Establishment of an Ultra-High Accuracy 670 PVTt Gas Flow Primary Standard at NMIA

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Abstract

A new PVTt standard for gas flow has been commissioned at NMIA, which relies on measurements of pressure, volume, temperature and time. The main aim of developing this new standard was to reduce the ±1000 ppm (±0.10%) uncertainty of measurements made with NMIA's bell and mercury-sealed piston provers. The uncertainty associated with measuring the mass flowrate using the new PVTt standard is estimated to be ±116 ppm (±0.012%), this significant improvement in uncertainty can be attributed to two reasons. Firstly, the volumes of existing provers at NMIA were measured to no better than ±400 ppm, whereas the volume of the new PVTt standard has been determined gravimetrically using water with an uncertainty of ±80 ppm. Secondly, existing provers are used in ambient air with a spatial temperature uniformity of 150 mK, while the new PVTt standard is immersed in a temperature controlled water tank with a temperature uniformity of 2 mK. In this paper, a description of the PVTt standard is presented. A comprehensive uncertainty analysis is also made and an example calibration is described.

1. Introduction

A 300 L bell prover and a set of 5 mercury-sealed piston provers are currently used at the National Measurement Institute Australia (NMIA) as primary standards for gas flowrates ranging from 1 cc min^{-1} to 25 m^3 h^{-1} with a least uncertainty of ±0.10% [1]. There are many advantages and disadvantages of using bell and mercury-sealed piston provers as primary flow standards and to state all of them is beyond the scope of this paper. However, some of the disadvantages are (1) safety concerns due to the use of mercury and oil as seals in these standards, (2) the limitation on using these standards for measurement with pressures higher than atmospheric due to the oil and mercury liquid seals, (3) difficulty in reducing the large spatial temperature non-uniformity in and around these standards while used in ambient air, which is currently assessed to be 150 mK, and (4) difficulty in determining the volumes of these provers to better than 400 ppm.

These bell and mercury-sealed piston provers are mainly used in the calibration of critical flow Venturi nozzles (CFVN). These CFVNs are then combined in parallel to be used in the calibration of other flow measuring devices for flowrates up to 7000 m^3 h^{-1} (normalised at 20°C and 101.3 kPa conditions). The current uncertainty associated with using these nozzle arrays is ±0.13% ($k = 2$). This measurement uncertainty is obtained from a two-tier build-up calibration that relies on existing provers to attain. NMIA is planning to expand its calibration services to include high pressure and high flow ranges in the near future while maintaining the competitive uncertainty needed to service the requirements of local industry. To establish measurement traceability of the planned high pressure facility, existing CFVN arrays will be used in the calibration of other CFVNs at higher upstream pressures. To achieve the needed uncertainty, a standard with a higher operating range of flowrates and pressures and reduced uncertainty is therefore needed.

A 670 L PVTt gas flow standard has recently been designed and constructed at NMIA. The aims of constructing this standard are to (1) replace the existing bell and mercury-sealed piston provers, (2) increase the range of flowrate up to 120 kg h^{-1}, and (3) improve the measurement uncertainties. This new standard is based on measurements of pressure, volume, temperature and time; hence the acronym PVTt.

In this paper, a description of the NMIA's PVTt standard is presented including the methodology followed in its design. A measurement uncertainty analysis is also included.

2. The PVTt Standard

2.1 Design and Mode of Operation

Several national metrology institutes around the world employ PVTt standards for ultra-high accuracy measurements of gas flowrate [2,3,4]. At NMIA, a PVTt standard with a nominal volume of 670 L has been designed and constructed as an ultra-high accuracy gas flowrate standard. The design of the PVTt standard is based on the same principles adopted by other laboratories but with several significant improvements which will be described here.

The PVTt system consists of eight annular stainless steel cylinders connected together by a circular pipe, shown schematically in Figure 1.

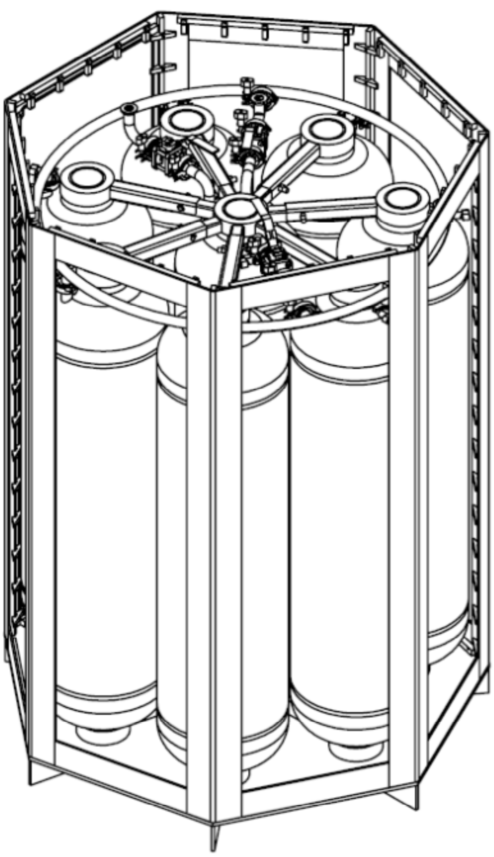

Figure 1. Schematic diagram of NMIA's PVTt standard.

A valve is used to separate the centre cylinder from the surrounding cylinders to enable two volume options with nominal values of 80 L or 670 L. The cylinders and connecting pipe-works are immersed vertically inside a tank filled with distilled water to achieve tight temperature uniformity and stability. NMIA's new design of annular cylinders allows heat transfer from the water to both the outer and inner stainless steel surfaces and then to the gas inside, hence reducing the time constant for transient heat transfer between the water and the gas. In addition, having all cylinders placed vertically inside the tank, as opposed to horizontally, improves the convective heat transfer along the cylinder surfaces and allows for faster stabilisation of the gas inside the cylinders.

A 3-way diverter valve with built-in limit switches is used to divert the flow from the DUT to the PVTt tank or from the DUT to atmosphere. The limit switches on the diverter valve are powered by a 24 Vdc signals that are used in conjunction with a high accuracy timer to record the time interval between the start and stop of gas flow into the PVTt tank. An example of the calibration setup of a CFVN using the PVTt standard is shown in Figure 2.

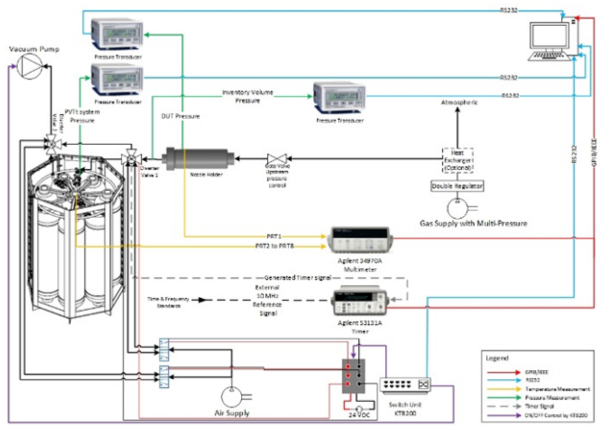

Figure 2. Setup for calibration of a critical flow Venturi nozzle using NMIA's PVTt standard.

The steps to conduct a measurement using the PVTt standard are: (1) evacuate the tank to the required pressure, (2) wait for the pressure and temperature to stabilise; it was observed that 30 minutes is sufficient, (3) record pressure and temperature of the tank at start conditions, (4) open the diverter valve so that the flow is from the DUT to the tank, (5) collect readings from the DUT; for a CFVN the collected readings are the upstream pressure and temperature (and relative humidity if using air), (6) close the diverter valve so that the flow is from the DUT to the atmosphere, (7) wait for the pressure and temperature to stabilise, and (8) record the pressure and temperature at end conditions.

The measurement procedure has been fully automated with a PC and custom software to minimise user errors and streamline data collection.

Usually, the measurements are conducted several times to establish a measure of their repeatability and reproducibility.

2.2 Volume Determination

The uncertainty of the tank volume is vital as it is a major component in the total uncertainty budget of the flowrate measurement. To minimise this component, the cylinders were designed to facilitate measuring their volumes gravimetrically using water. The volumes of the eight cylinders were measured individually with an uncertainty of ±80 ppm. All plumbing inventories used to connect the cylinders together and to the device under test were also measured gravimetrically using water with an uncertainty of ±100 ppm. The small volume of these inventories compared to that of the eight cylinders means that the effect of their uncertainty on the total volume uncertainty is minimal and a total uncertainty of the full volume can therefore be maintained at ±80 ppm.

To validate the total volume measurement using the gravimetric method with water, $V_{\mathrm{PVTt,water}}$, a second method using nitrogen gas was conducted. In this method, a known mass of nitrogen gas is deposited into the PVTt tank; this mass of nitrogen is discharged from a gas cylinder with its weight measured before and after by NMIA's chemical metrology group. Prior to depositing the nitrogen, the PVTt tank is evacuated to a very low pressure (<100 Pa) then allowed to stabilise; stable pressure readings are a good indication of steady state conditions of the gas inside the PVTt tank. After depositing the nitrogen, the tank is allowed to stabilise again. Pressure inside the PVTt volume and water temperature are measured at steady state conditions before and after the deposition of nitrogen. Using these measurements, the volume of the PVTt standard, $V_{\mathrm{PVTt,N_2}}$, is calculated using the following equation:

$$V_{\mathrm{PVTt,N_2}} = \frac{m_{\mathrm{N_2}}}{\rho_{\mathrm{e}} - \rho_{\mathrm{s}}} - V_{\mathrm{Inv,N_2}} \qquad (1)$$

where $m_{\mathrm{N_2}}$ is the measured mass of nitrogen deposited into the PVTt tank, ρ_{s} and ρ_{e} are the densities of nitrogen calculated from temperature and pressure measurements of the tank before and after depositing the nitrogen(e.g.: start and end), and $V_{\mathrm{Inv,N_2}}$ is the volume of the plumbing inventory used to connect the nitrogen cylinder to the PVTt tank.

As can be seen from Equation (1), the measurement uncertainty of $V_{\mathrm{PVTt,N_2}}$ is mainly influenced by (1) the uncertainty associated with measuring the mass of the nitrogen, $m_{\mathrm{N_2}}$, and (2) the uncertainty in calculating the nitrogen densities, ρ_e and ρ_s. The measurement uncertainty associated with $m_{\mathrm{N_2}}$ was determined by NMIA's chemical metrology group to be ±33 ppm. The uncertainty associated with the densities are dominated by the uncertainty in the equation of state obtained from [5] to be ±100 ppm (at 95% C.L.). On the other hand, the uncertainty contribution of $V_{\mathrm{Inv,N_2}}$ is considered to be negligible due to its small size (10 mL) when compared to the volume of the PVTt tank. In estimating the uncertainty in $V_{\mathrm{PVTt,N_2}}$, a more conservative approach was adopted by considering all of its components to have non-correlated uncertainties. A value of (663.415 L ± 221 ppm) was obtained for $V_{\mathrm{PVTt,N_2}}$. This value compares with $V_{\mathrm{PVTt,water}}$ of (663.358 ± 80 ppm). Both volume determinations are plotted in Figure 3 for easier comparison. A more comprehensive analysis of the volume measurement $V_{\mathrm{PVTt,N_2}}$ and its uncertainty is presented in [6].

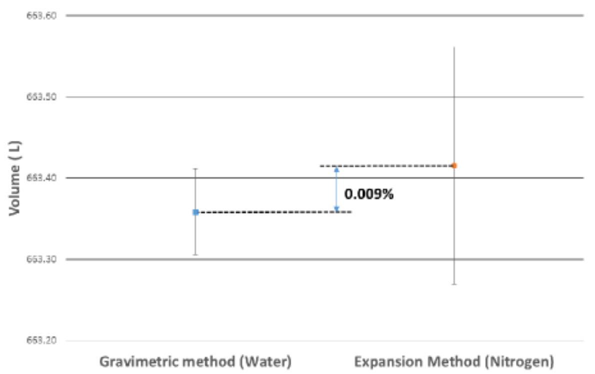

Figure 3. Comparison of the NMIA's PVTt tank volume measurements, with their error bars, using water and nitrogen.

As can been seen from Figure 3, measurements of the PVTt volume using water and nitrogen agree well within their estimated uncertainties. A difference of 90 ppm (or 0.009%) is observed with $V_{\mathrm{PVTt,water}}$ having lower uncertainty. Consequently, the value obtained from the gravimetric technique using water, $V_{\mathrm{PVTt,water}}$, is used as the PVTt tank volume.

2.3 Water Tank Temperature Control

As mentioned before, the stability and uniformity of temperature of the gas inside the cylinders is a major source of uncertainty in the flowrate measured using the PVTt standard. To minimise this contribution, the PVTt tank is immersed inside a water tank maintained at fixed temperature. The temperature of the water tank is controlled by using 400-watt heater elements connected to a PID controller with temperature read by an SPRT (Standard Platinum Resistance Thermometer) in its feedback loop. Dry air is bubbled throughout the water tank to (1) induce faster circulation and mixing of water hence increasing the convective heat transfer over the stainless steel surfaces of the cylinders, and (2) create cooling effect caused by the evaporation of water into the dry air, which is needed to act as a balance to the heater elements. Measurements of water temperature were recorded over a period of 15 hours and are plotted in Figure 4.

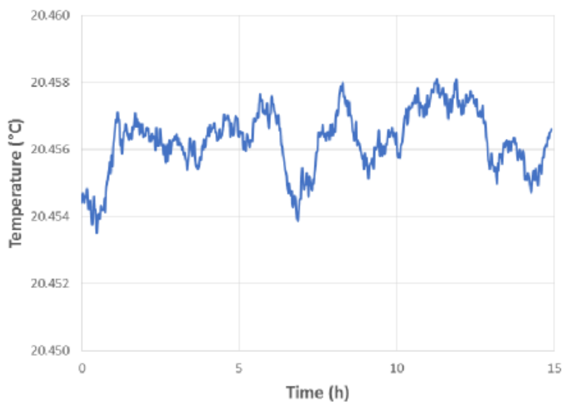

Figure 4. Plot of the NMIA's PVTt water tank temperature over a period of 15 hours.

As can be seen from Figure 4, the water temperature is stable within 2 mK with a standard deviation calculated to be 0.9 mK. In addition, seven PRTs placed at various positions inside the tank were used to assess the spatial temperature uniformity. Measurements conducted showed less than 2 mK of spatial temperature variation inside the water tank.

It can be concluded that the contribution of the temperature stability and uniformity to total uncertainty is negligible. This is a major improvement upon NMIA's existing standards, bell and mercury sealed piston provers, in which temperature stability and uniformity were a major source of their uncertainties.

3. Uncertainty Analysis

3.1 Model of the Measurement

Using the principle of conservation of mass, the mass flowrate measured by the PVTt, Q_m, is calculated using the following equation:

$$Q_m = \frac{(m_{PVTt}^e - m_{PVTt}^s) + (m_{Inv}^e - m_{Inv}^s)}{t} \quad (2)$$

where m_{PVTt}^e is the mass of the gas inside the PVTt volume at end conditions, m_{PVTt}^s is the mass of the gas inside the PVTt volume at start conditions, m_{Inv}^e is the mass of gas inside the inventory volume at end conditions, m_{Inv}^s is the mass of the gas inside the inventory volume at start conditions, and *t* is the time between the start and end of gas collection. Using the relation between mass (m), volume (V) and density (ρ), Equation (2) can be rewritten to give:

$$Q_m = \begin{bmatrix} V_{PVTt}(\rho_{PVTt}^e - \rho_{PVTt}^s) + \\ V_{Inv}(\rho_{Inv}^e - \rho_{Inv}^s) \end{bmatrix} / t \quad (3)$$

where V_{Inv} is the inventory volume between the diverter valve and the DUT. Note that the superscripts in Equation (3), e and s, denote the end and start conditions. The densities, ρ_{PVTt}^e and ρ_{PVTt}^s, are calculated from measurements of pressure and temperature in the PVTt tank. On the other hand, the densities, ρ_{Inv}^e and ρ_{Inv}^s, are calculated from measurements of pressures and temperatures in the inventory volume.

3.2 Uncertainty Analysis

The measurement uncertainty of Q_m can be determined based on the model given in Equation (3) using the root-sum-square of all its components with the assumption that these components are non-correlated. However, this may result in an overestimation of the total uncertainty as some of these components are fully or highly correlated. To calculate the uncertainty of a mathematical model, y, consisting of correlated components, x_i and x_j, the following equation can instead be used [7]:

$$u_c^2(y) = \sum_{i=1}^{N} c_i^2 u^2(x_i) + 2\sum_{i=1}^{N-1}\sum_{j=i+1}^{N} c_i c_j u(x_i) u(x_j) r(x_i, x_j) \quad (4)$$

in which $r(x_i, x_j)$ is the 'correlation coefficient' that expresses the degree of correlation between the input quantities, x_i and x_j. For a measurand with all of its components being non-correlated, the value of $r(x_i, x_j)$ is 0. In this case Equation (4) is simplified to the root-sum-square of these components or simply the case of considering all uncertainty components to be non-correlated. On the other hand, for a model with fully correlated uncertainty components, the value of $r(x_i, x_j)$ is either 1 or -1.

From Equation (3), the components V_{PVTt}, V_{Inv} and t are obtained by various measurement methods and therefore considered to have non-correlated uncertainties, hence $r(x_i, x_j) = 0$. On the other hand, the two sets of densities, $(\rho_{PVTt}^e, \rho_{PVTt}^s)$ and $(\rho_{Inv}^e, \rho_{Inv}^s)$, are calculated from measurements of pressure and temperature in conjunction with a published equation of state [5]. It can be safely assumed that the measurement uncertainties for each set of densities are fully correlated since they are all measured using the same equipment and the same equation of state to calculate their values, hence $r(x_i, x_j) = 1$; same concept work has been adopted by other researchers [2,3,4]. A more conservative approach is adopted herein by considering these densities to have highly correlated uncertainties with $r(x_i, x_j) = 0.95$.

It follows from the above that the uncertainty in Q_m, or u_{Q_m}, can be calculated using:

$$u_{Q_m} = \sqrt{\begin{aligned} &\left(c_{V_{PVTt}} u_{V_{PVTt}}\right)^2 + \left(c_{\rho^e_{PVTt}} u_{\rho^e_{PVTt}}\right)^2 + \\ &\left(c_{\rho^s_{PVTt}} u_{\rho^s_{PVTt}}\right)^2 + \left(c_{V_{Inv}} u_{V_{Inv}}\right)^2 + \\ &\left(c_{\rho^e_{Inv}} u_{\rho^e_{Inv}}\right)^2 + \left(c_{\rho^s_{Inv}} u_{\rho^s_{Inv}}\right)^2 + \left(c_t u_t\right)^2 \\ &+2 c_{\rho^e_{PVTt}} c_{\rho^s_{PVTt}} u_{\rho^e_{PVTt}} u_{\rho^s_{PVTt}} r\left(\rho^e_{PVTt}, \rho^s_{PVTt}\right) \\ &+2 c_{\rho^e_{Inv}} c_{\rho^s_{Inv}} u_{\rho^e_{Inv}} u_{\rho^s_{Inv}} r\left(\rho^e_{Inv}, \rho^s_{Inv}\right) \end{aligned}} \quad (5)$$

where the terms c and u are the corresponding sensitivity factors and uncertainty components of each term in Equation (3) respectively.

As can be deduced from Equation (5), it is expected that u_{Q_m} is minimised when the mass collected in the PVTt tank, $(m^e_{PVTt} - m^s_{PVTt})$, is much larger than the inventory mass difference between the start and end of gas collection, $(m^e_{Inv} - m^s_{Inv})$. The inventory volume was therefore designed to be as small as possible, less than 20 mL. This volume is measured to ±1% (or ±0.2 mL) equating to 0.3 ppm of V_{PVTt}. At the same time, having a small inventory volume reduces the effect of pressure variation on the mass of gas collected when switching the flow from the DUT to the PVTt tank.

3.3 Example Calibration

A CFVN with a nominal diameter of 2 mm was connected in series with the PVTt standard. The starting pressure in the PVTt tank was set to ~100 Pa. Dry nitrogen, with a purity better than 99.999% produced by the boil-off of liquid nitrogen at NMIA cryogenic facility, was allowed to flow into the PVTt tank through the CFVN for a period of 420 s giving an end pressure of ~39 kPa. The temperature of the tank's water was set to 20.450°C and this set point was maintained within ±0.9 mK. Measurements of the PVTt tank pressures and water temperatures were recorded. These measurements were repeated seven times. Based on these values, a list of the uncertainty components associated with Q_m is given in Table 1.

The uncertainties of Q_m at 66%, u_{Q_m}, and 95%, U_{Q_m}, Confidence Limits (C.L.) are also reported in the table; note 66% C.L. and 95% C.L. correspond to coverage factors of $k = 1$ and $k = 2$.

Table 1: Uncertainty components associated with the measurement of Q_m using NMIA's PVTt standard for a given measurement scenario.

Gas used: dry nitrogen, N_2
Starting Pressure = 100 Pa, End Pressure = 39×10³ Pa
Temperature = 20.450°C,
V_{PVTt} = 0.663 358 m³, V_{Inv} = 18×10⁻⁶ m³, t = 420 s

Components	$\boldsymbol{u}$(@1SD or k=1)		Source
		ppm	
$\boldsymbol{V_{PVTt}}$	27 mL	40	Cal report
$\boldsymbol{\rho^e_{PVTt}}$			
Pressure	5 Pa	132	[6]
Temperature	6 mK	21	[6]
Equation	2.2×10⁻⁵ kg m⁻³	50	[5]
$\boldsymbol{\rho^s_{PVTt}}$			
Pressure	5 Pa	131	[6]
Temperature	6 mK	0.06	[6]
Equation	6.2×10⁻⁸ kg m⁻³	0.2	[5]
$\boldsymbol{V_{Inv}}$	0.18 mL	0.1	[6]
$\boldsymbol{\rho^e_{Inv}}$			
Pressure	5 Pa	0.004	[6]
Temperature	6 mK	<0.001	[6]
Equation	2.3×10⁻⁵ kg m⁻³	0.002	[5]
$\boldsymbol{\rho^s_{Inv}}$			
Pressure	5 Pa	0.004	[6]
Temperature	6 mK	<0.001	[6]
Equation	2.3×10⁻⁵ kg m⁻³	0.001	[5]
$\boldsymbol{t}$	5×10⁻³ s	12	[6]
if all uncertainties are non-correlated $\boldsymbol{u_{Q_m}}$(k = 1, r = 0) = 182 ppm			
Let r = 0.95 *(highly correlated uncertainties)*			
$\boldsymbol{u_{Q_m}}$(k = 1, r = 0.95) = 57.8 ppm			
$\boldsymbol{U_{Q_m}}$ (k = 2, r = 0.95) = 116 ppm			

The reported pressure uncertainties in Table 1 are the results of combining (1) the calibration uncertainty of the pressure transducers used, (2) contribution from any hysteresis using these transducers, and (3) any fluctuation of pressure readings during measurements. Similarly, all temperature uncertainties reported are the results of combining (1) the calibration uncertainty of the PRTs used, and (2) uncertainties due to fluctuations of these temperature readings during measurements. On the other hand, the uncertainty associated with time measurement consists of (1) contribution from the high precision digital timer used, which has been calculated to be 10^{-9} s (negligible), and (2) contribution from time delay caused by the mechanical switching of the diverter valve, which was measured as 5 ms [6].

Other measurement uncertainty components associated with the volume of the plumbing that connects the PVTt tank to various instruments and the small volume of the tank not submerged in water are not listed in Table 1 due to their small size. The uncertainty contribution to U_{Q_m} associated with this plumbing volume is estimated to be <1 ppm [6].

On the other hand, the CFVN is calibrated by calculating a nozzle coefficient given by the following equation [8]:

$$N = \frac{Q_m}{\sqrt{\rho_N p_N}} \qquad (6)$$

where ρ_N and p_N are the density and pressure of the dry nitrogen at the nozzle's upstream conditions respectively. The density is calculated from temperature and pressure measurements at upstream nozzle conditions [5]. The uncertainty is then calculated based on this mathematical model. Table 2 gives a list of uncertainty components associated with calculating N. The uncertainties of N at $k = 1$, u_N, and $k = 2$, U_N, are also included in the table.

Table 2: List of uncertainty components associated with the measurement of the CFVN's nozzle coefficient.

Components	$\boldsymbol{u}$(@1SD or k=1)		**Source**
		ppm	
$\boldsymbol{Q_{m,STD}}$	3.9×10^{-8} kg s^{-1}	58	Table 1
$\boldsymbol{\rho_N}$			
Pressure	7.9 Pa	39	Combined
Temperature	4 mK	7	Combined
Equation	5.9×10^{-5} kg m^{-3}	25	[5]
$\boldsymbol{p_N}$	7.9 Pa	39	Combined
Repeatability (7 trials)	1.4×10^{-10} m^2	15	Measured
$u_N(k = 1, r = 0)$ = 85.5 ppm			
$U_N(k = 2, r = 0)$ = 171 ppm			

From the table, the resultant uncertainty, u_N or U_N, is calculated based on the assumption that all uncertainty components are non-correlated ($r = 0$). It can also be noted that uncertainties of the mass flowrate and pressure are major contributors. The repeatability value of 15 ppm is the calculated standard error of the average of the seven measurements collected with a standard deviation of 40 ppm.

Conclusions

Improvement of flowrate uncertainty associated with NMIA's bell and mercury-sealed piston provers has been achieved using the newly commissioned PVTt standard. The measurement uncertainty has been improved by almost a factor of 10, from 1000 ppm (or 0.10%) to 116 ppm. This improvement is mainly credited to (1) having a well determined volume with an uncertainty of ±80 ppm, and (2) a volume with stringent temperature control and uniformity of ±2 mK.

Further improvements on the uncertainty of Q_m reported in Table 1, can be achieved by (1) reducing the measurement uncertainty associated with measuring the tank pressure by using more accurate transducers, and (2) conducting further investigations on increasing the correlation among various components from highly correlated , $r = 0.95$, to fully correlated, $r = 1$.

Improvements on the measurement uncertainty of calibrating a CFVN, reported in Table 2, can also be made by (1) employing better pressure transducers, and (2) placing the CFVN in a more uniform and better controlled temperature environment; for example inside a temperature controlled water tank.

Although a preliminary comparison with NMIA standards has shown good agreement with existing provers within the specified uncertainties [6], a formal comparison that involves other national measurement institutes around the world might be necessary to establish more confidence in the claimed measurement uncertainty using the NMIA PVTt standard.

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