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Establishment and Verification of Mercury-Sealed Piston Prover for Primary Standard

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This paper presents the establishment and verification of a mercury-sealed piston prover, which is commonly used for low pressure gas flow calibration. The calibration gases could be dry air, nitrogen, argon, helium, oxygen and carbon dioxide. The flow capacity of the new system covers from 0.002 L/min to 40 L/min at 23 ℃ and 101.325 kPa, and some overlapped flows are between various columns. The relative expanded uncertainty of mass flow measurement is less than 0.08 % at 95 % confidence level.

We were also successfully altering the temperature sensor construction, temperature sensor placement, data logger system to real-time monitor the temperature difference of the entering gas. The data showed that the temperature difference between the entering gas and column could approximate in the calibration period.

The newly piston prover measurement result between different column was less than 0.01 %. A comparison between new-constructed and original CMC submitted piston prover that both are in CMS was conducted, and the *E*n value was less than 0.4. The results indicate that the measurement capabilities of each column of newly piston prover were identical with expectation.

1. Introduction

Low pressure gas sensing components such as laminar flowmeters, rotary flowmeters, and sonic nozzles are widely used in several industries which as semiconductor, chemistry, energy, environment and safety to control, monitor gas flow and trade. Therefore, if we want to maintain the measurement accuracy and measurement traceability of the sensing components, they should be ensured by calibration. Currently, bell prover, piston prover and PVTt system are the conventional primary standard facilities of the low pressure gas flow calibration, and most laboratories are continuously to systematically improve to meet the technical development of related low pressure gas sensing components[1][2].

The original piston prover calibration system in CMS/ITRI was constructed at 1987, and each year has been done the calibration more than 100 units. Since some equipment is old and difficult to obtain, it is necessary to rebuild for the purpose of prevent the system can’t provide the necessary calibration services. A renew project was created at 2017 to construct a newly system that the measurement uncertainty and cover flow rate range could meet our requirements. We were carefully considered the affect factors [3] [4] and then designed our newly piston prover calibration system. For example, how to position the thermometers that could measure a more representative temperature of the gas inside the column during calibration [5] was described herein.

In this paper, we also describe how to evaluate the measurement uncertainty of the gas flow rate. To verify the measurement capability and performance of the newly established piston prover, an intra-comparison between original and newly piston prover was conducted. The comparison results shown that two systems are in a good agreement were also addressed in this paper. Therefore, the capability of newly piston prover calibration system was valid. Then newly piston prover calibration system could provide calibration service at flow rate from 0.002 L/min to 40 L/min at (23 ± 1) °C.

2. Establishment of Primary Standard

2.1 Piston prover design

The newly established piston prover is shown in Figure 1. It is composed of five precision-machined glass columns (inside diameter ± 0.01 mm). Each column has the independent inlet and outlet pipes, and was covered by the acrylic lid respectively to reduce the influence of temperature exchange. A standard thermometer built in each column, and it was disposed at the gas inlet and outlet junction under the column. The pressure gauge together with the standard thermometer was also same placed at the bottom of the column. We used the low power fiber optical sensor to sensing the piston position and also it could less the heat arise.

The arrangement of five columns in different size was aligned at the central of the glass column. Then the Laser Doppler Scale (LDS) could be driven at one direction back and forth among the columns to simplify the control. Different size of columns can be selected in accordance with the flow rate for shortening the collecting time. The newly piston prover system also has auto-calibration process to use manpower sparingly.

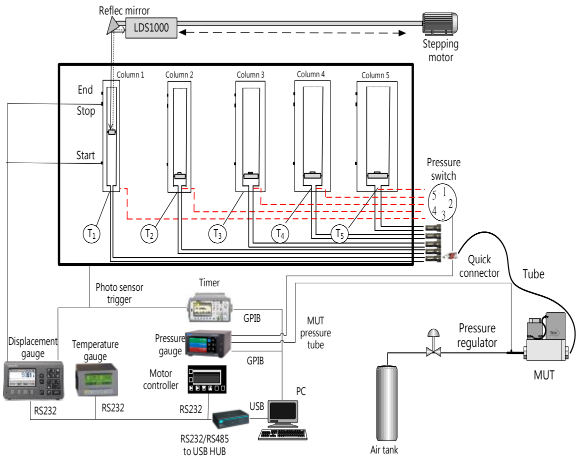

Figure 1. Diagram of newly piston prover.

2.2 Evaluation of uncertainty

According to the theory of the mass flow rate measurement could be obtained the equation (1). The equation (2) was shown the relative combined standard uncertainty of mass flow rate which was derived from the equation (1). The measurement uncertainty of the gas flow rate was evaluated in accordance with the recommendation by the ISO/IEC Guide 98-3: 2008 [6]. Regarding the uncertainty sources mentioned previously, the variation of temperature influences dimensional measurement, and the variations of temperature and pressure influence density. Thus, the contributions to the relative uncertainty of measured mass flow rate were evaluated from every effective factor via equations (3) to (4), which were listed below. All of the uncertainty components multiplied its sensitivity coefficients first, and then combined by the root-sum-square (RSS) and multiplied by a coverage factor to obtain the expanded uncertainty at 95 % confidence level. Subsequently, the contributions to relative uncertainty of measured mass flow rate from every effective factor were illustrated separately in follows.

$$q_{\mathrm{m},s} = \frac{\rho_s\times\pi\times D_s^2\times L_s\times 60}{4t\times10^6} + \frac{\Delta\rho_{\mathrm{cv}}\times V_{\mathrm{cv}}\times 60}{t} + \rho_s\times q_l = f(D_s, L_s, \rho_s, t, \Delta\rho_{\mathrm{cv}}, V_{\mathrm{cv}}, q_l) \quad (1)$$

$$\frac{u_c(q_{m,s})}{q_{m,s}} = [\left(\frac{2u(D_s)}{D_s}\right)^2 + \left(\frac{u(L_s)}{L_s}\right)^2 + \left(\frac{u(\rho_s)}{\rho_s}\right)^2 + \left(\frac{-u(t)}{t}\right)^2 + \left(\frac{4\times10^6 V_{\mathrm{cv}}}{60\pi D_s^2 L_s}\frac{u(\Delta\rho_{\mathrm{cv}})}{\rho_s}\right)^2 + \left(\frac{\Delta\rho_{\mathrm{cv}}}{\rho_s}\frac{4\times10^6 u(V_{\mathrm{cv}})}{60\pi D_s^2 L_s}\right)^2 + \left(\frac{4\times10^6 t u(q_l)}{60\pi D_s^2 L_s}\right)^2]^{1/2} \quad (2)$$

$$V_{\mathrm{cv}} = \frac{\pi\times D_s^2\times L_s}{4\times10^6} \quad (3)$$

$$\rho_s = \frac{P_s M}{Z(P_s,T_s)R_u T_s} \quad (4)$$

2.2.1. Uncertainty of column diameter

We used five ring gauges which have different diameters couple with Linear Variable Differential Transducer (LVDT) to measure the internal diameter of each column respectively. The results of average inner diameter of glass columns and uncertainty analysis were listed as the Table 1.

Table 1 Measurement data of inner diameter

Column no.	Diameter (mm)	$u_c(D_s)$ (μm)	k	U (μm)	v_i
Column 1	16.4950	0.39	1.96	0.8	520
Column 2	27.0021	1.24	2.18	2.8	12
Column 3	44.9827	1.56	2.18	3.4	12
Column 4	79.9868	1.17	2.08	2.5	21
Column 5	160.0225	1.86	2.03	3.8	33

2.2.2 Relative uncertainty of the piston displacement

Laser Doppler Scale was used to measure the displacement of the piston. LDS was calibrated by Dimensional Laboratory/CMS. Uncertainty due to calibration traceability was less than 0.0001 % and can be ignored. The maximum cosine error produced by the measurement process was estimated as 0.01 mm. If we assuming the probability distributions was rectangular and then the standard uncertainty of this item was 0.006 mm, the degrees of freedom was 50.

2.2.3 Relative standard uncertainty of density

The density value was determined through the temperature measurement and pressure measurement, as shown in Equation (4). The relative standard uncertainty of gas density measurement could be calculated from Equation (5).

$$\left(\frac{u(\rho_s)}{\rho_s}\right) = \left\{\left[\frac{u(T_s)}{T_s}\right]^2 + \left[\frac{u(P_s)}{P_s}\right]^2 + \left[\frac{u(M)}{M}\right]^2 + \left[\frac{u(R_u)}{R_u}\right]^2 + \left[\frac{u(Z(P_s,T_s))}{Z(P_s,T_s)}\right]^2\right\}^{1/2} \quad (5)$$

There were four factors in temperature measurement, (a) Calibrated uncertainty of thermometer was 0.018 ℃, and the associated degrees of freedom was 60. (b) The error of calibrated thermometer was within ±0.003 ℃. If estimating with rectangular distribution, the standard uncertainty of this term was 0.002 ℃, with degrees of freedom was 50. (c) The long-term stability of temperature gauge was less than 0.01 ℃. If estimated with rectangular distribution, the standard uncertainty of this term was 0.006 ℃, with degrees of freedom was 50. (d) The measurement error that was caused by the non-uniform temperature distribution, around 0.10 ℃ and we assumed that was a rectangular distribution. Then, the uncertainty of this item was 0.058 ℃, and the associated degrees of freedom was 50. Therefore, we could estimate the combined relative standard uncertainty was 0.021% at lowest operating temperature nearly 295.15 K with degrees of freedom was 62.

In pressure measurement, we estimated three factors. They were (a) Calibrated uncertainty of pressure gauge was 6 Pa, and the associated with degrees of freedom was 60. (b) Long-term stability of the pressure gauge. We using the pressure gauge drift from the manufacturer claims to evaluate. The maximum variation was estimated as 15 Pa. Then we could obtain the standard uncertainty was 8.7 Pa when we assuming that was a rectangular distribution, the degrees of freedom was 50. (c) Another uncertainty was the measurement error due to the installation position and sampling. The maximum deviation was 10 Pa between the pressure gauge sampling position and the average pressure appeared of the gas inside the column. Thus, the standard uncertainty could be calculated as 5.8 Pa, and the associated with degrees of freedom was 50. Finally, we can obtain the relative standard uncertainty at lowest operating pressure (100 kPa) was 0.012 %, degrees of freedom was 132.

Universal gas constant was a fixed number, thus the uncertainty could be ignored. The gas molecular weight was referred to as the publication of NIST [7]. The relative standard uncertainty of air was less than 0.019 %. The relative standard uncertainty of the other gases was less than 0.002 %, the associated degrees of freedom was 100. The relative standard uncertainty of gas compression was calculated by the NIST REFPROP software. Therefore, the relative standard uncertainty of gas compression can be estimated as 0.001 % and the associated degrees of freedom was 146.

Finally, the relative uncertainty due to the measurement of gas density was obtained of 0.031 % via RSS method, and the associated degrees of freedom was 200 as shown in Table 2.

Table 2 Relative uncertainty of density

Composition of density	$u(x_i)$	$\frac{u(x_i)}{x_i}$ (%)	c_i	$\lvert c_i\rvert\frac{u(x_i)}{x_i}$ (%)	v_i
T_s	0.061		1/295.15	0.021	62
P_s	0.012		1/100	0.012	132
M		0.019	1	0.019	100
R_u		0.000	1	0.000	100
Z_s		0.001	1	0.001	146
ρ_s				**0.031**	200

2.2.4 Relative standard uncertainty of time

The influence factor of the time relative standard uncertainty assessment included three items. (a) Universal counter was employed as a standard timer for piston prover system. The standard uncertainty was less than 0.0001 ms according to the calibration report. Thus, the uncertainty of this item could be ignored. (b) The maximum difference between the timer and the LDS counter trigger synchronization from the experiments less than 0.001 s. If the minimum collecting time of 15 s was employed to calculate the relative standard uncertainty and assuming that was a rectangular distribution, then we could obtain the value as 0.004 % with the value of degrees of freedom was 12. (c) The resolution of timer was 0.0001 s, and it was assumed as a rectangular uncertainty distribution. Based on a minimum collecting time of 15 s, the relative standard uncertainty was 0.0004 %. The degree of freedom was 50. Then we can obtain the combine relative standard uncertainty of time measurement was 0.004 % and the degrees of freedom was 12.

2.2.5 Relative standard uncertainty due to gas density variation inside the control volume during the beginning and the end of collecting period

The density change of gas inside control volume during the calibration period was less than 0.00015. If estimating with rectangular distribution, the relative standard uncertainty of gas density variation was 0.009 % and the degrees of freedom was 50. The sensitivity coefficients of columns were from 0.3 to 0.7, and then the relative standard uncertainties were located at 0.0027 % to 0.0063 % as table 3.

Table 3 Relative uncertainty of control volume during the beginning and the end of collecting period

	Column 1	Column 2	Column 3	Column 4	Column 5
$\frac{u(\Delta\rho_{\mathrm{cv}})}{\rho_s}$	0.009 %	0.009 %	0.009 %	0.009 %	0.009 %
$\frac{4\times10^6 V_{\mathrm{cv}}}{60\pi D_s^2 L_s}$	0.7	0.3	0.3	0.3	0.4
$\frac{4\times10^6 V_{\mathrm{cv}}}{60\pi D_s^2 L_s}\frac{u(\Delta\rho_{\mathrm{cv}})}{\rho_s}$	0.0063 %	0.0027 %	0.0027 %	0.0027 %	0.0036 %
Degrees of freedom	50	50	50	50	50

2.2.6 Relative standard uncertainty due to volume measurement of control volume

The density change of gas inside control volume during the calibration period was less than 0.00015. The control volume was estimated by connect pipe size, thus the sensitivity coefficient could be estimated as 0.2 for all columns. Therefore, the relative standard uncertainty of control volume can be evaluated as 0.003 %, and the degrees of freedom was 12.

2.2.7 Relative standard uncertainty due to leakage of control surface

The leakage flow rate was assessed from the actual leakage test data. The dry air enters the column to push the piston to a position then close the inlet valve. Then, LDS continuously to monitor the variance of displacement of piston for a period, and the temperature, pressure at same time. Thus, we could use the collection values from time, temperature, pressure, and piston displacement to obtain the leakage flow. The low limit flow rate of each standard column was used as the flow rate in leakage test, the leakage flow rate of every column was shown in Table 4. Use this value to calculate the standard uncertainty of this item.

Table 4 Uncertainty of leakage flow rate

Column no.	Flow rate (L/min)	$q_{v,l}$ (L/min)	ν_{i}
Column 1	0.002	5×10^{-7}	100
Column 2	0.025	1×10^{-6}	100
Column 3	0.1	2×10^{-6}	100
Column 4	0.5	1×10^{-5}	100
Column 5	2	5×10^{-5}	100

Table 5 and Table 6 are shown the uncertainty budgets of column 1 operated at 0.002 L/min and of column 5 operated at 40 L/min. In table 5, we could found that column 1 had 5×10^{-7} L/min of the actual leakage flow rate at minimum flow rate, and the relative standard uncertainty of the leakage flow rate was 0.014 %. Therefore, the relative combined standard uncertainty of column 1 was 0.036 %. In table 6, we could found that the relative standard uncertainty of gas density was the biggest uncertainty source. The relative standard uncertainty of the leakage flow rate far less than the relative standard uncertainty of gas density. Then, we could found that the relative combined standard uncertainty of column 5 was 0.032 % is different as column 1. Also, we found that the influence of the variance of gas density in the collecting volume was different for each standard column due to the difference sensitivity coefficients.

In table 7, we shown the relative combined standard uncertainty and the flow rate ranges of each column of our newly piston prover. The capabilities of each columns were meet our requirements. The relative standard uncertainty of gas density is the biggest uncertainty source of this newly piston prover. In the relative standard uncertainty of gas density, the influence of temperature is bigger than other factors. How to reduce the temperature influence in the system is the future work.

Table 5 Uncertainty budget of Column 1 (0.002 L/min)

Components	$\frac{u(x_{\mathrm{i}})}{x_{\mathrm{i}}}$ (%)	c_{i}	$\lvert c_{\mathrm{i}}\rvert\frac{u(x_{\mathrm{i}})}{x_{\mathrm{i}}}$ (%)	ν_{i}
D_s	0.003	2	0.006	520
L_s	0.002	1	0.002	58
ρ_s	0.031	1	0.031	200
t	0.000	1	0.000	12
$\Delta\rho_{\mathrm{cv}}$	0.009	0.7	0.0063	50
V_{cv}	20	0.00015	0.003	12
$q_{v,l}$	0.014	1	0.014	100
$u_c(q_{\mathrm{m,s}})/q_{\mathrm{m,s}}$			**0.036**	329
k			**1.97**	
U			**0.08**	

Table 6 Uncertainty budget of the Column 5 (40 L/min)

Components	$\frac{u(x_{\mathrm{i}})}{x_{\mathrm{i}}}$ (%)	c_{i}	$\lvert c_{\mathrm{i}}\rvert\frac{u(x_{\mathrm{i}})}{x_{\mathrm{i}}}$ (%)	ν_{i}
D_s	0.002	2	0.004	33
L_s	0.001	1	0.001	60
ρ_s	0.031	1	0.031	200
t	0.004	1	0.004	12
$\Delta\rho_{\mathrm{cv}}$	0.009	0.4	0.0036	50
V_{cv}	20	0.00015	0.003	12
$q_{v,l}$	0.0001	1	0.0001	100
$u_c(q_{\mathrm{m,s}})/q_{\mathrm{m,s}}$			**0.032**	225
k			**1.97**	
U			**0.07**	

Table 7 Uncertainty budget of the piston prover

Column no.	Flow rate range (L/min)	$u_c(q_{m,s})/q_{m,s}$ (%)	k	U (%)
Column 1	(0.002 to 0.1)	0.036	1.97	0.08
Column 2	(0.01 to 0.3)	0.034	1.97	0.07
Column 3	(0.1 to 1)	0.033	1.97	0.07
Column 4	(0.5 to 5)	0.032	1.97	0.07
Column 5	(2 to 40)	0.032	1.97	0.07

Nomenclature	
$q_{m,s}$	Standard mass flow rate [g/min]
ρ_s	Gas density of the collecting volume[g/L]
D_s	Average inner diameter of the column [mm]
L_s	Displacement of piston [mm]
t	Collection time [s]
$\Delta\rho_{cv}$	Density change of gas in the control volume at the beginning and end of calibration [g/L]
V_{cv}	Volume in the control volume [L]
$q_{v,l}$	Leakage rate during the calibration process [L/min]
V_c	Collecting volume inside the column [L]
T_s	Absolute temperature of gas in the collecting volume [K]
P_s	Absolute pressure of gas in the collecting volume [kPa]
M	Gas molecular weight [g/mol]
R_u	Universal gas constant [J/mol·K]
Z	Gas compressibility constant [J/mol·K]
U	Relative expanded uncertainty
u_c	Relative combined standard uncertainty
k	Coverage factor
$u(x_i)$	Standard uncertainty
c_i	Sensitivity coefficient
v_i	Degrees of freedom for $u(x_i)$

3. Verification of the newly Piston Prover

3.1 Comparison of new piston prover between different columns

A comparison between different columns was carried according to the overlap of flow rate range to confirm the performance consistency. Four Laminar flow elements have good stability that was used as the transfer standard (TS). The calibration gas was dry air. The test results were shown in Figure 2. The consistencies among different standard columns of newly piston prover were authenticated, and the maximum deviation was less than 0.01 %.

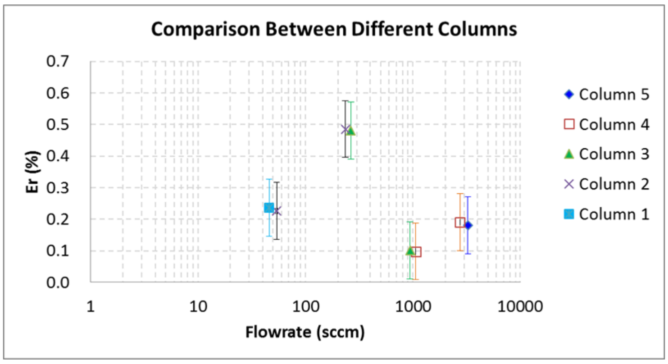

Figure 2 Comparison between different columns

3.2 Intra-comparison between original and newly established piston prover

A comparison between new-constructed and original CMC submitted piston prover was conducted to validate the capability of newly piston prover fit the bill for industry calibration needs. In table 8 were shown the results of the comparison.

The E_n value was used as the index for measuring the test capability of two systems. The calculation way of E_n value was shown in Equation (6).

$$|E_n| = \left|\frac{E_1 - E_2}{\sqrt{U_1^2 + U_2^2}}\right| \quad (6)$$

Where, E_1 was the measurement result for the relative error of new piston prover. E_2 was the measurement result for the relative error of original piston prover. U_1 was 0.09 % of the expanded uncertainty of new Piston Prover system. Prover. U_2 was 0.11 % of the expanded uncertainty of the original piston prover system. Under the same flow rate, the differences of the original system and new system were located from 0.00 % to 0.05 %. The evaluation of En value were equal or less than 0.4 at all tests. The verification results were identical with expectation.

Table 8 Summary of the comparison results between different Piston Prover

Laminar flowmeter	Flow rate (sccm)	E_1 (%)	E_2 (%)	U_1 (%)	U_2 (%)	$\lvert E_n \rvert$
m-1247	250	0.45	0.45	0.09	0.11	0
	200	0.39	0.39	0.09	0.11	0
	150	0.34	0.35	0.09	0.11	0.1
	100	0.32	0.30	0.09	0.11	0.1
	50	0.35	0.33	0.09	0.11	0.1
m-977A	1000	0.11	0.15	0.09	0.11	0.3
	750	0.09	0.12	0.09	0.11	0.2
	500	0.09	0.10	0.09	0.11	0.1
	250	0.09	0.12	0.09	0.11	0.2
m-1286	5000	0.33	0.35	0.09	0.11	0.1
	4000	0.24	0.25	0.09	0.11	0.1
	3000	0.19	0.21	0.09	0.11	0.1
	2000	0.14	0.17	0.09	0.11	0.2
	1000	0.10	0.15	0.09	0.11	0.4
m-1092	20000	0.46	0.49	0.09	0.11	0.2
	15000	0.43	0.46	0.09	0.11	0.2
	10000	0.44	0.48	0.09	0.11	0.3
	6000	0.48	0.53	0.09	0.11	0.4

Note: sccm is standard cubic centimeter per minute @0˚C

4. Conclusion

A newly mercury-sealed piston prover was established and verified at CMS/ITRI. The flow capacity of this new system covers from 0.002 L/min to 40 L/min at 23 ℃ and 101.325 kPa. The relative expanded uncertainty of mass flow measurement was less than 0.08 % at 95 % confidence level. The comparison results indicate that the measurement capabilities and system stability of each column of newly piston prover were identical with expectation.

5. Acknowledgement

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