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# PVTt primary flow standard for small gas flow rates

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**Abstract**

A PVTt primary flow standard operates on the principle of determining the change of density of the measured gas in the tank of a known volume and the corresponding time interval. The PVTt standard presented in this paper is based on the diverter-operated flying start and finish method. It contains the gas collection tank that is constructed as a dismountable assembly, which enables determining its internal volume by dimensional measurements. The standard is designed for the flow range from 0.12 mg/min to 12 mg/min and it achieves the relative expanded measurement uncertainty between 0.12% and 0.24%. We performed a comparison of the realized PVTt flow standard with the piston-prover flow standard for flow rates above 1.2 mg/min, and the results were found successful in view of normalized errors, $E_n$.

## 1. Introduction

The general principle of the operation of a pressure, volume, temperature, and time (PVTt) primary gas flow standard is based on determining the change of density of the measured gas in the tank of a known volume and the corresponding time interval [1–4]. The measured gas flow can be collected in the measuring tank (increase of gas density) or generated from the measuring tank (decrease of gas density). The PVTt standards usually operate in the flying start-and-finish method, which assure nearly constant flow rate through the device under test. This method can be designed with static or dynamic measurements of the fluid density.

This paper deals with the realization of the PVTt primary gas flow standard, which is based on the static (diverter-operated) flying start-and-finish method. The system is designed for the volume flow rates between 0.1 cm$^3$/min and 10 cm$^3$/min at standard gas conditions; i.e., for the mass flow rates of the air-like gas between 0.12 mg/min and 12 mg/min. The core of the standard is the collection measuring tank, which is constructed as a dismountable assembly, so that its internal volume can be determined directly by dimensional measurements. The metrological characteristics of the realized PVTt standard were compared with the piston prover gas flow standard, but so far only for flow rates above 1.2 mg/min, which is the minimum flow limit of the employed piston prover.

The paper is organized as follows: Section 2 presents a configuration of the measurement system of the PVTt flow standard; Section 3 discusses main contributions to the measurement uncertainty and evaluates the expanded uncertainty of the measured mass flow rate; and Section 4 introduces the results of a comparison between the PVTt standard and the piston prover.

## 2. Measurement system

A top view of the realized PVTt flow standard is presented in Figure 1.

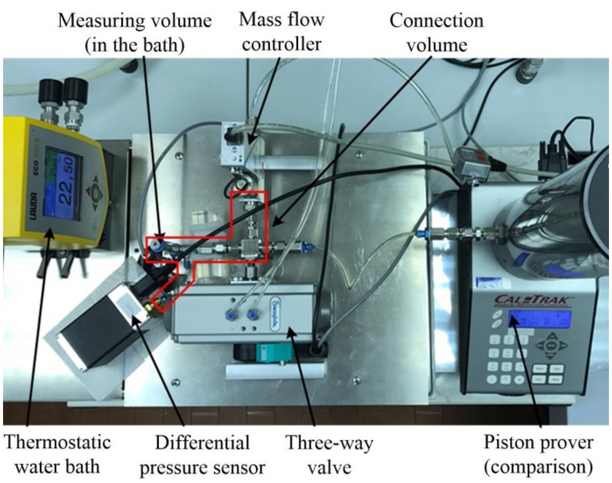


**Figure 1:** Top view photo of the PVTt measurement system.

The main part of the collection volume is the stainless steel cylindrical tank of about 100 cm$^3$, which is placed into the thermostatic water bath (Lauda ECO Gold) and is for that reason not visible in this photo. The PVTt standard requires a stable mass flow rate of the measured gas, which is ensured by the thermal flow controller (Bronkhorst F-201CV). The gas flow is diverted into or out of the collection tank by the three-way valve positioned downstream of the flow controller. The valve is equipped with a double-acting pneumatic actuator, the position of which is controlled by using solenoid valves. An electrical output of the valve position sensor is used to trigger the measurement of the time interval, which is performed by the DAQ board (National Instruments USB-6341).

The filling of the tank starts at the ambient pressure conditions and finishes when the gauge pressure reaches the target value. The gauge pressure is measured by the differential pressure sensor positioned at the entrance to the tank (Mensor CPT6100), and the ambient pressure is measured by the absolute pressure sensor (Mensor CPG2500). Temperature is measured by the resistance temperature sensor positioned in the tank wall (TetraTec WIT-S + PicoTech, PT-104). Gas density is calculated for the stationary conditions before and after the filling stage by using the REFPROP fluid properties database [5]. The maximum pressure in the collection volume is limited by the upper range limit of the gauge pressure sensor that is 2500 Pa.

The PVTt standard is automated by using a LabVIEW acquisition and programming environment.

## 3. Measurement model and uncertainty

The mass flow reading of the presented PVTt gas flow standard is calculated by the following measurement model:

$$q_m = \frac{V \Delta\rho}{\Delta t}, \tag{1}$$

where $V$ is the collection volume, $\Delta t$ is the effective collection time interval, and $\Delta\rho = \rho_2 - \rho_1$ is the difference of gas densities in the collection volume after and before the filling stage.

The measuring volume of the gas $V$ is the whole gas collection volume downstream of the mass flow controller and can be written as a sum of the volume of the tank $V_e$ and the connection (dead) volume $V_d$ between the mass flow controller and the tank including the internal volume of the gauge pressure sensor; i.e., $V = V_e + V_d$. The internal volume of the tank was determined by traceable dimensional measurements of the internal diameter and length. The connection volume was measured by the gas expansion method using the tank as the reference volume. The tank and connection volumes are $V_e$ = 99.6928 cm$^3$ and $V_d$ = 7.5738 cm$^3$, respectively, so the total measuring volume equals $V$ = 107.2666 cm$^3$. The relative standard uncertainty of the measuring volume is estimated to $u(V)/V = 4.0 \cdot 10^{-4}$. The largest contribution represents the uncertainty of the connection volume, estimated as experimental standard deviation of the measurement results.

The effective collection time interval $\Delta t$ is determined as a sum of the time interval measured by the DAQ board $\Delta t_m$ and the diverter correction time $\Delta t_d$. The diverter correction was determined at different flow rates by approximately following the procedure in [6]. If the constant mass flow rate is measured for two different collection times, we assume:

$$q_\mathrm{m} = \frac{V \Delta\rho_1}{\Delta t_{m,1} + \Delta t_d} = \frac{\Delta\rho_2}{\Delta t_{m,2} + \Delta t_d}, \tag{2}$$

therefore the diverter correction time can be estimated as:

$$\Delta t_d = \frac{\Delta\rho_1 \Delta t_{m,2} - \Delta\rho_2 \Delta t_{m,1}}{\Delta\rho_2 - \Delta\rho_1}. \tag{3}$$

The average value of the diverter correction time is $\Delta t_d$ = 0.533 s and its standard measurement uncertainty, estimated as experimental standard deviation of the results, is $u(\Delta t_d)$ = 0.015 s. The standard uncertainty of the effective time interval $u(\Delta t)$ is calculated as the root-sum-square of $u(\Delta t_d)$ and the standard uncertainty of the measured time interval $u(\Delta t_m)$, where the latter is estimated to $u(\Delta t_m) = 3 \cdot 10^{-5}\, \Delta t_m$.

Gas density is determined by using the REFPROP database for the selected gas and the time-averaged measured values of the absolute pressure (calculated as a sum of the ambient pressure and the gauge pressure) and the temperature. The relative standard measurement uncertainty of the density change is estimated as:

$$\frac{u(\Delta\rho)}{\Delta\rho} = \sqrt{\left(\frac{u(\Delta p)}{\Delta p}\right)^2 + \left(\frac{u(T)}{T}\right)^2 + \left(\frac{u(M/Z)}{M/Z}\right)^2}, \tag{4}$$

where $u(\Delta p)$ = 0.5 Pa is the standard measurement uncertainty of the pressure change, $u(T)$ = 0.1 K is the standard measurement uncertainty of the temperature, and $u(M/Z)$ considers uncertainties of the gas composition and the density model (for the dry air used in measurements in this paper the last term in Eq. (4) is estimated to $2 \cdot 10^{-4}$).

Furthermore, leak testing was performed for the collection volume using the pressure decay method. The leakage mass flow rate was found to not exceed $q_{m,l} = 1.2 \cdot 10^{-4}$ mg/min, therefore, $u(q_{m,l}) = q_{m,l}/\sqrt{3}$.

The relative combined standard measurement uncertainty of the mass flow rate is evaluated as:

$$\frac{u(q_m)}{q_m}=\sqrt{\left(\frac{u(V)}{V}\right)^2+\left(\frac{u(\Delta t)}{\Delta t}\right)^2+\left(\frac{u(\Delta\rho)}{\Delta\rho}\right)^2+\left(\frac{u(q_{m,l})}{q_m}\right)^2}\,. \tag{5}$$

Finally, the expanded measurement uncertainty of the mass flow rate measured by the PVTt standard is estimated as:

$$U(q_m)=k\,u(q_m)\,, \tag{6}$$

using the coverage factor $k$ for 95.45% confidence interval of a $t$-distribution with effective degrees of freedom obtained from the Welch-Satterthwaite formula [7]. Finite degrees of freedom were considered for uncertainty components associated with the connection-volume and correction-time measurements.

Figure 2 shows the relative expanded measurement uncertainty of the mass flow rate, $U_r(q_m) = U(q_m)/q_m$, for the pressure changes of 2500 Pa and 1000 Pa. The corresponding time interval is nearly 1600 s at 0.12 mg/min and nearly 16 s at 12 mg/min for the 2500 Pa pressure change, and it is 2.5-times lower for the 1000 Pa pressure change.

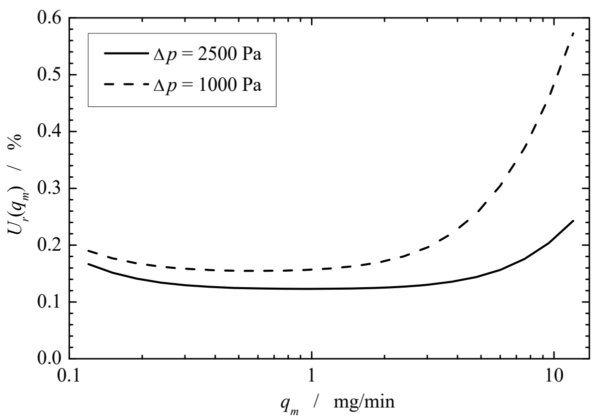


**Figure 2:** Variation of the relative expanded measurement uncertainty with the mass flow rate of dry air for two different pressure changes.

For the maximum pressure change of 2500 Pa the expanded uncertainty varies from 0.12% to 0.24% of the measured mass flow rate. The largest contributions to the uncertainty are associated with the leakage effects at the smallest flow rates and the time-interval measurements at the highest flow rates. The volume and temperature measurements are the largest contributions to uncertainty for the intermediate flow rates. For the pressure change of 1000 Pa the expanded uncertainty increases to the values between 0.15% and 0.57% of the measured mass flow rate. Such an increase is the result of relatively larger effects related to the pressure and time measurements.

## 4. Comparison results

The realized PVTt gas flow standard was tested by comparison with the piston prover gas flow standard (Sierra Instruments Cal=Trak SL-800), which has the flow range between 1.2 mg/min and 600 mg/min and the expanded measurement uncertainty of $U(q_m)$ = 0.015 mg/min + $2.5{\cdot}10^{-4}$ $q_m$ [8]. Because of the minimum flow limit of the employed piston prover, we performed a comparison in the flow range of 1.2 mg/min to 12 mg/min. The piston prover was connected to a free port of the three-way diverter valve of the PVTt standard. The measurements were carried out alternately with both flow standard and were repeated three times at each flow rate of dry air. The PVTt standard was used with the collection time corresponding nearly to the maximum pressure change of 2500 Pa.

Figure 3 shows relative deviations between the mass flow readings of the PVTt system and the piston prover (index "*PP*"),

$$e_r=\frac{q_{m,PVTt}-q_{m,PP}}{q_{m,PP}}\,, \tag{7}$$

and the relative expanded uncertainties associated with measurements by both standards, $U_r(q_{m,PVTt})$ and $U_r(q_{m,PP})$. The observed relative deviations are within ±0.19% for the largest four flow rates and within ±0.41% for the smallest two flow rates. In all cases the deviations do not exceed the expanded uncertainty of the piston prover.

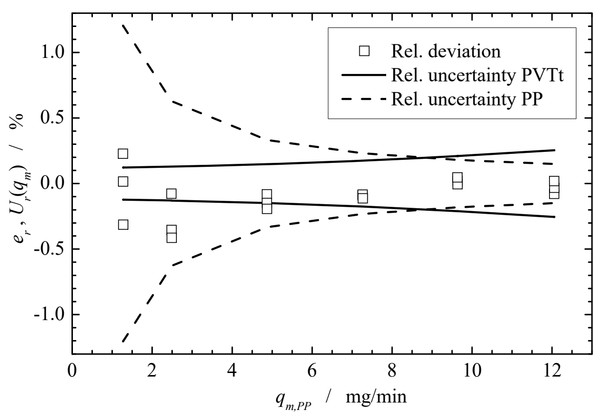


**Figure 3:** Relative deviations between the mass flow readings of the PVTt standard and the piston prover, and relative expanded uncertainties associated with both standards.

The comparison results are also evaluated in terms of normalized errors:

$$E_n = \frac{q_{m,PVTt} - q_{m,PP}}{\sqrt{U\left(q_{m,PVTt}\right)^2 + U\left(q_{m,PP}\right)^2}} . \qquad (1)$$

As shown in Figure 4, the normalized errors lie between –0.65 and 0.19. Because all $E_n$ values are within the ±1 range, the comparison results can be considered as statistically satisfactory. At least at higher flow rates, where the uncertainties of both standards are of the same order of magnitude, these results can be considered as a confirmation of metrological characteristics of the realized PVTt gas flow standard.

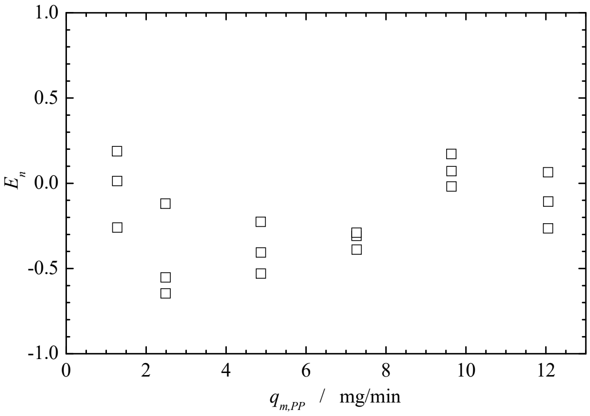


**Figure 4:** Results of a comparison between the PVTt system and the piston prover in terms of normalized errors.

## 5. Conclusions

The aim of this paper was to present a realization of the PVTt primary flow standard, which is based on the diverter-operated flying start-and-finish method. The standard is designed for the flow range of 0.12 mg/min to 12 mg/min and it achieves the relative expanded measurement uncertainty between 0.12% and 0.24% when used with the maximum pressure change of about 2500 Pa. The largest contributions to the measurement uncertainty are associated with the leakage effects at the smallest flow rates and the time-interval measurements at the highest flow rates. The volume and temperature measurements are the largest contributions to measurement uncertainty for the intermediate flow rates.

The metrological characteristics of the realized PVTt standard were compared with the piston prover gas flow standard, but so far only for flow rates above 1.2 mg/min, which is the minimum flow limit of the employed piston prover. The comparison results evaluated in terms of normalized errors are found to be statistically satisfactory, with $E_n$ values between –0.65 and 0.19. Nevertheless, one should bear in mind that the uncertainties of the piston prover are relatively large at its minimum flow limit.

## References

[1] Wright J D, Johnson A N, Moldover M R, Kline G M, *Gas flowmeter calibrations with the 34 L and 677 L PVTt standards* (Gaithersburg, MD: NIST), 2003.

[2] Nakao S, "Development of the PVTt system for very low gas flow rates", *Flow Measurement and Instrumentation* 17 (2006) pp. 193-200.

[3] Morgado I, Legras J C, Clodic D, "Primary standard for the calibration of refrigerant leak flow rates", *Metrologia* 47 (2010) pp. 135-145.

[4] Levine P D, "A new approach to gas flow calibration", *Measurement* 43 (2010) pp. 1644-1648.

[5] Lemmon E W, Huber M L and McLinden M O, *NIST Reference Fluid Thermodynamic and Transport Properties – REFPROP Version 9.0* (Gaithersburg, MD: NIST), 2010.

[6] ISO 4185: *Measurement of liquid flow in closed conduits – Weighing method*, 1980.

[7] JCGM 100: *Evaluation of Measurement Data – Guide to the Expression of Uncertainty in Measurement*, 2008.

[8] Bobovnik G, Kutin J, Bajsić I, "Uncertainty analysis of gas flow measurements using clearance-sealed piston provers in the range from 0.0012 g min$^{-1}$ to 60 g min$^{-1}$", *Metrologia* 53 (2016) pp. 1061–1068.