On the determination of a piston prover dead volume

Original
On the determination of a piston prover dead volume / Spazzini, Pier Giorgio; Piccato, Aline; La Piana, Gaetano; Santiano, Marco. - In: MEASUREMENT. SENSORS. - ISSN 2665-9174. - (2025). [10.1016/j.measen.2024.101547]

Publisher:
Elsevier

Published
DOI:10.1016/j.measen.2024.101547

Measurement: Sensors 38 (2025) 101547

Contents lists available at ScienceDirect

Measurement: Sensors

journal homepage: www.sciencedirect.com/journal/measurement-sensors

On the determination of a piston prover dead volume

ARTICLE INFO

Keywords:
Dead volume
Piston prover
Uncertainty PACS
47.80.-v
06.20.F-

ABSTRACT

Dead volume in a piston prover is the part of the internal volume which is never occupied by the piston.
It obviously affects measurements performed through the prover, the specific effect depending on the data analysis procedure. In INRIM test rigs, which apply the mass balance method (difference between the estimates of the gas mass at the beginning and at the end of the measurement), the evaluation of the corrections requires knowledge of the initial measurement volume, which depends on the dead volume.
Currently, INRIM evaluates the dead volume by geometrical analysis, which involves a large uncertainty.
We will propose a method for the dead volume evaluation based on the addition of a well-defined quantity of gas (measured by integration of the output of a calibrated MFC) and by measuring the variation of thermodynamic conditions within the piston. The uncertainty budget of the measurement will be analysed in full detail.

1. Introduction

INRIM realizes the primary reference measurements for gas flow rate through use of three test rigs, namely one bell prover and two piston provers. All three devices allow to perform measurements in two modalities, i.e. accepting (gas is flowing towards the accumulating volume) or delivery (gas flows from the accumulating volume).

These devices are fully described, alongside with the validation of their performance, in several papers (see, e.g., Refs. [1–4]).

In all cases, the accurate determination of the gas quantity is based on the so-called mass balance, i.e. the initial and the final gas masses within the containing volume are estimated, then the difference of the two provides the mass variation, which can be used either to determine the gas quantity or the gas flow rate (through division by the measurement time).

We will from now on concentrate on the case of the piston provers, but in the case of the bell measurements the process is essentially the same.

The initial gas estimate is obtained by multiplying the gas density (which is obtained by measurement of the thermodynamic conditions) times the initial volume of gas contained within the cylinder. The latter quantity is the sum of the piston displacement and the dead volume, i.e. the free volume within the cylinder when the piston is at its lowest possible position. In the same fashion, the final mass is obtained by multiplying the gas density times the final volume, which is obtained from the initial one by adding the volume associated to the movement of the piston during the measurement.

It can be shown that the contribution to the uncertainty from the dead volume depends on the value and on the uncertainty of the dead volume itself, and on the difference between initial and final thermodynamic conditions.

The dead volume is therefore an influential quantity and, as such, a source of uncertainty; it is not possible to eliminate it entirely, because is consists of the various mechanical details within the cylinder which are necessary for the correct operation of the machine (e.g. seats for thermometers, connection tubing to pressure measurement devices, free spaces for the passage of mechanical parts, safety spacers etc.) and implicit in the design (e.g. the bottom of the cylinder itself where the piston cannot reach) although a careful design can reduce it to manageable values.

The determination of the dead volume of our pistons is currently obtained through geometrical considerations, i.e. by considering the design dimensions of the various parts and computing the corresponding volumes. Unfortunately, this method includes a large possibility of errors and its associated uncertainty is quite high. For instance, in the case of our small piston prover, which will be used throughout this paper as a test case, the estimate for the dead volume is of 262 cm^3, with an uncertainty of 3 %. The associated contribution to the final measurement uncertainty, due to the small value of the associated sensitivity coefficient, is typically of the order of 0.0025 %, which is of course relatively small but still a noticeable fraction of the total uncertainty (overall standard uncertainty of the test rig: 0.025 %, expanded uncertainty 0.05 %): in the perspective of improving the total uncertainty of measurement, and in consideration of other improvements that are programmed, we also have set the objective of reducing this uncertainty contribution.

Dead volume, and therefore the uncertainty associated to it, can be important also in applications different from the piston prover machine (see, e.g., Refs. [5,6]), therefore the method described could be useful for other applications.

In section 2 of the present paper we will describe the theory underlying the proposed method and the associated equations, while in Section 3 we will face the task of evaluating its uncertainty. In section 4, the experimental setup that we used for testing the concept will be presented, while in Section 5 we will present and discuss the results thus obtained.

https://doi.org/10.1016/j.measen.2024.101547

2. Principle of the method

The method we propose for the evaluation of the dead volume within a piston prover is based very simply on the compressibility properties of a gas and on the ability of accurately measuring the thermodynamic conditions.

If the prover is sealed and the piston is not moving, then adding a known quantity of gas through a suitable gas flow regulator will cause a variation of pressure and temperature, which can be measured and put in relation to the volume to be determined.

2.1. Basic equations

The model definition starts from the perfect gas equation, formulated as follows:

$$pV = nR^*T \tag{1}$$

Where p is the pressure, V is the volume, n is the number of moles, R^* is the specific gas constant and T is the temperature.

For a sealed system in thermal equilibrium (indicated here as the initial condition, and marked with the subscript "$_0$"), equation (1) can be rewritten as:

$$p_0V_0 = n_0R^*T_0 \tag{2}$$

By adding a known quantity of gas and after allowing for stabilization, a new condition (final condition, indicated with the subscript "$_1$") will be reached, in which:

$$p_1V_1 = n_1R^*T_1 \tag{3}$$

Where $V_1 = V_0$ since it is assumed that the piston was held stationary, and R^* is the same in the two cases since the gas is the same; it is therefore possible to obtain, dividing eq. (2) by eq. (3):

$$\frac{n_0}{n_1} = \frac{p_0}{p_1}\cdot\frac{T_1}{T_0} \Rightarrow n_0 = n_1\cdot\frac{p_0}{p_1}\cdot\frac{T_1}{T_0} \tag{4}$$

The gas is added through a calibrated mass flow controller and measured by integration of the mass flow controller signal; since the instrument provides its indication in SCCM, i.e. flow rate in specified thermodynamic conditions, it is possible to compute the number of added moles (n_A) by simply inverting eq. (1) for n, considering the specific conditions and the added volume V_A at those conditions:

$$n_A = V_A\cdot\frac{p_{ref}}{R^*T_{ref}} \tag{5}$$

It is also true that:

$$n_A = n_1 - n_0 \tag{6}$$

Then, substituting eqs. (4) and (5) in eq. (6) one gets:

$$V_A\cdot\frac{p_{ref}}{R^*T_{ref}} = n_1\cdot\left(1-\frac{p_0}{p_1}\cdot\frac{T_1}{T_0}\right) \tag{7}$$

Solving for n_1 and replacing in eq. (3) finally gives the solution for the volume $V_1 = V_0$:

$$V_1 = V_A\cdot\frac{p_{ref}}{R^*T_{ref}}\cdot\frac{R^*T_1}{p_1}\cdot\frac{1}{1-\frac{p_0}{p_1}\cdot\frac{T_1}{T_0}} \tag{8}$$

and, after simplifying:

$$V_1 = V_A\cdot\frac{p_{ref}}{T_{ref}}\cdot\frac{T_1}{p_1}\cdot\frac{1}{1-\frac{p_0}{p_1}\cdot\frac{T_1}{T_0}} \tag{8b}$$

Equation (8b) shows that it is possible to obtain the value of the dead volume from the combination of the thermodynamic conditions and the added volume.

2.2. Determination of the volume V_A

As stated in the previous paragraph, the volume addition was performed through a Mass Flow Controller (MFC, see Section 4 for details of the setup). The calibration of the instrument, performed beforehand, allowed to correct its output and therefore to obtain a reliable flow rate signal. This signal was integrated according to the trapezoidal rule (see e.g. Ref. [7]); despite the relatively high acquisition frequency that could be obtained (≈ 6 Hz), though, the initial and final transients displayed a certain amount of scatter, which harmed the quality of the integration in these phases. This problem was partially overcome by using relatively low maximum flow rates, which allowed long acquisition periods and therefore to reduce the influence of the transients.

Nevertheless, the applied parameters allowed to obtain appreciable repeatability of the computed volume.

3. Uncertainty estimate

In this paragraph the measurement model equation (8b) will be analysed according to the uncertainty propagation methods as described in the GUM document [8]. In the following, a detailed description of each source of uncertainty will be presented:

3.1. Contribution from the added volume V_A

This contribution was evaluated based on the integration uncertainty evaluation method described in Ref. [9]; the method was applied considering the following input values:

- Time uncertainty of 1 ms, rectangular distribution;
- MFC resolution of 0.001 SCCM;
- Flowrate uncertainty from interpolation of the MFC calibration uncertainties at the various flow rates, Gaussian distribution;
- Correlation between flow rate measurements corresponding to the calibration reference uncertainty, i.e. 0.05 %;

The resulting uncertainty $U(V_A)/V_A$ was found to be in all cases of about 0.2 %.

3.2. Contribution from the term $\frac{p_{ref}}{T_{ref}}$

Since both values are not measured but simply reference values, the uncertainty contribution associated to this term is null by definition.

3.3. Contribution from the term $\frac{T_1}{p_1}$

The contribution from this term depends on the uncertainties of the instruments used for the measurement of pressure and temperature, respectively. Regarding the temperature contribution, the following components were considered:

- calibration uncertainty of the thermometer, corresponding to 0.015 K (Gaussian distribution);
- resolution uncertainty of the thermometer, 0.01 K (rectangular distribution);
- fluctuation during the measurement, typically 0.02 K (rectangular distribution).
- Due to the temperature conditioning method (see 4.1) and the averaging over several acquisitions for the initial and final temperature measurements, we consider the contribution from possible spatial inhomogeneity of the temperature to be negligible.

These components were summed in quadrature, providing a total uncertainty of 0.027 K, or in percentage terms 0.009 %.

Correspondingly, for the barometer the following values were considered:

- calibration uncertainty of the barometer, corresponding to 5 Pa (Gaussian distribution);
- resolution uncertainty of the barometer, 0.1 Pa (rectangular distribution);
- fluctuation during the measurement, typically 1 Pa (rectangular distribution).

Hence a total pressure uncertainty of 5.2 Pa, or 0.005 %.

Overall the uncertainty contribution of this term has been estimated at 0.01 %

3.4. *Contribution from the term* $\frac{1}{1-\frac{p_0}{p_1}\cdot\frac{T_1}{T_0}}$

The contribution from this term can be performed in two parts. First of all, the contribution from the pressure/temperature part can be considered.

$\frac{T_1}{p_1}$ can be treated like in 3.3, providing thus the same result, and the same is valid for $\frac{T_0}{p_0}$, since the same instruments are used. The two measurements can be considered independent, therefore the simple rule of summing (in quadrature) the percentage uncertainties can be applied, leading to a total uncertainty of 0.014 %.

On the other hand, the experimental values of the term $\frac{p_0}{p_1}\cdot\frac{T_1}{T_0}$ are quite close to 1, therefore even such a small percentage value will impact heavily on the total uncertainty of the overall term. Assume for example that a value of $\frac{p_0}{p_1}\cdot\frac{T_1}{T_0}=0.98$ is obtained; its absolute uncertainty would therefore be of 0.00014. This would also be the absolute uncertainty on the overall term, whose absolute value is then of 0.02, leading thus to a percentage value of 0.75 %. This is clearly the dominating component of the overall uncertainty; additionally, it strongly depends on the ratio between initial and final pressures.[1]

One way of reducing this uncertainty component is to increase the pressure difference between initial and final conditions.

3.5. *Uncertainty of the dead volume V_1*

Based on the results of the previous paragraphs, it is now possible to compute the overall uncertainty of the dead volume evaluation by applying the basic rule for uncertainty propagation in multiplicative models, i.e. the sum in quadrature of the relative uncertainties.

It comes out that, for a pressure difference of about 2000 Pa, the uncertainty on the computed dead volume V_1 is typically of 0.75 % (see Section 5 for the actual computed values). As will be described in detail in the following, every measurement was repeated three times to check for repeatability; the standard deviation of these measurements was computed, and its value was added (in quadrature) to the average of the uncertainties of the three measurements to account for the repeatability component.

4. Experimental setup

4.1. *Test rig and associated instruments*

Experimental measurements were performed using INRIM's small piston prover [2–4]. This is a test rig of the plunger piston type, with a total capacity of approximately 3000 mL and a very accurate temperature control, obtained by flowing water conditioned by a thermal bath through both the piston and the cylinder walls.

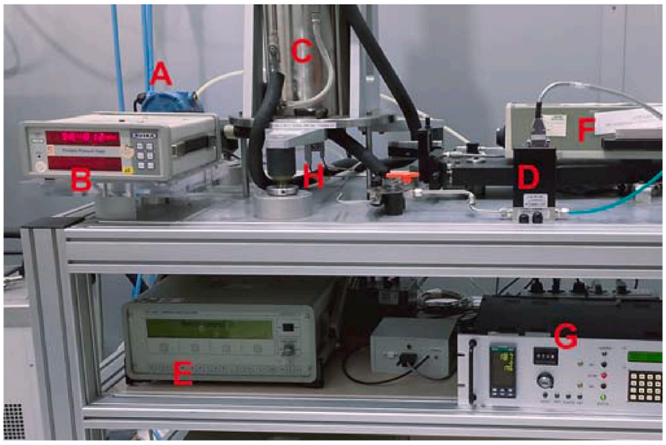

Fig. 1. Experimental setup picture; A: Differential pressure transducer; B: Barometer; C: Piston prover; D: Mass Flow Controller; E: Thermometer; F: Interferometer; G: piston movement control system; H: PT100 probes connections.

The movement of the piston is measured by an interferometer whose overall accuracy is better than 0.5 μm, while the diameter of the piston is traceable to the SI with an uncertainty of 2.5 μm.

The cylinder is also connected to a differential pressure transducer, whose other input is open to the atmosphere; this instrument, in the normal configuration of the test rig, is used as the input to the control system of the piston and ensures that the pressure within the cylinder is equal to the atmospheric pressure. For measurements at very low flow rates, which require long measurement times, variations of the atmospheric pressure could influence the results and therefore, in such cases, the control is based on the output of the barometer, thus maintaining the internal pressure to a preset value.[2]

The present estimate for the dead volume is of 262 mL, with an uncertainty of 3 %, i.e. ≈ 7.9 mL. The test rig is routinely used for the calibration of flow meters at flow rates ranging from 0.1 to ≈ 1500 mL/min with an accuracy as low as 0.05 %.

Pressure within the piston is measured by a RUSKA series 6200 barometer, whose calibration is performed at the pressure laboratory of INRIM, providing an overall uncertainty for the pressure measurements of 5 Pa; temperature is measured by two PT100 probes located in two suitable hollows in the cylinder wall, connected to a CORRADI 7000 thermometer; the thermometric chain is calibrated in the temperature laboratory of INRIM, and the overall accuracy of the chain is of 0.015 K. These instruments were used to record the initial and final thermodynamic conditions, used for the computations described in Section 2.

Fig. 1 presents a picture of the test rig in its typical arrangement.

4.2. *Added volume measurement*

The measurement of the added volume was performed by an MKS Mass Flow Controller, with a maximum range of 5 SCCM, which was controlled by a suitable software program; the acquisition program allowed to set the MFC parameters and steady state flow rate, to command the start of the flow delivery and to record the instrument signal at a prescribed frequency. The MFC was calibrated beforehand using, as reference, the test rig itself. The calibration was carried out using our standard Procedure, which calls for seven calibration points repeated three times.

---

[1] The term depends strongly on the temperature difference also, but as will be discussed in detail in Sec. 4 the temperature within the prover used for this experiment is very stable, therefore the main influence comes from the pressure measurements.

[2] Though, this method cannot be applied for all flow rates because the response time of the barometer is too slow for a satisfactory control at flow rates in excess of ≈ 10 mL/min.

In order to obtain accurate calibration curves, data were processed using INRIM CCC Software for nonlinear regression [10,11]. Fig. 2 reports the calibration data alongside with the plot of the calibration curve thus determined.

This calibration was applied to the measured values of flow to get a more accurate evaluation of the actual flow rate and hence of the totalized volume, which was computed by applying the trapezoidal rule. In order to check the influence of the flow measurements, a series of tests were performed by reaching the same pressure increase with nominal flow rates of 1, 2 and 4 SCCM; results from these tests (not reported here) showed that the flow rate employed did not influence significantly the results. It was decided to maintain the flow rate of 2 SCCM for the final tests (Section 5) because the higher flow rate was considered less reliable due to the shorter duration, and therefore higher influence of the transitories which are the most delicate part of the fluid delivery, while the lower flow rate implied a (slightly) larger uncertainty of the flow measurements.

Before starting the measurements, the MFC was run for about an hour at 50 % of its maximum flow rate in order to stabilize its conditions; on the other hand, in between two successive measurements it was left inactive. Since the time in between two measurements is of the order of 15 minutes in order to guarantee stability of the thermodynamic conditions, it is possible that the internal temperature of the instrument in successive measurements was not perfectly stable, which can contribute to the small drift observed (see 5.2).

5. Results and discussion

Data gathered as described in Section 4 will be presented in this Section, alongside with results obtained from their elaboration.

5.1. Data set description

Five different measurements have been performed, each of which consisted of three repetitions of measurements in the same nominal conditions.

The baseline measurement was performed with the piston completely lowered and in the standard configuration of the test rig, i.e. with the differential pressure transducer (see 4.1) online. However, this transducer has an internal volume and therefore it contributes to the dead volume of the system, thus it would be useful to avoid it when the transducer is not online. From the standpoint of this analysis, moreover, this offers the opportunity of obtaining measurements with different dead volumes in the same conditions; therefore, a second measurement with the piston in the same position has been performed but excluding the differential transducer.

Following this, and to confirm the reliability of the results, the piston has been moved by a known quantity and the measurements were repeated; by subtracting the known volume associated to the piston movement from the volume computed as described earlier, it is expected to obtain the same result for the dead volume.

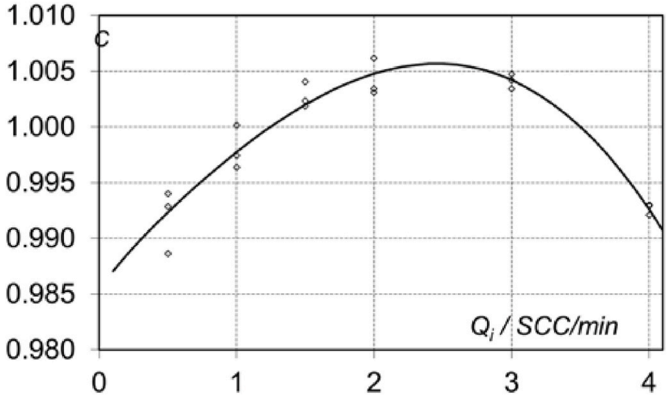

Fig. 2. Calibration of the MFC.

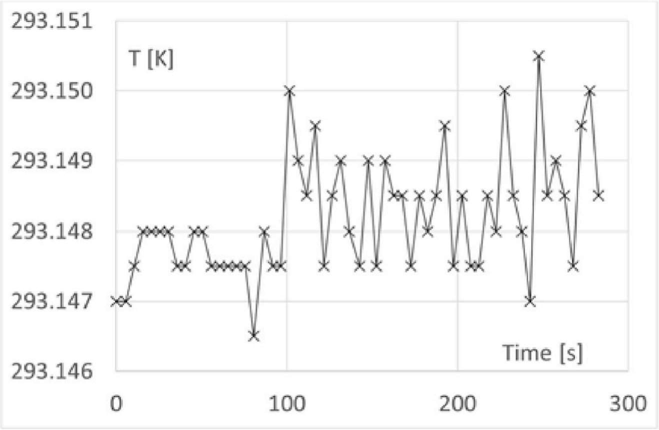

Fig. 3. Temperature recorded during measurement run #2.

In the first test, the piston was moved by about 6 mm (corresponding to a volume of ≈ 70 mL) and measurements were repeated both in the baseline configuration and with the differential transducer excluded from the circuit; in the second test, the piston was moved by about 12 mm, and measurements were performed only in the baseline configuration.

5.2. Rough measurement

In this section some examples of the measurements of pressure, temperature and flow rate are described in detail. Figg 3 and 4 display typical examples, respectively, of the temperature and pressure recorded during a single measurement:

It can be observed that the temperature is stable to well within 0.01 K; the pressure is stable in the first and last part of the measurement (before the start and after the end of the gas delivery), while it displays a regular growth during the operation of the MFC.

In order to demonstrate the pressure stability, in Fig. 5 a detail of Fig. 4 (referring to the final part of the measurement) is presented:

The pressure is shown to be steady to within a few tenths of a Pascal, which confirms the sealing of the piston, and in addition, the accuracy and reliability of the pressure measurements.

Fig. 6, on the other hand, reports the flow rate measurement performed during the same test.[3]

It can be observed that the flow rate, in most of the measurement run, is very stable; on the other hand, both the initial and final transients display slight overshoots, which, albeit being considered in the integration phase, can provide some disturbance to the overall measurements; in order to minimize this disturbance, it was decided to perform measurements having a duration of at least 120 s.

5.3. Results elaboration

Data presented in Sec. 5.2 were subsequently elaborated to produce estimates for the dead volume according to what described in Section 2, and their uncertainty was estimated as described in Section 3. The results thus obtained are summarized in Table 1.

The first observation that can be made is that the uncertainty

---

[3] The two measurements were not synchronized; flow rate measurements were generally started only slightly before launching the MFC, which was done after the stability of pressure and temperature in the initial part were confirmed.

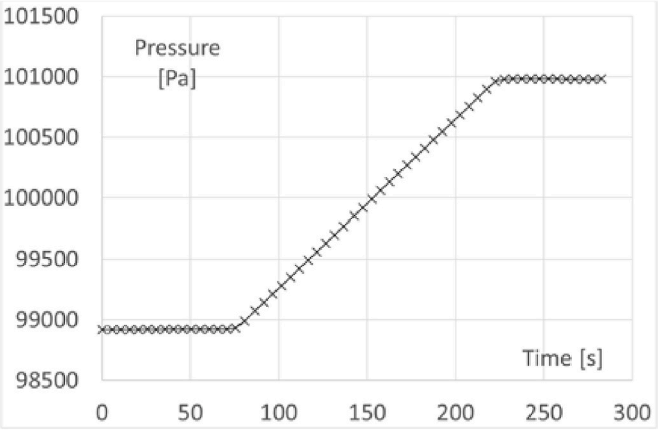

Fig. 4. Pressure recorded during measurement run #2.

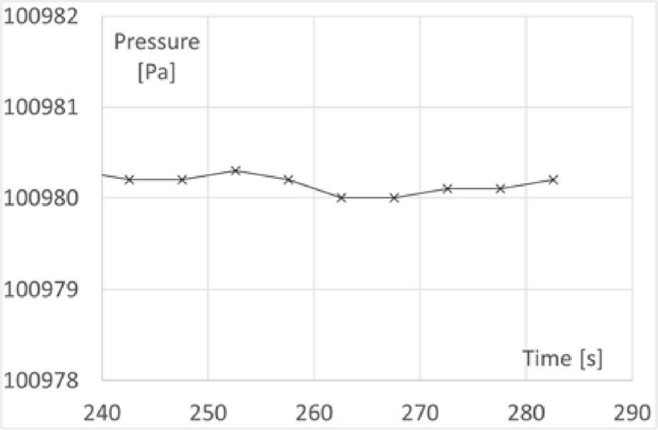

Fig. 5. Detail from Fig. 4.

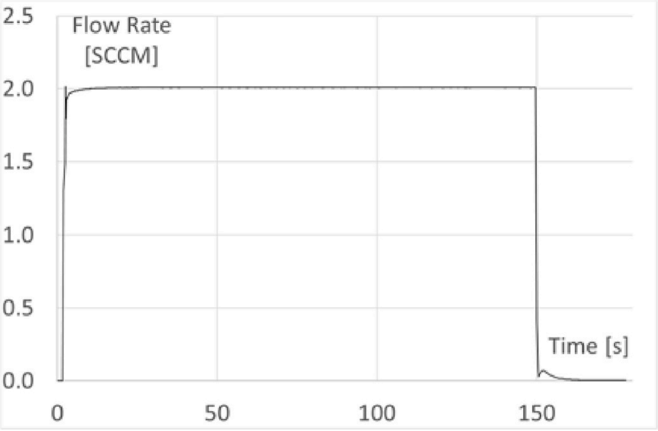

Fig. 6. Flow rate measurements during run #2, nominal flow rate 2 SCCM.

associated to the dead volume, in all cases, is largely lower than the currently applied value of 7.9 mL (see Sec. 4). This proves the validity of the method, which allows in this case an almost fourfold reduction of the uncertainty.

Also, the obtained results, for the cases including the differential pressure transducer (tests #1, #3 and #5) are well compatible with the currently applied value of 262 mL.

Another observation that can be made is that the results obtained in corresponding cases are well compatible with each other, and that the uncertainty in all cases is essentially the same; the small deviations between the different measurements presumably are due to a small zero-drift effect that was observed on the MFC during measurements, possibly associated to relatively long periods of inactivity of the instrument as described in 4.2.

Table 1
Experimental results and associated uncertainties, all tests.

Test #	Computed Volume (mL)	Expanded Uncertainty (%)	Expanded Uncertainty (mL)
1	261.70	0.76	1.98
2	243.24	0.75	1.83
3	261.26	0.76	1.99
4	242.61	0.75	1.82
5	260.38	0.77	2.00

The consistency of the results in all tests shows that the method is reliable and therefore, after another round of tests for check and verification, such results will be applied to the piston prover at INRIM. From the data at hand, the expected value of dead volume to be applied is considered to be 261 mL with an associated uncertainty of 1 % (increased with respect to the computed value for safety) in the case of the baseline configuration, and 243 mL associated to the same relative uncertainty in the case when the pressure transducer is not connected.

The impact of this modification will be quite reduced in itself but, in conjunction with other improvements in progress for the test rig [4], should bring the overall uncertainty of the test rig to a measurable reduction, which will be inserted in the laboratory CMCs once validated.

6. Conclusions

The present paper presented an experimental method for the accurate evaluation of the dead volume in sealed volumes. The principle of the method is very simple, but its correct application calls for a very accurate measurement of the thermodynamic conditions and a precise control of the additional gas quantity that is the basis of the principle.

It was shown that the method, when applying the correct parameters, actually allows to reduce the uncertainty associated to the dead volume by a factor of three, although the impact on the overall uncertainty of the test rig will be lower due to the small sensitivity coefficient associated to this uncertainty component.

Nevertheless, the application of the method in synergy with work on other uncertainty components is expected to allow a reduction of the claimed uncertainty associated to CMCs.

Shortly a new set of measurements will be performed as a check of the results presented here, and in a successive phase the same method will be applied also to the other piston prover available in the INRIM Gas Flow Laboratory.

References

[1] G. Cignolo, A. Rivetti, G. Martini, F. Alasia, G. Birello, G. La Piana, The national standard gas provers of the IMGC-CNR, in: Proceedings of FLOMEKO 2000, 2000, p. 8.

[2] F. Berg, G. Cignolo, Nist-imgc comparison of gas flows below one litre per minute, Metrologia 40 (2003) 154–158.

[3] G. Cignolo, F. Alasia, A. Capelli, R. Goria, G. La Piana, A primary standard piston prover for measurement of very small gas flows: an update, Sens. Rev. 25 (1) (2005) 40–45.

[4] P.G. Spazzini, A. Piccato, P. Pedone, Reduction of a gas prover uncertainty, in: Proceedings of the 10th International Symposium on Fluid Flow Measurement (ISFFM), Queretaro, Mexico, 2018.

[5] J.A. Bocanegra, F. Scarpa, V. Bianco, L.A. Tagliafico, The effect of dead volumes on the performance of magnetic refrigerators, Int. J. Refrig. 151 (2023) 26–38.

[6] V. Grosfils, R. Hanus, A. Vande Wouver, M. Kinnaert, Parametric uncertainties and influence of the dead volume representation in modelling simulated moving bed separation processes, J. Chromatogr. A 1217 (47) (2010) 7359–7371, https://doi.org/10.1016/j.chroma.2010.09.030.

[7] P.J. Davis, P. Rabinowitz, in: Methods of Numerical Integration, second ed., Academic Press, USA, 1984.

[8] I.E.C. Bipm, I.L.A.C. Ifcc, I.U.P.A.C. Iso, O.I.M.L. Iupap, Evaluation of measurement data - guide to the expression of uncertainty in measurement, Joint Committee for Guides in Metrology, JCGM 100 (2008) 2008.

[9] P.G. Spazzini, W. Bich, Uncertainty in discrete-time integration — the case of static gas meters, Measurement 224 (2024) 113821.

[10] M. Lecuna, F. Pennecchi, A. Malengo, P.G. Spazzini, Calibration curve computing (ccc) software v2.0: a new release of the inrim regression tool, Meas. Sci. Technol. 31 (11) (2020) 114004, https://doi.org/10.1088/1361-6501/ab7d6e.

[11] A. Malengo, F. Pennecchi, P.G. Spazzini, Calibration Curves Computing – CCC Software, 2015 available for download at, http://www.inrim.it/download/CCC/index.html. Release 1.3.

Pier Giorgio Spazzini*, Aline Piccato, Gaetano La Piana, Marco Santiano
INRIM, Strada delle Cacce, 91, Torino, 10135, Italy

* Corresponding author.
E-mail addresses: p.spazzini@inrim.it (P.G. Spazzini), a.piccato@inrim.it (A. Piccato), g.lapiana@inrim.it (G. La Piana), m.santiano@inrim.it (M. Santiano).