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A NEW PRIMARY GAS FLOW STANDARD FOR FLOW RATE MEASUREMENT FROM 0.001 TO 1000 *NANO mol/S*

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Abstract: A primary gas flow standard was designed and developed in SPRING for the calibration of gas flow meters or leak rate standard at nano-flow rate range of 10^{-6} to 10^{-12} mol/s. It is also used to generate very low gas flow for SPRING's continuous expansion vacuum primary standard. The primary standard consists of two piston-cylinder flow units with diameters of 10mm and 25mm, which provide wider measurement ranges. A stepping motor is used to drive the piston moving at precise speed via a digital micrometer. Two methods, constant pressure method and constant volume method[1][2], are used for the measurement of flow rate. Flow meters can be calibrated automatically using a software program developed at SPRING. The expanded measurement uncertainty of the primary gas flow standard is estimated to be 1% of the readings.

Keywords: Nanofluidics, Gas flow, Primary Standard, Measurement Uncertainty.

1. INTRODUCTION

The measurements of gas flow at very low flow rate are often required in the biomedical, pharmaceutical as well as semiconductor industries for the detection and control of gas leakages[2][6]. Another application is in the generation and measurement of the gas flow required for the continuous expansion primary vacuum standard[1][2][3][8], in which the vacuum level is derived from the inlet gas flow rate, the pumping speed and the conductance of an orifice[3][4][6][8]. A continuous expansion vacuum primary standard was established recently at SPRING for defining the vacuum level, and accurate measurement of flow rate is critical for lowering the measurement uncertainty of this primary vacuum standard. In addition, the new gas flow standard is also required for the calibration of leak rate standard and gas flow meters between 0.001 and 1000 nano-mol/s[7]. The primary standard set up at SPRING is shown in Fig. 1. The principle of operation, system design and performance as well as its uncertainty evaluation are given in this paper.

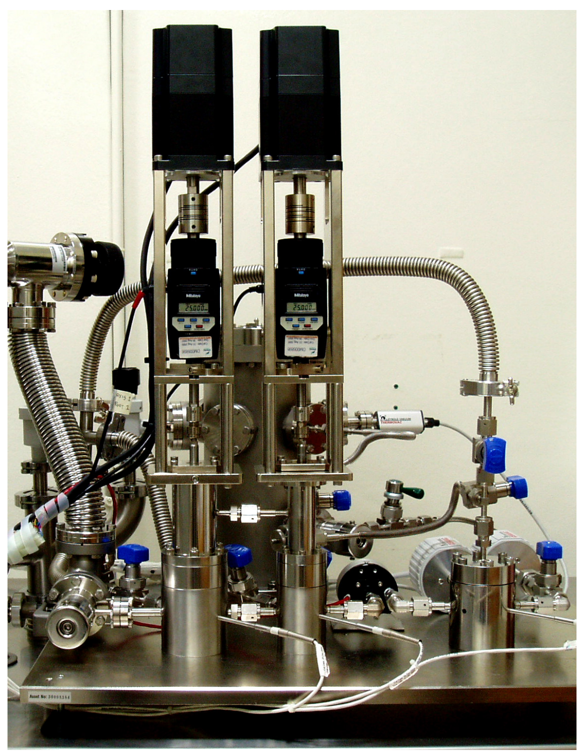


Fig. 1 The primary low gas flow standard of SPRING Singapore

2. PRINCIPLE OF THE FLOW STANDARD

Considering an enclosure of volume V with a small outlet filled with a particular gas, the total number of molecules can be expressed as:

$$N = \frac{P \cdot V}{R \cdot T} \qquad (1)$$

Where: *N(mol)* is the number of molecules in moles; *P(Pa)* is the pressure; $V(M^3)$ is the volume of flow meter; *T(K)* is the temperature; *R* is the ideal gas constant *(J/mol·K).*

The gas flow rate passing through the outlet is given by:

$$dN/dt = \frac{P}{R \cdot T} \cdot (dV_i/dt) + \frac{V}{R \cdot T} \cdot (dP/dt) - \frac{P \cdot V}{R \cdot T^2} \cdot (dT/dt) \qquad (2)$$

Where: *dt* is the time interval of flow rate measurement. d*P (Pa)* is the drop pressure in flow meter; d$V(M^3)$ is the piston displacement volume in the measured time intervals.

In practice, the definition of flow rate of the primary gas flow standard at nano-flow rate range can be realized by two methods which can be expressed mathematically as follows:

Constant pressure method (*dP*=0):

$$dN/dt = \frac{P}{R \cdot T} \cdot (dV/dt) - \frac{P \cdot V}{R \cdot T^2} \cdot (dT/dt) \qquad (3)$$

The temperature variation during a measurement is controlled within 0.1^{o}C, so that the $P*V/R*T^2(dT/dt)$ is insignificant to the *dN/dt*. We counted it as measurement uncertainty value rather than doing a correction. The equation (4) therefore can be simplified as:

$$dN/dt = \frac{P}{R \cdot T} \cdot (dV/dt) \qquad (4)$$

If the pressure and temperature in the gas enclosure as well as the rate of change of its volume can be accurately determined, the flow rate can be determined from equation from equation[2] (4).

In the same way, the equation (2) can be simplified as (5) when the constant volume method (*dV*=0) is used:

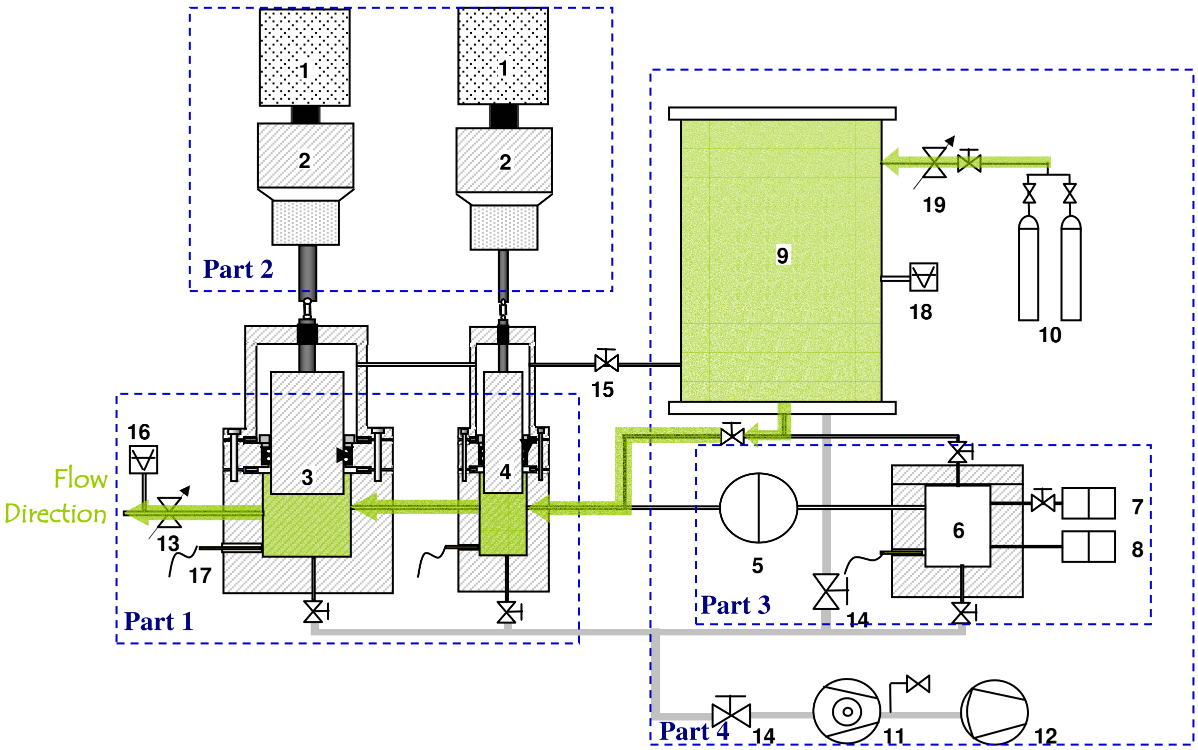
$$dN/dt = \frac{V}{R \cdot T} \cdot (dP/dt) \qquad (5)$$

If the volume and temperature in the gas enclosure as well as the rate of change of its pressure can be accurately determined, the flow rate can be determined from equation from equation[2] (5).

3. DESIGN AND OPERATION

3.1. Design of the Primary Standard

Fig. 2 shows the schematic diagram of SPRING's primary gas flow standard. The system can be grouped into four parts: *Part 1*: the chamber of gas flow meters and flow rate regulator, *Part 2*: stepper motors and micrometers, *Part 3*: constant volume and pressure measurement employing a differential pressure gauge 2 CDGs (Capacitance Diaphragm Gauge), *Part 4*: ballast volume and UHV vacuum pumping system.



1.Stepper motor, 2.Micrometer, 3.ϕ25mm piston flow meter, 4.ϕ10mm piston flow meter, 5.Differential pressure sensor, 6.Reference pressure volume, 7.10 Torr pressure sensor, 8.1000Torr pressure sensor, 9. Ballast tank, 10. Gas cylinders, 11. Turbomolecular pump, 12. Diaphragm pump. 13. All metal leak valve. 14. Angle valve 15. Diaphragm valve. 16. Penning Gauge, 17. PRT probe. 18.Pirani gauge. 19. All metal leak valve.

***Fig. 2** Schematic diagram of the primary gas flow standard*

The gas flow meters (3, 4 in Fig2) are two similar piston-cylinder type flow meters with ϕ10mm and ϕ25mm in diameters. Details of the ϕ25mm piston flow meter is shown in Fig 3. The flow meters are made from 316 stainless steel. All surfaces of the flow meters are electro-polished. The flow meter volume is the cylindrical tank at the lower section of the flow meter with ϕ35mm x 50mm for ϕ25mm piston flow meters and ϕ16mm x 50mm for ϕ10mm piston flow meters. They are covered by the annular shape flanges at the top. The pistons are sealed with sliding O-rings which are mounted at the internal edge of the annular shape flanges. The seals are enhanced by the guard pressure which is maintained at the upper volume of the flow meter (see Fig3). The volume for keeping the guard pressure is connected to the ballast tank (9 in Fig2). The guard pressure is always maintained to be the same as the pressure in the ballast tank which in turn is approximately the same as the pressure in the flow meters. The connections between flow meters, ballast tank and valves are though ϕ6mm stainless steel pipes which are kept as short as possible to minimize volume ratio to the flow meter volume.

The diameters of the pistons have been calibrated and the measured values are ϕ9.9894 mm and ϕ24.9743 mm, respectively. The uniformity in diameters and roundness were found to be better than 0.8 µm. One side of the piston is connected to the micrometer through a shaft linkage (with 50 mm in length) and a coupling rod. The sliding seal for the linkage shaft sliding into or out of the guard pressure volume also helps maintain the alignment of the piston along its axis of motion.

Two digital micrometers (2 in Fig2) are employed to drive the pistons and measure the traveling distances. The micrometers have a measuring range of 50 mm and uncertainty of 0.001 mm. The traveling distances of the pistons are automatically acquired through the RS232 interface of the micrometers. The micrometers are driven by two stepper motors (1 in Fig2) which were selected based on two factors: i) the stepper motor should have a maximum driving force of at least 20 times higher than the torque force caused by friction between sliding seals and pistons in order to minimize possible step lost; ii) it should also have higher resolution in motion steps and wider range of rotating speed so that the traveling speed of pistons can be controlled with sufficient accuracy and sensitivity and the pressure drop can be accurately compensated in order to keep the pressure in the flow meters as constant. The stepper motors are controlled using a 2-Axis motion control card made by National Instrument. Software programs have been developed to operate the motors and drive the pistons (via micrometers) at speeds ranging from 0.01 μm/s to 2 mm/s. The uncertainty of effective piston moving speed driven by the stepper motors is better than 0.01 %. The minimum resolution of the stepper motor is 0.005 μm per step. Both motors can be driven simultaneously, so that the measurement range can be extended to higher flow rate by combining the movement of both pistons. The motion control software obtains feedback from a differential pressure sensor which provides data on the variation of the differential pressure with time. This information is used to guide the motion speed of the pistons to maintain the pressure in the flow meters constant.

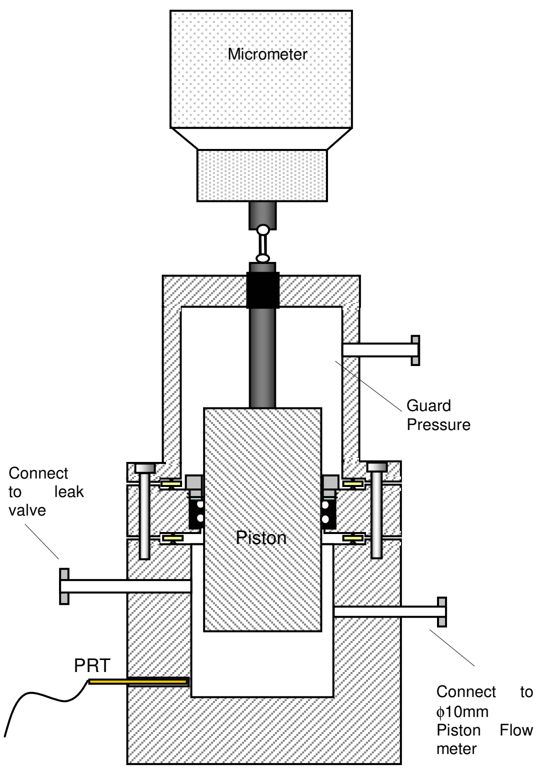


Fig.3 *The ϕ25mm piston gas flow flow meter*

Three 4-wire PT100 probes (17 in Fig 2), supported by Agilent 34970A data acquisition unit, are employed to measure the temperature of two gas flow meters and the reference volume. The temperature probes are inserted into the ϕ3mm channels at each volume as shown in Fig 3. The end of channels is located near the internal surface within 1mm. The temperatures are obtained via an IEEE communication interface for the calculation of flow rate.

The wall thicknesses of the flow meters (3 in Fig2) are from 20 mm to 25 mm. This helps maintain the temperature in each volume within 0.1 °C for about 3 hours under our laboratory conditions, which is sufficient to make a single flow rate measurement. The pressure stability in the flow meter is however very sensitive to the temperature variation, particularly at high pressure. As an additional measure to improve temperature stability, the entire system is placed in an enclosure made of 10-mm persplex. The enclosure also has the effect of improving the uniformity of pressure between the flow meters and the reference pressure volume.

The design of the reference volume (6 in Fig2) for pressure measurement is essentially the same as the flow meters. The interior volume is 62.3 ml. It is connected to a ballast tank (9 in Fig2) and the flow meter with ϕ6mm stainless steel pipes. During measurements, all valves connecting to other parts of the primary standard are closed, keeping the interior pressure in the reference volume constant under stable temperature. Two capacitance vacuum gauges (7,8 in Fig2) having a measurement range from 1.3 to 133000 Pa are mounted on the reference volume to measure the pressure. A differential pressure sensor (5 in Fig2) with a range of 100 Pa and resolution of 0.001Pa is connected between the flow meter and the reference volume to measure the pressure differences between the two sections. The pressure in the gas flow meter can then be calculated from:

$$P_{\text{flow meter}} = P_{\text{reference volume}} + P_{\text{difference}} \qquad (6)$$

A 3 liter ballast tank is used for buffering pressure in the standard and maintaining stable flow rate for extended periods. High purity Nitrogen or Argon are commonly used for the measurement. An all-metal leak valve (19 in Fig2) regulates the gas flow rate by discharging the gas into the ballast tank to set desired pressure level. Pressures in the ballast tank can be set from 1×10^{-2} to 1300 mbar. A calibrated Pirani vacuum gauge (18 in Fig2) was mounted onto the ballast tank to monitor the pressure. The ballast is connected to the high vacuum pump system using ϕ35mm pipes to evacuate the gas from ballast before each measurement. The leak valve is also used to regulate the flow rate by refilling the gas in ballast whenever pressure drops by more than 0.1%. The pressure in the ballast tank can be manually stabilized within 0.1%.

A NI6601 time & frequency PCI card is used for time interval measurement. The triggering of time is accomplished in two ways depending on the measurement method being used. When the constant pressure method is used, time is triggered by start and stop positions of piston motion. Since the resolution of the stepper motor is much higher than that of the micrometer by about 150 times, and it updates faster than the digital micrometers which communicates with PC via RS232, it is advantageous to use the step pulse rather than the signal from micrometers to trigger the time counter. The micrometer is used to check the position only before and after triggering at start and stop positions. When the constant volume method is used, the output signal from the differential pressure sensor is used for triggering the time counter.

A vacuum pumping system (11,12 in Fig2), which consists of a 70L/s turbo-molecular pump and a diaphragm forepump, is used to generate high vacuum in the entire systems. The volumes and ballast tanks are connected to the vacuum system using ϕ16mm stainless flexible hose and ϕ35 mm tube. Because only metal seals are used for all connections, the standard can be baked up to 250 °C. However, the flow meters are not baked due to the rubber sliding seal being used. Baking of the standard is accomplished by using an 1KW heating band warped on the standard. A Penning gauge (16 in Fig2) mounted at outlet position of the flow meter measures the pressure while the flow meter is being pumped to the high vacuum level. The pressure can often reach $1x10^{-5}$ Pa after about 24 hours of baking and pumping.

The software for automatic control and collection of data was developed by SPRING using Labview 7.1. This multiple-functions program was designed to allow the automatic calibration of flow meter and calculation of the flow rate. The program structure is shown in Fig. 4.

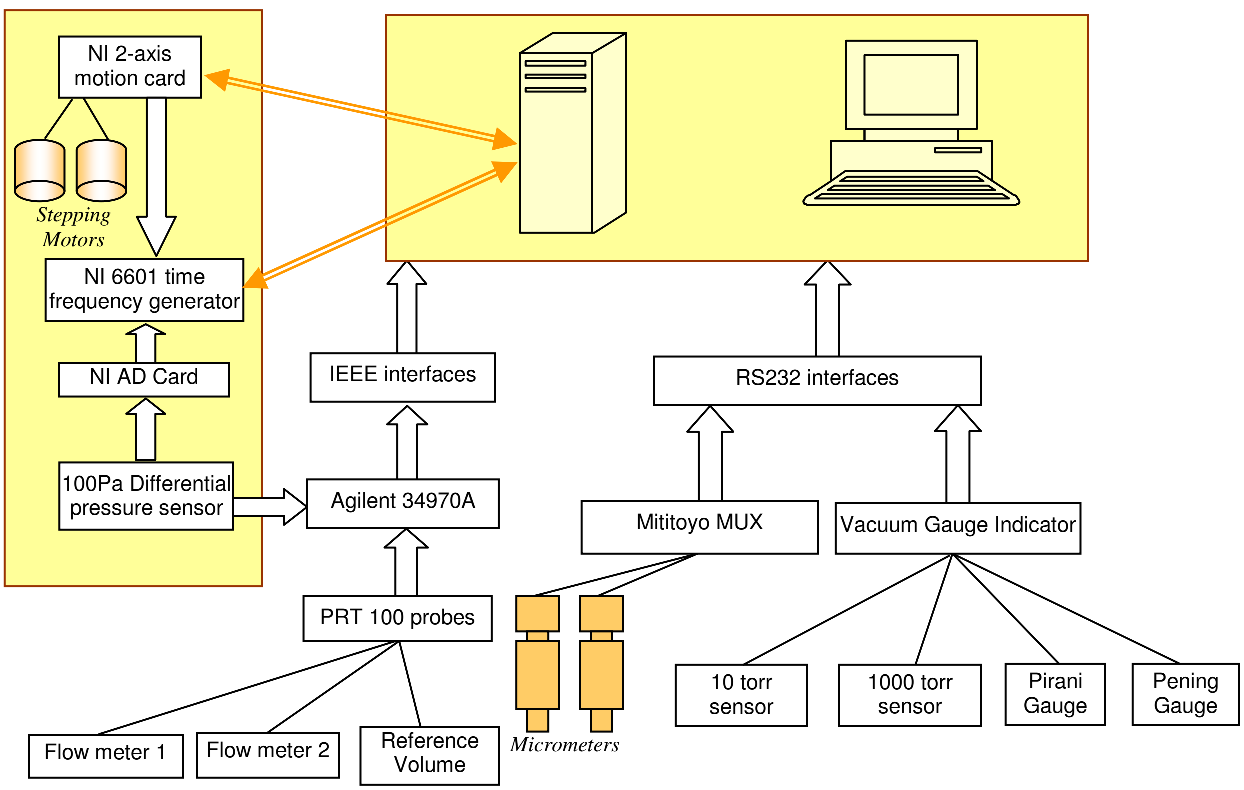


***Fig. 4** Data acquisition and control system*

3.2. Operation

Before each measurement, the standard is baked for at least 24 hours to pump down to a pressure of less than 1x10^{-5} Pa. The pumping system is then isolated from the flow meters by shutting off the valve. The gas will be admitted into the flow meters and ballast tank to bring interior pressure up to the required level to generate the desired flow rate. An all-metal leak valve (13 in Fig2) at the outlet section of the flow meter presets the flow rate to the desired level.

When equilibrium is achieved, the valves between gas flow meters, the reference volume and the ballast tank are shut off. If no disturbance is caused by the valve closure, the pressure in the gas flow meter volume would be the same as that of the reference pressure volume. The pressure in constant volume is kept constant, while the pressure in the gas flow meters drops with time. The signal of pressure drop ΔP will be instantly obtained by measuring the output voltage value of the differential pressure sensor. The flow rate is thereafter measured by ether of the two methods described below.

3.2.1 Constant pressure method

The pressure in the flow meters is maintained constant during the flow rate measurement. Before measuring the flow rate, pressure variation with the gas flow meter is observed and calculated from several measurement runs to obtain the required piston traveling speed, *ΔL/s*, for the compensation of pressure drop. Correct piston traveling speed is calculated by the software to fit the pressure drop rate. The piston is then driven continuously into the flow meter at *ΔL/s* so as to keep pressure P_m constant. From our experience, the stability of the pressure difference between the flow meter and the reference volume is often better than 0.05 Pa, which can be corrected in the software.

A measurement result obtained from the constant pressure measurement method is shown in Fig. 5.

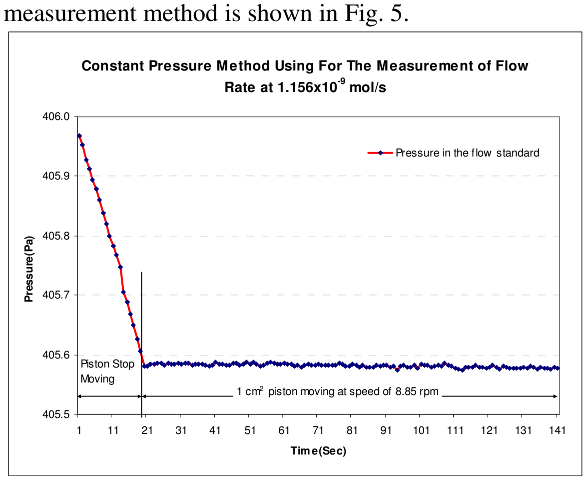


Fig. 5 *Result of a flow rate measurement using constant pressure method*

3.2.2 Constant volume method

Flow rate can also be determined by measuring pressure drop in time, *ΔP/Δt*, when the piston stops. However, this method requires accurate determination piston moving speed to feed into the gas flow meter by fitting the pressure drop rate that is commonly cost longer time to practice. In addition, the noise in temperature and pressure with time also affects the flow rate measurement. A saw tooth method[1], instead of constant volume method, is therefore used to measure the flow rate.

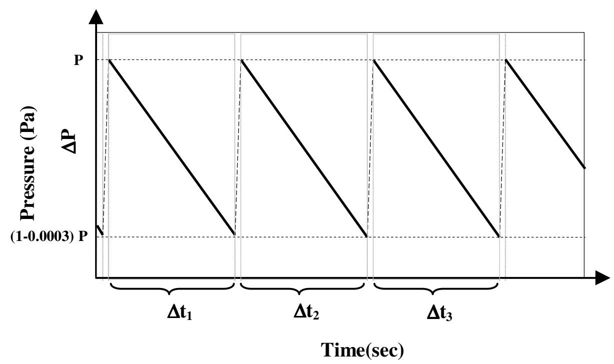


Fig.6 *The sawtooth method for flow rate measurement*

The method of sawtooth measurement is shown in Fig 6. The pressure was allowed vary within a small band from P to (1-0.0003) P. In the common practices, P was set from 100 to 10000Pa depending the flow rates to be measured. When pressure in the flow meter equals to P, the piston is stopped to let the pressure drop from P to (1-0.0003) P. The piston is moved into the flow meter with a constant speed to raise the pressure back to P. *Δt* is the time interval of pressure dropping from P to (1-0.0003) P. *ΔP/Δt* is obtained by averaging identical *ΔP/Δt*. Displacement volume *ΔV* is obtained from the piston advancing distance *Δl* and its cross sectional area *r*. The flow rate can then be calculated using equation (5).

A sample measurement using saw tooth measurement method is shown in Fig 7.

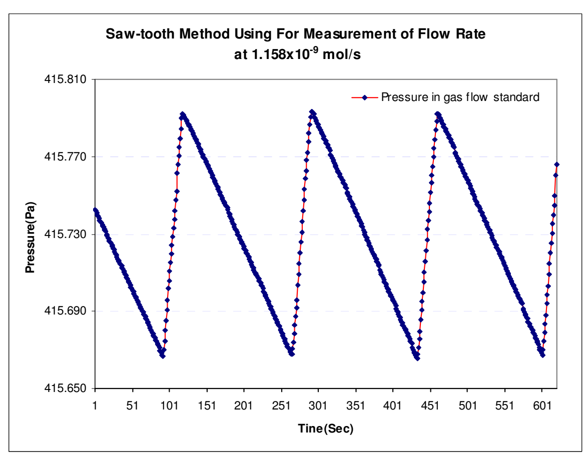


Fig. 7 *Using Saw-tooth method for flow rate measurement*

The first method has the advantage of measuring the flow rate at a constant value. It is convenient when the flow meter (UUT) requires stable flow. The second method has the advantage that the software can perform real time fit of *ΔP/Δt* curve for the compensation of temperature variation and pressure noise.

4. UNCERTAINTY

From equation (4), the relative combined uncertainty of *dN/dt* can be obtained from:

$$\frac{\Delta(dN\ /dt)}{(dN\ /dt)} = \sqrt{\begin{aligned}&(\Delta P\ /P\ )^2 + (\Delta T\ /T\ )^2 \\ &+ [\Delta(dV\ /dt)/(dV\ /dt)]^2\end{aligned}} \quad (7)$$

where:

$$\Delta(dV\ /dt)/(dV\ /dt) = \sqrt{\begin{aligned}&(\Delta dV\ /dV\ )^2 \\ &+ (\Delta t/t)^2\end{aligned}} \quad (8)$$

From equation (5), the relative combined uncertainty of *dN/dt* can be obtained from:

$$\frac{\Delta(dN\ /dt)}{(dN\ /dt)} = \sqrt{\begin{aligned}&(\Delta V\ /V\ )^2 + (\Delta T\ /T\ )^2 \\ &+ [\Delta(dP\ /dt)/(dP\ /dt)]^2\end{aligned}} \quad (9)$$

where:

$$\Delta(dP\ /dt)/(dP\ /dt) = \sqrt{\begin{aligned}&(\Delta dP\ /dP\ )^2 \\ &+ (\Delta t/t)^2\end{aligned}} \quad (10)$$

If considering on the possible leakage from the standard and adsorption and desorption of molecular at internal surface of flow meter chamber, the combined measurement uncertainty can be expressed as:

$$u_c = \sqrt{\begin{aligned}&(\Delta(dN\ /dt)/d(N\ /di)^2 + [\Delta(l)/l]^2 \\ &+ [\Delta(A)/(A)]^2\end{aligned}} \quad (11)$$

l is the leakage of the gas flow meter (Mole/s) during a measurement period; *A* is the adsorption and desorption (Mole/s)of gas from the internal surface and sliding seals.

4.1. Volume ΔdV/dV and ΔV/dV

4.1.1 ΔdV/dV

The displacement volume of piston is obtained by:

$$dV = \int_{L_1}^{L_2} \pi r^2 dL \quad (12)$$

Where: *r(M)* is the average diameter along piston; *dL(M)* is the displacement of piston.

The measurement uncertainty is given by:

$$\Delta dV\ /dV = \sqrt{\begin{aligned}&(2*\Delta r/r)^2 + (\Delta dL/dL)^2 \\ &+ \Delta V_T\ /V_T\end{aligned}} \quad (13)$$

Where: ΔV_T is the volume variation of piston due to change in ambient temperature.

The diameters of pitons were calibrated against the reference length standard of SPRING. The uncertainty of average piston diameter was combined from the uniformity of diameter at different positions along the piston, the roundness, straightness and the uncertainty of the reference length standard. The relative piston diameter u_r is given by:

$$u_r = \sqrt{\begin{aligned}&(\frac{\Delta r_{uniformity}}{2\sqrt{3}\cdot r})^2 + (\frac{\Delta r_{roundness}}{2\sqrt{3}\cdot r})^2 \\ &+ (\frac{\Delta r_{straightness}}{2\sqrt{3}\cdot r})^2 + (\frac{\Delta r_{reference}}{2\cdot r})^2\end{aligned}} \quad (14)$$

The combined uncertainties u_r are 0.002 % and 0.006 % for ϕ25mm piston and ϕ10mm piston, respectively.

The traveling distances of the piston are measured by two digital micrometers. The calibration uncertainty for both micrometers is 0.001 mm. The displacements of the pistons are measured in advance direction only so that backlash errors of the micrometers can be eliminated.

There are two measurement methods which described at 3.2.1 and 3.2.2 that the minimum piston displacement distances are 25 mm and 2.5 mm for constant pressure method and constant volume method, respectively. The relative measurement uncertainties based on the minimum displacement distances are 0.0032 % and 0.032 % for constant pressure method and constant volume method, respectively.

There is a 50 mm long stainless steel shaft linking the micrometers and the pistons. The maximum length including the shaft and the micrometer is 100 mm. The primary gas standard is working under the laboratory condition of 20 ± 1 ^{o}C, and the maximum error caused by the thermal expansion of materials is 0.0011 mm under this ambient condition. Combining with the calibration uncertainty, the standard uncertainty for the measurement of the displacement distance is 0.00081.

The linear thermal expansion rate of stainless steel is 16 ppm per ^{o}C. The cylindrical tank volume thermal expansion $\Delta V_i/V_i$ can be calculated from the materials linear thermal expansion rate as 48 ppm per ^{o}C.

The combined standard uncertainties of $\Delta dV/dV$ are obtained from the equation (13).

When constant pressure method is used:

$$\Delta dV\ /dV\ (25mm) = \pm 0.005\%$$

$$\Delta dV\ /dV\ (10mm) = \pm 0.007\%$$

4.1.2 ΔV/V

When saw tooth method is used, the uncertainty V is given as:

$$\Delta V/V = \sqrt{(\frac{\Delta V_{thermal}}{V})^2 + (\frac{\Delta dV}{V})^2} \qquad (15)$$

Where $\Delta V_{thermal}$ is the uncertainty due to the thermal expansion of materials of flow meter chamber. This is insignificant as the temperature variation during a single measurement is frequently less than ±0.1°C.

The gas flow chamber volume change due to the uncertainty of piston moving into the flow meter chamber is calculated using equation (13):

$$\Delta dV\ /dV\ (25mm) = \pm 0.032\%$$

$$\Delta dV\ /dV(10mm) = \pm 0.033\%$$

In the worse case, the minimum chamber volume V comparing to the maximum piston volume change for a single moving step into the chamber dV is 100 to 1. The relative uncertainties of V in worse case are:

$$\Delta V\ /V\ (25mm) = \pm 0.00032\%$$

$$\Delta V\ /V(10mm) = \pm 0.00033\%$$

4.2 Pressure ΔP/P and Δ dP/dP

The pressure in the gas flow meters is measured by a differential pressure sensor as mentioned in section 3.1. The differential pressure sensor was calibrated against a laser interferemetric mercury manometer of SPRING. The expended uncertainty of the differential pressure sensor calibration is 0.25 Pa. Agilent 34970A, is used for measuring the signal output form the differential pressure sensor in DC voltage. The long-term stability of Agilent 34970A is 2mV that contributes additional measurement uncertainty of 0.015 % in pressure measurements.

$\Delta P/\Delta T$ is the differential pressure variation between the gas flow meters and the reference volume due to the different temperature variations at different parts of the primary standard. The whole system is enclosed in the Perspex enclosure so that the temperature difference can be controlled within 0.1°C in short term for a single measurement. This gives rise to an error of not more than 0.05 Pa in pressure measurement.

The pressure in the reference volume is measured by the 10 torr CDG and 1000 torr CDG vacuum gauges. The 10 torr CDG was calibrated against two reference vacuum standard 1-torr and 10-torr CDGs, which were in turn calibrated using a primary vacuum standard with an expanded measurement uncertainty of 0.15 %. The 10-torr CDG was calibrated against both reference standard and fitted the results as a curve. The calibration uncertainty of the 10-Torr was estimated at 0.21% of the reading, which included the uncertainty of the reference vacuum standard and fitting of the results.

The 1000-torr CDG was calibrated against a gas deadweight pressure standard of SPRING. The measurement uncertainty of calibration is (1+0.01%*P)Pa.

Both CDGs are heated up to 45°C during measurement. The thermal transpiration effects are compensated using following equations[5][9]:

$$f = e_{vis} + (e - e_{vis}) \cdot \frac{\sqrt{T_{head}/T_{cal}} - 1}{\sqrt{T_{head}/T_{use}} - 1} \qquad (16)$$

Where: f is the thermal transpiration correction factor, e_{vis} is the error of CDG in the viscosity flow regime and e is the error of CDG in the molecular flow and transition regime. T_{head} is the temperature of CDG head, T_{cal} is the gas of temperature during calibration of the CDG and T_{use} is the temperature in the reference volume. The uncertainty f, based on our ambient laboratory condition, is 0.05% of pressure reading.

In summary, the uncertainty of pressure measurement is combined from four uncertainty sources: $u_{P(d)}$-the uncertainty of differential pressure sensor, $u_{P(T)}$- the deviation of differential pressure due to the different temperature variations at different parts, $u_{P(CDG)}$ -the uncertainty of CDG sensors, $u_{P(f)}$ the uncertainty of thermal transpiration correction factor.

$$\Delta P = \sqrt{u_{p(d)}^2 + u_{p(T)}^2 + u_{p(CDG)}^2 + u_{p(f)}^2} \qquad (17)$$

In the worse case, the relative uncertainty of pressure measurement $\Delta P/P$ is ±0.25% of reading.

When the saw tooth method is used, the $\Delta dP/dP$ is determined by the pressure drop and the uncertainty of differential pressure sensor as well as the pressure stability in reference volume during the measurement. In our case, the combined uncertainty of $\Delta dP/dP$ is not more than ±0.5% of reading.

4.3 Temperature ΔT/T

The temperatures of the gas flow standard are measured using the 4-wires PRT 100 probes supported by Agilent 34970A. The expanded uncertainty for the calibration of PTR probes is 0.03 K. The long-term stability of Agilent 34970A for the measurement of temperature is 0.06 K.

Combined the uncertainties under worse cases, the uncertainty is less than 0.037 K or 0.013 % when the gas flow standard works under the ambient condition of 20 ± 1 ^{o}C.

4.4 Time Δt/t

Time is measured by using NI6601 digital timing generator which has a 1-MHz clock. It was calibrated with an uncertainty of les s than 1 Hz or 1 x 10^{-6} s.

The start and stop are triggered in different ways depending on the measurement method used for the flow rate measurement.

When constant pressure method is used, time is triggered by the motion controller of stepper motors. The time delay due to triggering in a typical measurement is verified to be much less than 1 ms. The minimum time interval of a flow rate measurement is more than 30 seconds so that the $\Delta t/t$ is 0.0033 %.

When the saw tooth method is used, the output voltage of different pressure sensor is used to trigger the time counter at arbitrary points preset in the control program. The time delay due to triggering process is also limited within 1 ms.

A part of the time delay due to triggering time counter, the other sources of uncertainty may lead to the errors in time measurement:

- the random scatter of Δt arising from the effects of pressure sensor noise, zero fluctuations and resolution.
- the random scatter of pressure drifts $\Delta p/\Delta t$ at different rate with the time elapsed.
- the random scatter due to fitting $\Delta p/\Delta t$.
- the pressure variation due to temperature change in time $\Delta P/\Delta T$.

Combining the above sources, the maximum uncertainty across the whole measurement range is less than 0.02 %.

As time interval Δt is determined from 20 successive measurements of flow rate, the Type A uncertainty is included in the combined uncertainty.

4.5 Leakage Δl/l

The leak rate of system was checked using helium leak detector. It was found that the leak rate at every connections was not more 1 x 10^{-10} Pa/s.

The leakage due to the sliding seal is not significant as the guard pressure prevents the gas leaking though the sliding seal. We had deliberately established a large differential pressure across the sliding seal and observed the rate of pressure rising. We did not find any gas leaking though the sliding seal that may significantly affect the flow rate measurement. A conservative estimation at the worse case, the uncertainty due to the leakage of standard is less than ±0.01%

4.6 Absorption and Desorption of gas ΔA/A

The gas absorption and desorption from the internal surfaces of flow meter chamber and the sliding seals were analysed by monitoring the pressure stability in the gas flow meter when the supply and the evacuation of gas were stopped.

After filling the flow meter with gas, the pressure in the gas flow meter was maintained at a constant value for at least 300 minutes to obtain the equilibration between the gas absorption and desorption. The pressure stability in the reference volume is better than 0.001% per hour in the worse case after achieving equilibration. In the flow meter chamber, it was higher at about 0.015% per hour. This may have been caused by the higher gas adsorption rate of sling seal. Desorption and adsorption of gas is compensated in the software as it analyses the rate of pressure change and calculate the quantity of gas affected. The effect was tested at various flow rates, and the error due the gas absorption and desorption is found to be less than ±0.005%.

The gas used for measurement is 99.999%. We purged system every time before processing the measurement. Considering with the affection from leakage or gas adsorption and desorption during the measurement process, the uncertainty due to purity of gas is limited within ±0.005%

The measurement uncertainties are summarized in Table 1. The expanded uncertainty was estimated to be 1% of reading at a confidence level of 95%.

Table 1. Summary of the measurement uncertainties of the primary low gas flow standard.

Uncertainty Sources	Relative uncertainty	
	Constant pressure method	Saw tooth method
$\Delta P/P$	±0.25%	-
$\Delta dP/dP$	-	±0.5%
$\Delta V/V$	±0.00033%	-

ΔdV/dV	-	±0.007%
ΔT/T	±0.013%	±0.013%
Δt/t	±0.02%	±0.02%
ΔA	±0.005%	±0.005%
Gas purity	±0.005%	±0.005%
l	Less than 0.01%	Less than 0.01%
Combined uncertainty	±0.25%	±0.5%
Expanded uncertainty (k=2)	±0.5%	±1%

5. CONCLUSION

The low gas flow primary standard was successfully established to accurately generate and measure the gas flow in the range from 0.001 to 1000 nano-mol/s with an expanded measurement uncertainty of 1 %. It supplies gas with accurately measured flow rate for a new high and ultra-high primary vacuum standard set up at SPRING, and serves as a reference for the measurement of flow meters and leak standards.

REFERENCES

[1] K. Jousten, H. Menzer and R. Niepraschk, "A New Fully Automated Gas Standard at The PTB for Flow rate between 10^{-13}mol/s and 10^{-6}mol/s" *Metrologia* Vol. 39, pp. 519-529, 2002.

[2] K. E. McClloh, C.R Tiford, C.D Erlich and F.G. Long "- Low-Range Standard Use With Vacuum And Leak Standard" *J.Vac.Sci. Technol* A5 , pp.376-38, 1987.

[3] K. E. McClloh, C.R Tiford, S.D Wood and D.F Martin "Summary Abstract: The national Bureau of Standard orificeflow primary high vacuum flow meter" *J.Vac.Sci. Technol* A4 , pp.362, 1986.

[4] P. D. Levine et. al. "Precision gas standard for vacuum calibration", *J. Vac. Sci. Technol*. A 15(3) (1997) 747

[5] K F Poulter, M-J Rodgers, P J Nash, T J Thompson and M P Perkin, Division of Mechanical and Optical Metrology, National Physical Laboratory,"Thermal transpiration correction in capacitance monometers" *Vacuum*, volume 33/number 6/pages 311 to 316 /1983.

[6] C.D.Ehrilich, "Low-range flow-meters for use with vacuum and leak. standards", J. Vac. Sci. Technol. A5(3) (1987) 376

[7] R,Hyland, C.D.Ehrilich, C.R.Tilford, and S.Thornberg, "A Note on Flow Rate and Leak Rate Units" *J,Vac.Sci Technol*, A4, 334 (1986).

[8] K Jousten, New, fully automated, primary standard for generating vacuum pressures between 10^{-10} Pa and 3 × 10^{-2} Pa with respect to residual pressure *Metrologia* **36** 493-497 (1999)

[9] C.R.Tilford, S.Dittmann, and K.E.McCulloh, "Recommended practices of calibration and use of capacitance. diaphragm gage for a transfer standard "*J.Vac.Sci.Technol.*A6(5), 2855 (1988)