text{cm}^3/\text{s} \) | \( -\dot{V}_2/V_s \) in \( 1/\text{s} \) |
|-------------------|------------------|------------------|
| 20                | \( 6.3 \times 10^{-4} \) | \( 3 \times 10^{-5} \) |
| 80                | \( 2.5 \times 10^{-3} \) | \( 1.3 \times 10^{-4} \) |
| 100               | \( 3.1 \times 10^{-3} \) | \( 1.6 \times 10^{-4} \) |

The volume term increases the pressure in \( V_2 \). It is independent of the gas species and countered by the conductance term when \( \Delta p(t) > 0 \).
4.2. Conductance term

The conductance \( C(t(z)) \) strongly depends on the position \( z(t) \) of the valve plate relative to the valve body. Before entering the conical section of the valve seat (see Figures 2 and 3), no significant pressure difference \( \Delta p(t) = p_2 - p_1 \) is possible because of the large gap between valve plate and valve body. In this range, the flow conductance \( C \) is about 5l/s. Due to the conical shape of the valve body, \( C(t) \) decreases until the sealing point is reached. For the position of the valve plate sketch in Figure 2, the gap is about \( \Delta r = 0.27 \text{mm} \).

In the molecular regime, the conductance of the annular gap \( C_{\text{gap}} \) can be estimated from the model of a slot with height \( b = \Delta r = r_o - r_i \), width \( a = \pi (r_o + r_i) \) and length \( l \) [4]:

\[
C_{\text{mol}} = \sqrt{\frac{RT}{2\pi M}} a b \left( 1 + \ln(0.433l/b + 1) \right) \frac{1}{l/b + 1} \tag{7}
\]

where \( R \) is the gas constant, \( T \) the temperature of the gas and \( M \) the molar mass of the gas molecules. The inner radius of the annular gap can be identified with the radius of the valve plate: \( r_i = r_{\text{plate}} \). The outer radius is given by the radius of the valve seat that depends on the position of the valve plate. Until the sealing point at \( z = 1 \text{mm} \) is reached, the outer radius is given by \( r_o = r_i + m_{\text{seat}}(1 \text{mm} - z) \).

Equation (7) is valid for the molecular flow regime. At the lowest filling pressure of 100 Pa, the mean free path is \( \lambda = 66 \mu \text{m} \) for nitrogen corresponding to a Knudsen number of \( Kn \approx \lambda/\Delta r = 2.5 \) at \( z = 0.9 \text{mm} \). If the valve plate continues to move along the \( z \)-axis, \( \Delta r \) decreases and \( Kn \) increases. On the other hand, with increasing filling pressure \( Kn \) decreases such that the assumption of molecular flow is no longer valid.

In the viscous regime and laminar flow conditions, the conductance of the slot \( (a \gg b) \) is given by [4]:

\[
C_{\text{vis}} = \frac{1}{24\eta} \frac{a b^3}{l} (p_1 + p_2) \tag{8}
\]

with \( \eta \) the viscosity of the gas. Note that due to \( p_1/p_2 \approx 1 \) no choking of the gas flow occurs. Hence, the conductance strongly increases with increasing pressure. The conductance over the entire pressure range is approximated via \( C_{\text{gap}} \approx C_{\text{mol}} + C_{\text{vis}} \) [4]. Interestingly, the strong increase in conductance only leads to moderate reduction in the relative pressure difference of about 33% for a filling pressure of 100 Pa and 100000 Pa. The reason is that only close to the sealing point a significant pressure difference builds up and that \( C_{\text{gap}} \) asymptotically approaches \( C_{\text{mol}} \) when \( b \) approaches zero.

Beside the pressure dependency, \( C(t) \) also depends on the gas species. This affects \( p_2 \) via Equation (3) and therefore the amount of gas enclosed in the starting volume once the valve is closed.
Figure 4: Time dependence of the starting volume (a), flow conductance (b), pressures (c) and relative pressure difference (d) for $\omega = 80$ rpm and $g_{spring} = 0.02$. Four positions of the valve plate at $z = 0.90, 1.00, 1.01$ and 1.02 mm are indicated by vertical lines.
Table 2: Estimation of the conductance in the molecular regime at room temperature and nitrogen for an annular slot of length $l = 0.2\ mm$ according to Equation 7. $z$ is given relative to the position shown in Figure 2.

| $z$ (in mm) | $\Delta r$ (in mm) | $C_{mol}$ (in cm$^3$/s) |
|------------|---------------------|--------------------------|
| 0          | 0.267               | 1250                     |
| 0.9        | 0.0267              | 48                       |
| 0.99       | 0.00267             | 1.0                      |

4.3. Pressure evolution

The time-dependent pressure evolution can be obtained by numerical solution of Equations 4 and 5. The change in volume is solely attributed to $V_2$, and $\dot{V}_1$ is set to zero. Other choices are possible but will not affect the result because a significant pressure difference only builds up shortly before the closing position of the valve plate is reached.

Figure 4 shows the time-dependent evolution of the starting volume (a), flow conductance (b), pressures (c) and relative pressure difference (d) for $\omega = 80\ \text{rpm}$ and $g_{spring} = 0.02$. Four positions of the valve plate at $z = 0.90$, 1.00, 1.01 and 1.02 mm are indicated by vertical lines. The first line marks the position at which a significant pressure difference builds up. The second line marks the position at which the valve plate starts to seal against the valve seat and the velocity of the valve plate is strongly reduced to $v_{spring}$. The third line marks the positions up to which a potential leak after sealing against the valve seat persists. The last line marks the position at which the specified closing torque of the valve is reached and the movement of the valve plate is stopped.

In Figure 4 the pressure in the starting volume $p_2$ (yellow) and in the volume of the gas inlet $p_1$ (blue) are plotted as a function of time together with the pressure evolution for a situation in which a leak of 0.2 cm$^3$/s persists up to $z = 1.01\ mm$. Four different phases of the valve-closing process can be identified. Up to about $z = 0.90\ mm$, the change in volume is constant and causes a relative change in pressure according to $\dot{p}_1/p_1 = \dot{p}_2/p_2 = -\dot{V}_2/(V_1 + V_2)$. The flow conductance is decreasing but no significant pressure difference is generated down to about 300 cm$^3$/s (see Figure 2a and 2b). Below this conductance, the pressure in the starting volume rises strongly approaching $p_2/p_2 = -\dot{V}_2/V_2$. When the valve plate reaches the sealing point ($z = 1.00\ mm$) its movement is strongly reduced because of the force exerted. The change in pressure in the starting volume is now $\dot{p}_2/p_2 = -g_{spring}\dot{V}_2/V_2$. The increase in pressure may partly counteracted by a gas flow through a leak until the valve is fully sealed at $z = 1.01\ mm$ (gray lines in Figure 4c and d). The pressure in the starting volume continues to increase until the closing torque is reached and the movement of the valve plate is stopped ($z = 1.02\ mm$). The model contains four valve-specific parameters that must be adjusted to match the measurements: the reduction in movement of the valve plate $g_{spring}$ once the sealing point is reached, the position at which the closing torque is reached, the size of a potential leak, and the length of the channel of the annular conductance formed by the valve.
Figure 5: Experimental setup. During the valve closing process the differential pressure $\Delta p(t)$ across the valve plate as well as the servomotor current $i_{\text{servo}}$ are recorded. The experiment was realized with two different valves of the same model.

plate and the seat. The latter significantly impacts the pressure difference that is built up in the moment the sealing position is reached and is chosen to be 0.2 mm here.

5. Experiment

The differential pressures were investigated systematically by varying the parameters $V_1$, $V_2$, $\omega$, $p_1$ and the gas species. Figure 5 shows a sketch of the experimental setup. $\Delta p(t)$ was measured by means of a differential capacitance diaphragm gauge with a full scale of 1.3 kPa. The heater of the CDG was turned off. For each measurement run, the valve plate was set about 2 mm in front of the closing position, where the offset corrected signal of the CDG is still zero. Then the closing process was initiated and the CDG measurement signal was recorded as a function of time until the closing torque was reached. In some cases the current consumption of the servomotor was recorded simultaneously. The measurements were repeated several times in order to examine the repeatability.

All measurements show more or less the characteristics plotted in Figure 6. The graph can be roughly separated in two parts. Up to the position marked with (A), the slope of the relative pressure change $r_p = \frac{\Delta p(t)}{p_1}$ depends on the volume $V_2$. The height of $r_p$ corresponds to the travel length, the valve plate can
move without an occurring $F_{\text{plate}}$ in the region with low conductivity. After the position marked with (A), the slope of $r_p$ depends mainly on the valve spring constant $D$. Since the velocity gets slower, the conductance term has a more significant influence.

Figure 6 shows $r_p$ and the current $i' = i_{\text{servo}} - i_{\text{servo},0}$ for three repeated measurements. $i_{\text{servo}}$ denotes the total current consumption of the servomotor and $i_{\text{servo},0}$ the current for $F_{\text{plate}} = 0$. $r_p$ and $i_{\text{servo}}$ are simultaneously recorded. At mark (A) $i'$ rises significantly. This can be related to the increase of the force on the valve plate by friction and an associated torque $\tau_{\text{servo}}$: $F_{\text{plate}} \propto \tau_{\text{servo}} \propto i'$. The spring deforms and the velocity of the valve plate is strongly reduced (see section 3.3). The associated volume flow is reduced by the same factor resulting in a flatter slope in the $\Delta p(t)/p_1$ vs. $t$ chart.

5.1. Volume variation

Equation 6 shows that $\dot{p}_2 \propto \frac{1}{V_2^2}$. This dependency is confirmed by the experimental results shown in Figure 7. Both series of measurements with three repetitions show the expected dependency in the slope of the resulting $r_p$. The diagram includes the theoretical values for the contribution of the volume term.
for the case $V_2 = 15$ ml and $V_2 = 25$ ml without the conductance term. Due to the omitted $C(t) \Delta p(t)$ part the theoretical slopes are larger compared to the measured slopes.

The measurements shown in Figure 9 aimed also on the examination of the influence of $V_1$ on $p_1$. $V_1$ has no measurable influence on $\Delta p(t)$ at least in the case $V_1 \gg V_2$.

5.2. Closing speed variation

The influence of the closing speed $\omega$ was examined by the measurements plotted in Figure 8. The conductance term causes a back flow $\Delta p_{bf}(t) = \int_{t_0}^{t} C(t') \Delta p(t') \, dt'$. This means that the longer the closing process lasts the more the pressure rise in $V_2$ is reduced by the conductance term. Like in Figure 7 the theoretical values for the contribution of the volume term for the case $V_2 = 25$ ml without the $C(t) \Delta p(t)$ part are included.

5.3. Influence of valve tolerances

The data shown in Figure 9 were measured using another valve of the same type as the one used for Figures 7 and 8. A different shape and height of the $r_p$ graph can be observed. Especially the constant or partially decreasing pressure in the range 30 s to 60 s is remarkable. An explanation may be a slowed down movement of the valve plate along the z-axis or a leak that persists until the valve is fully sealed as depicted in Figure 4d. During the period when the movement stops or slows down, the conductance term lowers the $\Delta p(t)$.
Figure 8: Relative pressure difference as a function of time for different closing speeds with $p_1 = 10$ kPa, $V_1 = 31$ and $V_2 = 25$ ml

Figure 9: Relative pressure difference as a function of time for different volumes $V_1$ and pressures $p_1$ with $V_2 = 25$ ml and $\omega = 80$ rpm
5.4. Pressure variation

The influence of $p_1$ on $\Delta p(t)$ in Figure 9 is expected from the considerations given in section 4.2: in the viscous flow regime an increasing pressure causes an increasing conductance. With a larger $C$ the amount of back flow from $V_2$ to $V_1$ becomes bigger and the pressure increase in $V_2$ is reduced more strongly (see Equation 6).

5.5. Influence of the gas species on the expansion ratio

$C(t)$ also depends on the gas species with $1/\sqrt{M}$ where $M$ is the molar mass. This means that helium should cause a lower final $\Delta p(t)$ than argon. This was investigated by determining the SE3 expansion ratios as shown in [6].

The expansion ratio $f$ is determined from the pressure ratio according to

$$f = \frac{p_{\text{after}}}{p_{\text{before}}} \frac{T_{\text{before}}}{T_{\text{after}}}.$$  \(9\)

In Equation 9 $p_{\text{before}}, T_{\text{before}}, p_{\text{after}}$ and $T_{\text{after}}$ are the pressures and temperatures before and after the expansion. $p_{\text{after}}$ is determined by means of the pressure balance FRS5 [6] and a differential CDG. $p_{\text{before}}$ is measured by using the SE3 group standard.

For this method the following has to be considered: since $p_{\text{before}}$ is measured after the closing of the inlet valve of the start volume, $\Delta p(t)$ does not affect $p_{\text{before}}$. However, $\Delta p(t)$ enlarges $p_{\text{after}}$ by $2 \times 10^{-4}$ to $6 \times 10^{-4}$ as the results of the previous section have shown. The value of the expansion ratio determined from a pressure ratio contains this effect: the enlarged $p_{\text{after}}$ causes an enlarged $f$.

The largest effect of the gas species on $f$ is expected for the smallest volume $V_s$, since the volume $V_2$ (see Equation 5) has to be small to gain a measurable effect. In order to use FRS5 for the measurement of $p_{\text{after}}$ with low uncertainties, the gas inside the calibration vessel has to be accumulated [5, 7]. With a filling pressure of 100 kPa and a nominal $f_s$ of $1 \times 10^{-4}$ one expansion step generates 10 Pa. The results of the investigation of the gas species dependency of $f_s$ are given in Table 3.

Table 3: Result of expansion ratio measurements carried out according the method described in [5, 7] and [6] for different gas species. Given are the determined values of $f_s$ and the expanded relative type A measurement uncertainties with a coverage factor of $k = 2$.

| gas    | $f_s$          | $u_a(f_s)$     |
|--------|----------------|----------------|
| helium | $1.05723 \times 10^{-4}$ | $7.2 \times 10^{-8}$ |
| nitrogen | $1.05759 \times 10^{-4}$ | $5.8 \times 10^{-8}$ |
| argon  | $1.05739 \times 10^{-4}$ | $3.7 \times 10^{-8}$ |

Due to the uncertainties the differences between the values are not significant.
6. Discussion

6.1. Temperature influence

The experiments described in section 5 were carried out in a room where the temperature changes are less than 0.15 K/h. This maximum temperature change results in a relative change in pressure of $8 \times 10^{-6} \text{min}^{-1}$. These variations have a random characteristics. A potential heat contribution caused by friction between the plate and the valve seat is considered to be negligibly small.

6.2. Influence of valve bellows volume change

The change in the volume of the valve due to deformation of its bellows $\Delta V_{\text{bellow}}$ is negligible too: on the one hand $\Delta V_{\text{bellow}}$ relates to the volume $V_1 + V_2$ until a significant $C$ takes effect. With a significant $C$, $\Delta V_{\text{bellow}} \ll V_1$ applies safely. At least, Figure 9 shows no indication of a significant influence of $\Delta V_{\text{bellow}}$.

6.3. Discontinuous closing movement

Figure 2 suggests that there is no contact between the valve plate and the seat until closing torque is reached. However, if the valve is open, the valve plate can be moved freely by about 1 mm to 2 mm perpendicular to the z axis. This is an intended engineering behavior since it is necessary to avoid a mechanical over determination. The observed flattening of the $r_p$ curve in Figure 9 can be explained by a discontinuous movement in combination with a small conductance $C$. Moreover, Figure 6 gives indications for a stick slip motion by the $i'_{\text{servo}}$ signal especially for small $\omega$. Since the stick slip friction has a random characteristic, the overall final $\Delta p(t)$ contains some random contributions.

7. Implications for the generated calibration pressure

The considerations in Section 4 showed that a pressure and gas species dependence of $r_p$ can be expected. Both dependencies are introduced by the conductance term. The closing process takes place within the viscous flow regime. Here a smaller $p_{\text{fill}}$ causes a smaller $C$. A smaller molecular mass causes a larger $C$ with $1/\sqrt{M}$. Due to the high measurement uncertainty and the small size of the effect, it was not measurable as Table 3 shows. However, a correction factor should be introduced.

7.1. Pressure dependent correction factor

For the calibration pressures generated by means of $V_s = 0.021$ the following correction $F_p$ is derived from the experimental data shown in Figures 6 to 9

$$F_p = (1 - 2.2 \times 10^{-9} \text{Pa}^{-1} p_{\text{fill}} + 2.2 \times 10^{-4})$$

$F_p$ has a relative standard measurement uncertainty of $2.3 \times 10^{-5}$. At 100 kPa, the filling pressure used to determine $f_s$, $(F_p-1)$ becomes 0. For 100 Pa, the smallest $p_{\text{fill}}$, $F_p$ is $2 \times 10^{-4}$. $F_p$ scales reciprocally with the size of $V_2$: if $V_2$ is ten times larger $F_p$ becomes ten times smaller.
7.2. Measurement uncertainty due to gas-species dependency

Since the differences between the values of $f_s$ for nitrogen, argon and helium are not significant (see Table 3), an uncertainty weighted mean value is calculated from all of the three values. The dependency of $f_s$ from the gas species shall not be corrected but taken into account with the following additional standard measurement uncertainty.

$$u_g = \left| 2.7 \times 10^{-4} \left( 1 - \frac{\sqrt{M}}{\sqrt{24 \text{ g/mol}}} \right) \right|$$

(11)

Here, $M$ denotes the molecular mass of the gas used during calibration. Table 4 summarizes the related additional measurement uncertainty for some gases. Also $u_g$ scales reciprocally with the size of $V_2$.

Table 4: Expanded relative measurement uncertainty for different gas species with a coverage factor of $k = 2$. $U_g$ is relative to the calibration pressure and calculated for $V_s = 0.021$.

| gas      | $U_g$ |
|----------|-------|
| helium   | $3.3 \times 10^{-4}$ |
| neon     | $4.9 \times 10^{-5}$ |
| nitrogen | $4.5 \times 10^{-5}$ |
| argon    | $1.6 \times 10^{-4}$ |
| krypton  | $4.9 \times 10^{-4}$ |

8. Summary and Conclusion

Due to the valve closing process pressure differences occur depending on the volume, closing speed, filling pressures and the gas species. The effects can be described by a differential equation derived from the flow conservation law. For a DN16CF valve and volumes of a size around $0.021$ (size of the smallest starting volume of SE3) relative pressure differences of some $10^{-4}$ are generated.

Since the expansion ratios at SE3 are determined by a pressure ratio which includes the closing process, the effect of an enlarged gas amount is reflected in the values of the $f_i$. It seems to be advisable to reevaluate the value of the $f_i$ if the inlet valve of the starting volume is replaced. The minor contribution of the filling pressure and gas-type dependencies are considered by a correction factor and by a measurement uncertainty contribution, respectively. These insights on the dynamic effects during the closing process of small volumes pave the path towards an accurate assessment of enclosed amounts of gas most relevant but not limited to the application in static expansion systems.

Acknowledgment

Kurt Sonderegger from VAT Group AG is acknowledged for the insights he provided on the mechanical design of the valve.
References

[1] Final report on the key comparison CCM.P-K4.2012 in absolute pressure from 1 Pa to 10 kPa, J. Ricker, J. Hendricks, Th. Bock, P. Dominik, T. Kobata, J. Torres and I. Sadkovskaya Metrologia, 54 (2017), Tech. Suppl., doi:10.1088/0026-1394/54/1A/07002

[2] Temperature relaxation of argon and helium after injection into a vacuum vessel, K. Jousten, Vacuum, 45 (1994), 1205–1208

[3] High-accuracy calibration in the vacuum range 0.3 Pa to 4000 Pa using the primary standard of static gas expansion, W. Jitschin, Metrologia, 39 (2002), 249–261

[4] Handbook of Vacuum Technology, 2nd edition, K. Jousten (editor), Wiley-VCH Verlag Co. KGaA, Weinheim (2016), ISBN 978-3-527-41338-6.

[5] KW Elliott, PB Clapham NPL report MOM 28 (1978)

[6] Reduction of the uncertainty of the PTB vacuum pressure scale by a new large area non-rotating piston gauge, Th. Bock, H. Ahrendt, K. Jousten, Metrologia, 46 (2009), 389–386

[7] A discussion of methods for the estimation of volumetric ratios determined by multiple expansions, F. J. Redgrave, A. B. Forbes, P. M. Harris, Vacuum, 53 (1999), 159–162