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A TECHNIQUE TO IMPROVE LOW LEAK RATE MEASUREMENT WITH A CONSTANT-VOLUME FLOWMETER

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Abstract:
Calibrating leak rates using a constant-volume flowmeter consists in measuring the pressure rise rate produced by the leak throughput in a measurement volume. When the leak rates to be determined are low, the latter should be as small as possible, and since it is composed of dead volumes of the tubing, the valves, the gauge, *etc.*, one needs to determine this dead volume accurately. This paper describes an original and simple technique which provides a relative uncertainty in the calibration of the dead volume of a constant-volume flowmeter (~ 60 cm^3) of approximately 0.1 % (k = 1). In turn, the bottom end of the calibration range of the flowmeter has been brought down to about 5 × 10^{-8} Pa·m^3·s^{-1}.

Keywords: vacuum; low leak rate calibration; constant-volume flowmeter

1. INTRODUCTION

Measuring a leak rate consists in determining its throughput q at a given temperature T, by means of volume (V) and pressure (p) measurements over time (t):

$$q = \frac{d(pV)}{dt} \,. \tag{1}$$

In leak metrology, the "state-of-the-art" instrument to achieve such measurements is the constant-*pressure* flowmeter [1], [2]. While the equation for the throughput can be written simply as:

$$q = p_0 \frac{\Delta V}{\Delta t} \,, \tag{2}$$

p_0 being the constant pressure set in the instrument, the associated volume variation device makes this instrument somewhat complicated to build [1], [2].

In the constant-*volume* flowmeter, the throughput is given by:

$$q = V_0 \frac{\Delta p}{\Delta t} \,. \tag{3}$$

On this basis, only a pressure gauge is necessary to make measurements, which significantly simplifies the design of the instrument. However, the volume V_0, inevitably composed of the dead volume V_d, which connects the different parts of the experimental setup including the connection volume of the artefact to the flowmeter, should be determined at each calibration of a leak rate. This lengthens the calibration time and slightly increases the associated uncertainty in the leak rate compared with a constant-*pressure* flowmeter.

In this work, a technique using a simple mechanical device is implemented in the *Laboratoire commun de métrologie* (LCM LNE-Cnam) constant-*volume* flowmeter. This device reduces the time of the dead volume determination, especially for feeble leak rates (typically below 1 × 10^{-6} Pa·m^3·s^{-1}), provides a better uncertainty in V_d and thus in the calibrated leak rate. It also stretches the calibration range of this flowmeter from 2 × 10^{-7} Pa·m^3·s^{-1} down to roughly 5 × 10^{-8} Pa·m^3·s^{-1}.

2. CONSTANT-VOLUME FLOWMETER

While only leak rates referred to vacuum are treated in what follows, one could also apply the method to leak rates referred to atmosphere, albeit for a bottom end of the flow range, higher than in vacuum configuration.

2.1. Principle

The experimental setup to calibrate a leak with our constant-volume flowmeter is shown Figure 1.

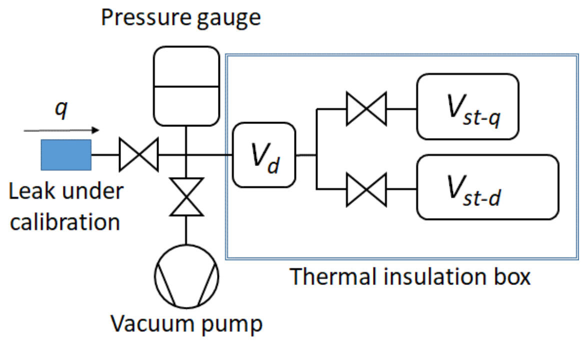

Figure 1: Principle set-up of the constant-volume flowmeter at LNE-Cnam.

q is the throughput of the leak in calibration, V_d the dead volume, $V_\mathrm{st\text{-}q}$ the standard volume for the leak calibration and $V_\mathrm{st\text{-}d}$ the standard volume used for the dead volume determination.

One should bear in mind that the relative standard uncertainty in $V_{\text{st-q}}$ or $V_{\text{st-q}}$ is about 2×10^{-4} (calibrated by means of the hydrostatic weighing method) and that of V_{d} is at best slightly below 1×10^{-3} [3].

The first step is to determine V_{d} using the standard volume $V_{\text{st-d}}$ (see details in section 2.2 below). Then, the throughput is deduced from equation (3) where V_0 can be either the sum of $V_{\text{st-q}}$ and V_{d} or only V_{d} when the leak rate to be calibrated is small. In the latter situation, the uncertainty in V_{d} becomes even more crucial.

2.2. Determination of the dead volume

In our previous work [3], we presented the procedure for the dead volume determination.

We let the throughtput q flow successively into the volume V_{d} associated to $V_{\text{st-d}}$ then into V_{d}, and we measure at each step the pressure rise rates $\dot{p}_{\text{st-d}}$ and $\dot{p}_{\text{d}}$ respectively. These rates are pressure slopes deduced from pressure recording over time. We saw in [3] that V_{d} is expressed as:

$$V_{\text{d}} = V_{\text{st-d}} \frac{\dot{p}_{\text{st-d}}}{\dot{p}_{\text{d}} - \dot{p}_{\text{st-d}}} \,. \tag{4}$$

To obtain an optimised uncertainty in V_{d}, the value of $V_{\text{st-d}}$ should be at least five times higher than V_{d} [3], which, in other terms means that in this case, $\dot{p}_{\text{st-d}}$ is six times lower than $\dot{p}_{\text{d}}$. In addition, to estimate the repeatability, the measurements should be repeated at least three times.

2.3. Limitation for low leak rates

The limitation to measure low leak rates down to 2×10^{-7} Pa·m^3·s^{-1} or even lower, is related to the time required to determine very small induced pressure rise rates with a sufficient accuracy despite the presence of a residual pressure rate $\dot{p}_{\text{res}}$ (the remaining pressure variation when no flow enters the flowmeter) due mainly to thermal effects. This implies that the uncertainty in V_{d} rises when the leak flow rate q to be determined falls. Since in this case the measurement volume is also V_{d}, this also increases the uncertainty in q.

Residual pressure rate

To address the smaller leak rate measurements, the pressure gauge in Figure 1 is a capacitance diaphragm gauge (CDG) of 130 Pa full scale. The measurement volume, V_{d}, is about 60 cm^3.

After pumping down the flowmeter for at least 60 hours and letting the pressure climb to about 45 Pa by means of the gas flowing through the leak under calibration, the valve downstream the leak artefact is shut down and the pressure in V_{d} is recorded over three hours. One can observe in Figure 2 that the pressure variation is periodic with a period of about 480 s. Application of a least-squares regression on a time interval overlapping five oscillations (see Figure 2(b)) yields $\dot{p}_{\text{res}} = -3.5 \times 10^{-6}$ Pa·s^{-1} giving a residual flow rate $\dot{q}_{\text{res}} = -2.1 \times 10^{-10}$ Pa·m^3·s^{-1}. By considering at least three oscillations for a proper estimation of $\dot{p}_{\text{res}}$, and a prior stabilisation time of 45 min after valve closure, a cycle including the pressure rate measurement due to the leak under calibration and the associated residual pressure rate measurement lasts about 1 h 15 min.

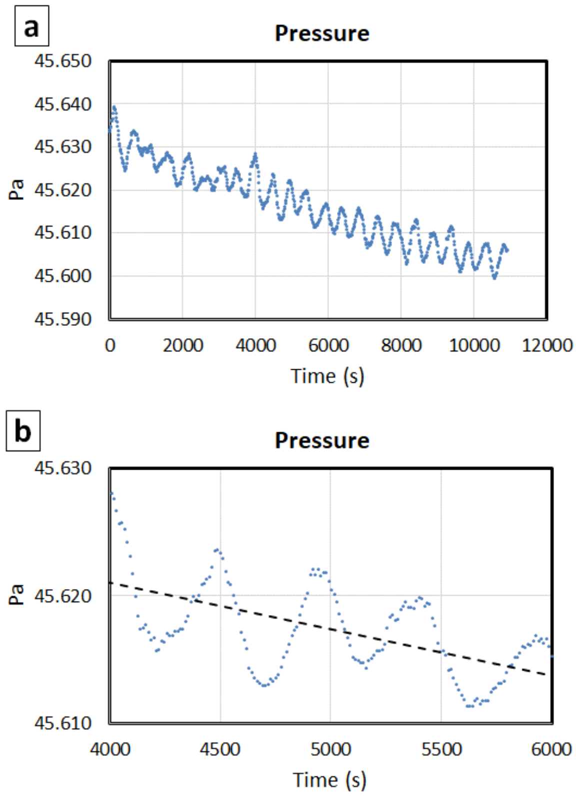

Figure 2: Recording of the residual pressure in the dead volume V_{d}

(a): overall recording; (b): zoom on the range where the mean residual pressure slope is estimated (represented by the dashed line)

As the necessary determination of V_{d} in three cycles requires four determinations of $\dot{p}_{st-d}$ and three determinations of $\dot{p}_{d}$ [3], to measure it with the best uncertainty would take six to seven hours.

Limitation for the uncertainty in V_{d}

In addition to the inconvenience of a long calibration time for V_{d}, the resulting relative uncertainty in V_{d} cannot be as low as that stated in [3], which is below 0.1 % (k = 1). The limitation lies in the stability of the residual pressure rate regarding the pressure rate caused by the leak under calibration, or simply said, in the ratio of the equivalent residual flow rate q_{res} stability to the measured flow rate q.

In the constant-volume flowmeter set-up, we assume a short-term stability of the residual flow rate estimated to be 3×10^{-10} Pa·m³·s⁻¹ ($k = 1$). An uncertainty $u(V_d) < 0.1$ % could be then obtained for q higher than 3×10^{-7} Pa·m³·s⁻¹, in the best scenario, assuming there are other uncertainty sources to be taken into account.

Summary for low leak rates measurements

The calibration method of V_d for low leak rates, cannot be achieved within a reasonable time if the best possible uncertainty is needed. For this reason, we have limited the calibration range of our constant-volume flowmeter to 2×10^{-7} Pa·m³·s⁻¹ to avoid the determination of the residual pressure rate during the dead volume measurement. It is however checked to ensure that the associated residual flow rate is lower than 5×10^{-9} Pa·m³·s⁻¹. The latter is considered as a non-applied correction in our standard uncertainty in a leak rate q which is, so far, estimated to be: $u(q) = 1.0\ \% \times q + 2.5 \times 10^{-9}$ Pa·m³·s⁻¹.

3. IMPLEMENTATION OF THE TECHNIQUE TO IMPROVE LOW LEAK RATES MEASUREMENT

3.1. Introductory statement

In the work [3], we used a capillary leak artefact (CL-H) model TL6 (Figure 3) available from Inficon GmbH (Germany), which flow rate is adjustable by varying the upstream pressure. A delivered rate of 1×10^{-5} Pa·m³·s⁻¹ was obtained with a nitrogen supply pressure of 700 kPa, leading to a ratio of the residual flow rate stability q_{res} to the measured flow rate q, estimated to be lower than 5×10^{-4}. The estimated relative uncertainty in V_d was 0.1 % (that is 0.06 cm³ for $V_d = 60$ cm³). The overall time needed to perform the calibration of V_d was 1 hour.

Should ever we wish to generate a low leak rate with the same leak artefact, it would suffice to reduce the upstream pressure. And, to calibrate it, we would use the dead volume determined with the higher leak rate. This would then shorten the calibration time while simultaneously improving the calibration uncertainty.

To take advantage of this fact in the general case where the leak artefact is not adjustable (or only adjustable to far below 1×10^{-5} Pa·m³·s⁻¹), we have developed a technique using a simple mechanical device.

3.2. Presentation of the technique

The leak artefact CL-H supplied with 700 kPa of nitrogen is denoted by CL-H700. If the latter is connected to the constant-volume flowmeter, in parallel with the leak under calibration the flow rate q of which is lower than 1×10^{-6} Pa·m³·s⁻¹ (corresponding to our definition of low leak rates in section 1), then the resulting nominal flow rate remains roughly equal to that of CL-H700. In this configuration, the dead volume V_d can be calibrated easily and quickly.

The next step of the calibration is to remove the leak CL-H700 from the set-up without changing the value of the measurement volume. To this end, we examined how the value of the constant-volume flowmeter dead volume was affected by the connection of CL-H (Figure 3).

The geometrical part of the artefact connected inside the flowmeter consists in a cylinder welded to an ISO-KF 16 blank flange. A plug occupying approximately the same volume of the artefact can be easily built from a metal cylinder and a blank flange (Figure 4).

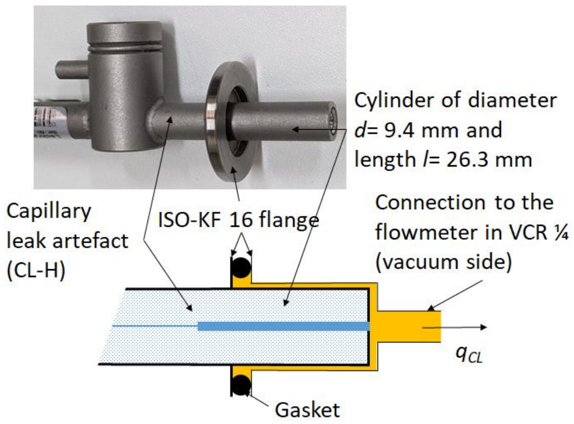

Figure 3: Capillary leak artefact Inficon and its connection to the constant-volume flowmeter. The internal part of the flowmeter, belonging to V_d, is shown in yellow

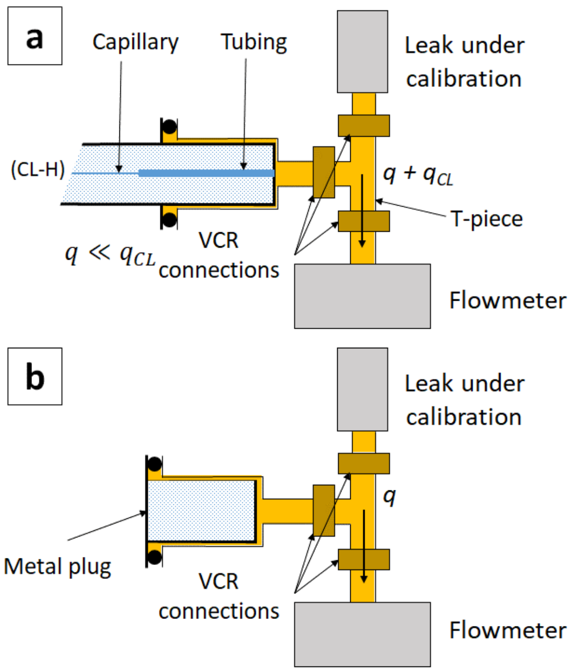

Figure 4: Implementation of the technique for small leak rates measurement

Note that with ISO-KF 16 connections, aluminium gaskets should be used to avoid the outgassing inevitable with elastomer seals, corresponding to the lower region of the decade 10^{-8} Pa·m^3·s^{-1}.

The technique to improve low leak rate measurements is implemented as follows. A T-piece (with VCR®1/4 flanges) is attached to the input port of the flowmeter, so both the leak under calibration (throughput q) and the leak CL-H (throughput q_{CL}) can be connected to the flowmeter (Figure 4, step (a)). During this step, V_d is determined within a short operating time and with the uncertainty $u(V_{d(a)}) = 0.060$ cm^3.

In step (b) (see Figure 4), CL-H is replaced by the metal plug for determining q. It is understood that before performing step (b), a sufficient pumping time estimated to be about 24 hours is respected.

3.3. Provisional uncertainty budget of V_0

Since the study of small leak rate measurements with a constant-volume flowmeter has only just begun, so only the main uncertainty sources have been taken into account in the uncertainty budget of V_0.

With the technique presented in section 3.2, the leak artefact CL-H is replaced by the metal plug, (picture in Figure 5). Leak-tightness for both elements is ensured by an aluminium gasket.

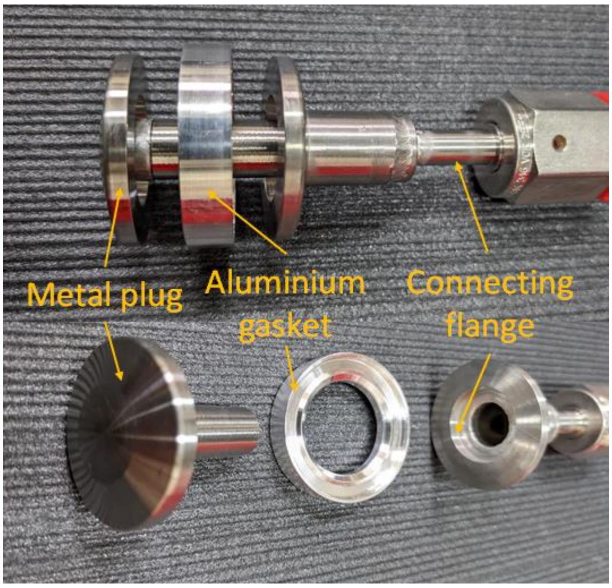

Figure 5: Picture of the metal plug with its connecting flange and aluminium gasket

The measurement volume V_0 with the plug installed on the flowmeter is then deduced from the relation:

$$V_0 = V_{d(a)} + \delta v, \tag{5}$$

where $V_{d(a)}$ is the dead volume determined by measurements during step (a) and δv the difference between $v_{CL\text{-}H}$ and v_{plug} the solid volume occupied respectively by the leak artefact and the metal plug in the constant-volume flowmeter. Since they both consist of a cylinder welded onto the ISO-KF 16 blank flange, the difference in volumes of these cylinders can be determined by dimensional measurements using a calliper. The small tubing through which the gas flows from the capillary to the end of the cylindrical part of the artefact (Figure 4 (a)) is estimated to be 0.6 mm in diameter and 35 mm in length. Its volume is thus $v_{tub} = 0.0099$ cm^3.

A standard measurement uncertainty of 0.01 mm is assumed for the length (≈ 26.3 mm) and the diameter (≈ 9.4 mm) of each cylinder. This yields an uncertainty in a single cylinder volume $u(v_{CL\text{-}H})$ (= $u(v_{plug})$) = 0.0039 cm^3. We also assume independent measurements for each cylinder and so obtain $u(\delta v) = 0.0056$ cm^3 which is hardly significant compared with $u(V_{d(a)})$.

In this uncertainty budget, we neglected to a first approximation:

- the uncertainty in v_{tub} ,

- the thickness difference of the tightened gasket between the configurations of steps (a) and (b), providing the tightening has been performed reproducibly.

Finally:

$$u(V_0) = \sqrt{u\left(V_{d(a)}\right)^2 + u(\delta v)^2}, \tag{6}$$

which leads to $u(V_0) = 0.060$ cm^3.

3.4. Extension of the smaller leak rates range

Now this new technique allows an effective determination of the measurement volume of the constant-volume flowmeter without one's spending too much time, the bottom end of the flowmeter range is limited by two factors. On the one hand the amplitude of the residual rate stability Δq_{res} on a given time interval of a calibration cycle Δt_{cy}, should be as small as possible with respect to the leak rate under calibration. The assumption of $\Delta q_{res} = 3 \times 10^{-10}$ Pa·m^3·s^{-1} ($k = 1$) in section 2.3 is available for Δt_{cy} estimated to be about 3 hours. On the other hand, the pressure variation Δp_q in V_0 produced by the leak rate during the time interval within Δt_{cy} dedicated this measurement[1], must be significantly higher than the resolution of the pressure gauge (CDG).

[1] Δt_{cy} also comprises a stabilisation time after valve closure and a measurement time for the determination of the residual pressure rate $\dot{p}_{res}$.

The most binding effect is the stability of the residual flow rate. Since it has a thermal origin (in a closed volume, the temperature variation produces a pressure variation), its stability must be mastered over the course of a leak rate calibration. This can be achieved by means of a temperature recording along with the pressure recording in combination with the determination of the residual pressure rate before and after the determination of the pressure rate due the leak flow. In a first approach, a standard uncertainty $u(\Delta q_{\mathrm{res}}) = 5.0 \times 10^{-10}$ Pa·m^3·s^{-1} in the stability of q_{res} is realistic.

An extension of the calibration range down to 5×10^{-8} Pa·m^3·s^{-1} with a standard uncertainty of a few percent meets the aforementioned criterion.

4. COMPARISON WITH OTHER WORK

Arai and Yoshida have developed a constant-volume flowmeter for leak rates flowing both to vacuum or atmosphere [4]. In their instrument, the measurement volume (denoted V_{A} in the aforementioned paper) should also be determined prior to the measurement of the leak rate. This is achieved using an additional volume (V_{D}) and performing a series of static expansions with calibrated balls placed successively inside V_{D}. The standard uncertainty finally obtained is $u(V_{\mathrm{A}}) = 0.1$ cm^3 for $V_{\mathrm{A}} \approx 33$ cm^3 (0.3 % in relative value).

In our preliminary study, the uncertainty in the measurement volume is $u(V_0) = 0.060$ cm^3 for $V_0 \approx 60$ cm^3. We did not include in our case other sources studied in [4], namely the volume variation of the capacitance diaphragm gauge when bulged and the volume variation of the valve isolating the volume $V_{\mathrm{st\text{-}d}}$ from V_{d} (Figure 1) between its open and close position. For the former, we estimated with the data in [4] (providing that a similar CDG was used), and for a pressure difference of 50 Pa between the phase of the determination of V_{d} and that of the leak rate calibration, a contribution equal to a maximum of 0.007 cm^3. While the latter has yet to be investigated, it seems nonetheless that both contributions to the uncertainty in V_0 are minor.

We conclude therefore that, given the relative uncertainty in V_0, hardly more than 0.1 %, the technique to improve low leak rate measurement presented in this paper is worthwhile.

5. SUMMARY

The calibration procedure of leak rates with the LNE-Cnam constant-volume flowmeter includes an initial determination of its dead volume V_{d}. This consists in measuring pressure rise rates due to the leak rate, successively in V_{d} then in V_{d} with a standard volume in series. For smaller leak rates, the determination of V_{d} with an adequate accuracy requires several hours.

We have presented an easy-to-implement technique, which reduces drastically the operating time for determining V_{d} and improves its uncertainty. In turn, it allows one to extend the calibration range down to 5×10^{-8} Pa·m^3·s^{-1}.

The technique consists in connecting to the constant-volume flowmeter a leak artefact with a relative high flow rate in parallel to the leak rate under calibration. The resulting flow rate provides a quick and accurate determination of the dead volume of the flowmeter, which is used as the measurement volume for low leak rates.

Although this technique is performed using a certain type of artefact, with a given shape connected inside the flowmeter, one could easily duplicate it with other currently available leak artefacts.

6. REFERENCES

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