

Bilateral Comparison of Primary Low-Pressure Gas Flow Standards Between NIMT and CMS

Chun-Min Su¹, Win-Ti Lin¹ and Sutham Masri²

¹Center for Measurement Standards/ Industrial Technology Research Institute, Taiwan

²National Institute of Metrology (Thailand)

30 Ta Hsueh Road, Hsinchu 30080, Taiwan

Tel: 886-3-5741205, Fax: 886-5-5710335, E-mail: CMSu@itri.org.tw

Abstract: To verify the measurement capability and performance of the newly established primary low-pressure gas flow standard, a mercury-sealed piston prover, at the National Institute of Metrology of Thailand (NIMT), a bilateral comparison with the piston prover at the Center for Measurement Standards (CMS, Taiwan) was conducted. The piston prover at NIMT has a measurement capability of 5 mL/min to 24 L/min flow range with a relative expanded uncertainty smaller than 0.13 %, whereas that of the system at CMS is 2 mL/min to 24 L/min with 0.10 % expanded uncertainty. The transfer standard (TS) used was a set of three critical flow venturis (CFV's) with dedicated thermometers. The TS was used at eight flow rates of dry air ranging from 42 mL/min to 14.5 L/min at 0 and 101.325 kPa (0.055 g/min to 18.87 g/min). Comparison showed that the differences in the normalized average discharge coefficient between NIMT's measurements and the reference values based on CMS's results were smaller than 0.06 % throughout the range of flow tested. The E_n values for the flow measurements at NIMT with reference to CMS were all well less than unity. The equivalence between the two participants was thus demonstrated.

Keywords: Piston Prover, Critical Flow Venturis, Degree of Equivalence

1. Introduction

In a collaboration project between the National Institute of Metrology of Thailand (NIMT) and Center for Measurement Standards (CMS, Taiwan), a mercury-sealed piston prover serving as the primary low-pressure gas flow standard at NIMT was established in 2009. In order to verify the measurement capability and performance of the piston prover, a bilateral comparison between NIMT and CMS was conducted. The standard facility of CMS, who serves as the pilot laboratory in this comparison, is also a piston prover.

A set of three critical flow venturis (CFV's), or sonic nozzles, with dedicated thermometers was used as the transfer standard (TS) for this comparison. As shown in Fig. 1, each of the CFV's has been installed in respective stainless steel holder. On the holder, a custom-made 4-wire PT-100 thermal sensor is installed and connected to a handheld digital display. Two quick connector type taps have also been administered for the measurement of pressure upstream and downstream of the CFV.

The three CFV's have measured throat diameters of 0.035 mm, 0.106 mm and 0.585 mm, and were identified as SN-003, SN-010 and SN-060, respectively. The CFV's were chosen as the TS flowmeter due to their robustness, long term stability and well-understood and relatively small sensitivities, and the fact that they have often been used in inter-comparisons [1–3]. Data acquisition at CMS was carried out using a self-developed, LabVIEW based program. A revised and compiled version of this program was used in the system at NIMT. The property database

developed by the National Institute of Standards and Technology (NIST), Refprop (Version 8.0), was used to obtain the gas properties.



Fig. 1 A photo of the transfer standard (TS) used in the bilateral comparison

The TS was used, with dry air as the test fluid, at eight flows ranging approximately from 42 mL/min to 14.5 L/min at standard condition of 0 and 101.325 kPa (about 0.055 g/min to 18.87 g/min). The specific TS used, nominal operation (inlet) pressure and corresponding approximate volume flow rate for each flow point are listed in Table 1.

Table 1 Measurement arrangement of the bilateral comparison

Flow point	TS	Operation pressure (kPa)	Approximate flow rate (mL/min)
1	SN-003	450	42
2	SN-003	500	47
3	SN-010	300	278
4	SN-010	400	374
5	SN-010	500	470
6	SN-060	300	8700
7	SN-060	400	11600
8	SN-060	500	14500

The first measurement was carried out at CMS from 13 to 14 August 2009. The TS was then carried to NIMT where measurement with its piston prover was conducted from 30 September 2009 to 1 October 2009. The last measurement took place back at CMS from 6 to 8 October 2009. The second CMS data set was used as the reference value for the comparison. The parameter

compared is the averaged discharge coefficient of the CFV's. The degree of equivalence as represented by the E_n value for NIMT at each flow is shown in Fig. 2. Being less than unity for all the E_n values indicates good agreement and valid claimed measurement capability.

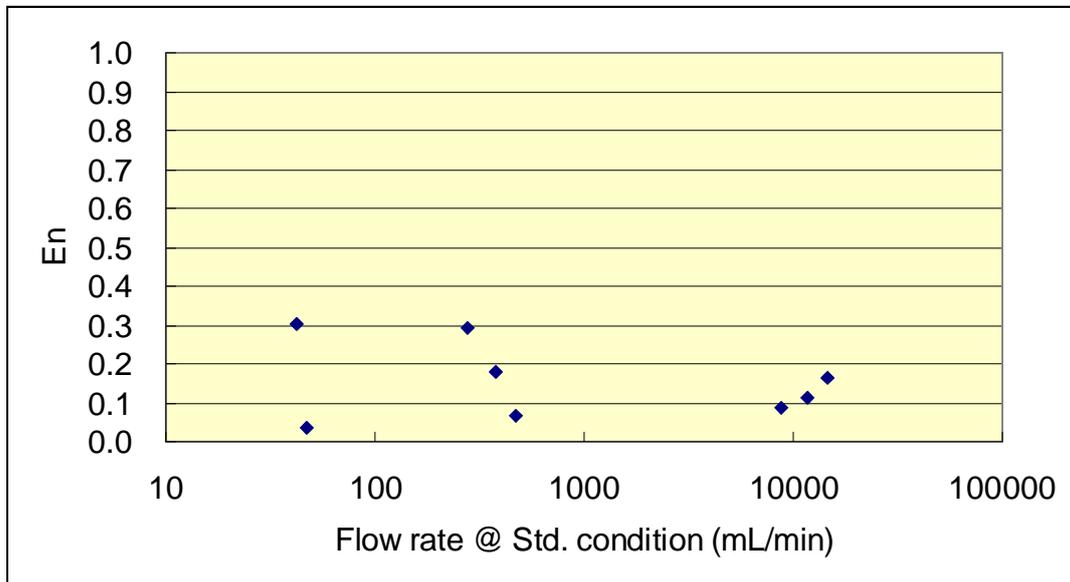


Fig. 2 The E_n values for NIMT in this comparison as reference to the second CMS data set

2. Equipment and Methods

2.1 Primary Low-Pressure Volumetric Gas Flow Standard at CMS

The CMS primary standard used in this comparison is a piston prover consisting of five precisely manufactured glass cylinders. The system is installed in a room with controlled temperature at (23 ± 1) and relative humidity at (45 ± 10) % and is capable of measuring flows ranging from 2 mL/min to 24 L/min at the reference condition (296.15 K and 101.325 kPa). Table 2 lists the mean cylinder inner diameter (I.D.), applicable range of flow and measurement uncertainty for each column.

Table 2 General information for each column of the piston prover at CMS

Column no.	Mean I.D. (mm)	Flow rate range (mL/min)	Relative standard uncertainty (%)
1	19.053	2 – 100	0.050
2	26.987	10 – 500	0.045
3	44.434	40 – 1600	0.048
4	76.209	100 – 6000	0.045
5	143.688	1000 – 24000	0.045

Each glass cylinder is equipped with a mercury-sealed piston capable of moving up and down freely, for accommodating the incoming gas and triggering the timing device. A corner cube is

placed on top of the piston as a movable reflector for a laser interferometer system located at the top of the piston prover. Two photo sensors, actuated by the mercury ring, control the start and stop of an electronic digital timer, making the operation completely automatic.

The distance traversed by the piston, and thus the collection volume, are dictated by triggering of two photo sensors and measured simultaneously by the interferometer system. This distance is measured for each and every flow calibration. Based on the measured distance, collection time, gas pressure inside the cylinder at the beginning and at the end of flow calibration, averaged gas temperature, and the pre-determined glass cylinder inner diameter, the standard mass flow rate, or the volume flow rate after conversion, can be determined based on equation (1).

$$q_{m,s} = \rho_s \cdot \frac{V_C}{t} + \Delta\rho_{CV} \cdot \frac{V_{CV}}{t} + \rho_s \cdot q_{v,l} = \rho_s \cdot \frac{D_s^2 \pi L_s}{4} \frac{L_s}{t} + \Delta\rho_{CV} \cdot \frac{V_{CV}}{t} + \rho_s \cdot q_{v,l} \quad (1)$$

Where $q_{m,s}$ is the mass flow rate measured by the standard system; ρ_s is the gas density at the standard system; V_C is the collection volume between two photo sensors; t is the collection time; $\Delta\rho_{CV}$ is the density change inside the control volume during calibration; V_{CV} is the control volume between the device under test and the standard system; $q_{v,l}$ is the estimated leakage volume flow rate; D_s is the averaged diameter of the glass cylinder; and L_s is the distance traversed by the piston. Figure 3 shows the experimental setup at CMS.

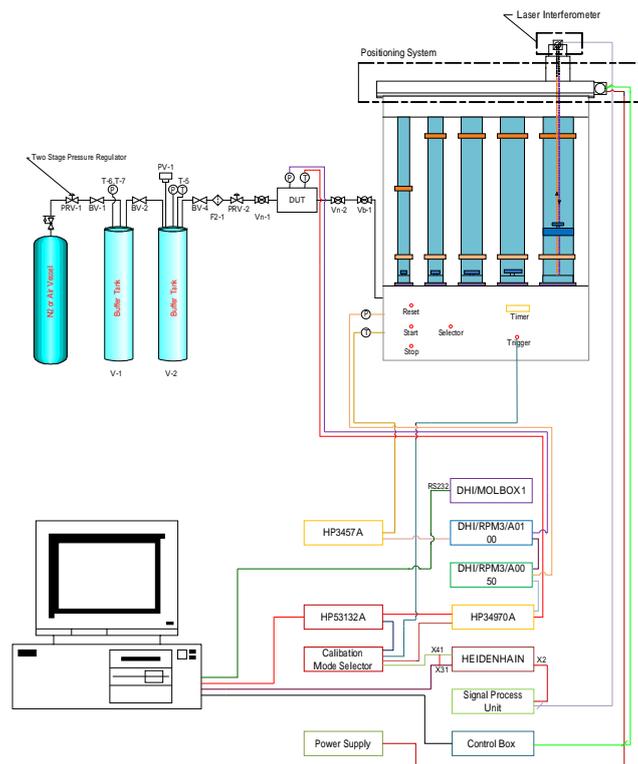


Fig. 3 Schematic of the experimental setup of the piston prover at CMS

The self-assembled interferometer uses a 633 nm laser from MELLES GRIOT and ND281B display unit from HEIDENHAIN. The interferometer is installed on a positioning system which allows the laser beam to move to the column intended to be used. HP 3457A digital multi-meter and HP 34970A data acquisition / switch unit are used, in combination with custom-made 4-wire

PT-100 sensors, to measure the temperatures of the gas flowing into the glass cylinder at various locations of the standard and, if applicable, at the device under test (DUT). HP 53132A universal counter serves as the timer. Two DHI RPM3 digital pressure gauges are used to measure the pressure at the standard and the DUT. For the measurements at CMS in this comparison, the nozzles SN-003 and SN-010 were calibrated on column no. 3 and the nozzle SN-060 on column no. 5.

The uncertainty of flow measurement at CMS was evaluated according to the international guidelines ISO GUM [4]. A summary of the relative standard uncertainty for flow measurement by the five columns is given in Table 2. With 95 % level of confidence, the claimed relative expanded uncertainty for the entire flow range covered is 0.1 %.

2.2 Primary Low-Pressure Volumetric Gas Flow Standard at NIMT

The NIMT primary standard used in this comparison is a piston prover with similar design as that of CMS consisting of three glass cylinders. The room where the system is installed is controlled at the temperature of (23 ± 1.5) . The designed capacity of the system is from 5 mL/min to 24 L/min at the reference condition. Table 3 lists the mean cylinder I.D., applicable range of flow and measurement uncertainty for each column. The measurement principle is the same as the piston prover of CMS, and the instrumentation is also similar. The main difference is that the traveling distance of the piston is now measured by a laser Doppler scale (LDS) instead of an interferometer.

Table 3 General information for each column of the piston prover at NIMT

Column no.	Mean I.D. (mm)	Flow rate range (mL/min)	Relative standard uncertainty (%)
1	19.0448	5 – 200	0.063
2	44.4507	50 – 1500	0.055
3	143.7143	500 – 24000	0.054

Figure 4 shows the schematic of the experimental setup at NIMT. The LDS sits steadily on top of the piston prover, and a set of specially designed guiding optics is used to direct the laser beam to the column of interest. A HEIDENHAIN ND281B receives the output signal of the LDS and displays the measured distance traversed by the piston. Four CHINO DB1000 digital indicating controllers are used, in combination with custom-made 4-wire PT-100 sensors, to measure the temperatures of the gas at each of the three glass cylinder and at the DUT. The timing device used is a RED LION CUB5T miniature electronic preset timer and cycle counter. A two-channel mensor 2500 digital pressure gauge is used to measure the pressure at the standard and the DUT. For the measurement at NIMT in this comparison, the nozzles SN-003, SN-010 and SN-060 were calibrated on columns no. 1, 2 and 3, respectively.

The uncertainty of flow measurement at NIMT was again evaluated according to ISO GUM [4]. Details can be found in Sutham et al. [5]. The standard measurement uncertainties for all the three columns are summarized in Table 3. With 95 % level of confidence, the expanded uncertainties for columns no. 1, 2 and 3 are 0.13, 0.11 % and 0.11 %, respectively.

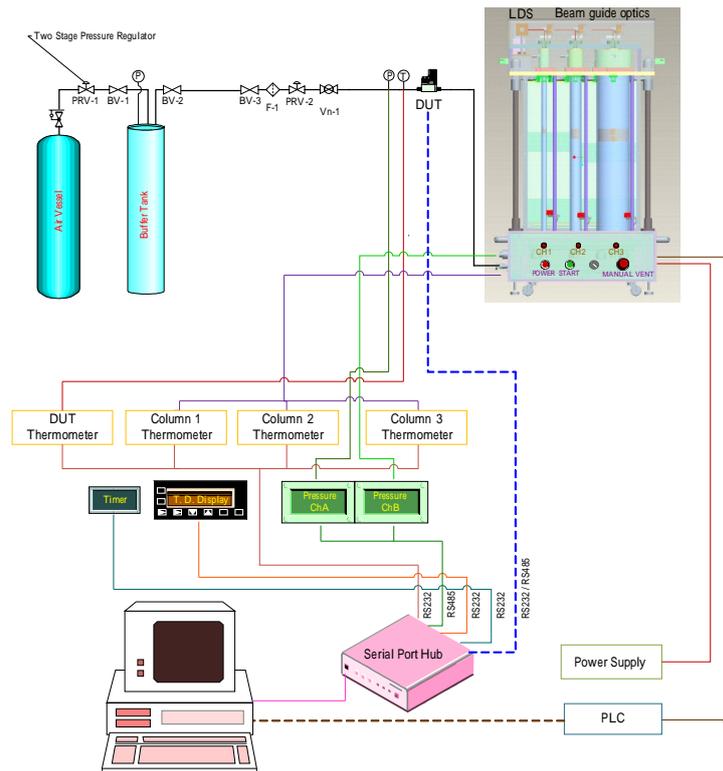


Fig. 4 Schematic of the experimental setup of the piston prover at NIMT

2.3 Data Analysis

2.3.1 Measurement Parameter

In this comparison, mass flow measurements from the two participating labs, NIMT and CMS, are compared using the discharge coefficient, C_d , for the three CFV's.

$$C_d = \frac{q_{m,s}}{q_{m,th}} = \frac{q_{m,s}}{A^* C^* P_0 / \sqrt{(R/M)T_0}} \quad (2)$$

Where $q_{m,s}$ is the mass flow rate measured by the standard system; $q_{m,th}$ is the theoretical CFV mass flow; R is the universal gas constant; M is the molecular weight of the gas (air); T_0 is the gas stagnation temperature; A^* is the throat area of the CFV; C^* is the critical flow function of sonic nozzle; and P_0 is the gas stagnation pressure.

The air supply in both labs were dehumidified to a dew point temperature of -40 or lower, therefore, the molecular weight and C^* were calculated based on dry air composition. Three repeated calibrations for the eight flows were carried out at each lab, yielding a total of 24 data points in each of the three set of measurements conducted in this comparison. The three C_d values for each CFV at each flow were averaged to obtain \bar{C}_d , which was then used for the comparison.

2.3.2 Uncertainty Analysis

The uncertainty of the discharge coefficient was evaluated in accordance with the CIPM CCM.FF-K6 comparison report [2]. The components of the uncertainty include:

- (1) the mass flow measured by the participating lab,
- (2) the standard deviation of the mean of repeated measurements at each flow, and
- (3) factors related to the determination of theoretical mass flow rate by the TS.

Starting from equation (2), the relative standard uncertainty of C_d can be expressed as the following equation (3).

$$\frac{u(C_d)}{C_d} = \sqrt{\left(\frac{u(q_{m,s})}{q_{m,s}}\right)^2 + \left(\frac{-u(q_{m,th})}{q_{m,th}}\right)^2} \quad (3)$$

The uncertainty of theoretical mass flow rate, $u(q_{m,th})$, as given in equation (4), can be further evaluated by components that can be grouped into two categories: (a) the first order effects of the parameters appear in equation (2), $u_1(q_{m,th})$, and (b) the second order effects of the parameters and other factors unaccounted for in the physical model, $u_2(q_{m,th})$.

$$u(q_{m,th}) \equiv \sqrt{u_1^2(q_{m,th}) + u_2^2(q_{m,th})} \quad (4)$$

For the uncertainty originated from mass flow measurement, the capabilities of the two participating labs described in sections 2.1 and 2.2 were used. Moreover, the maximum of the standard deviation of the mean of three C_d values obtained at each flow set point from each lab was used to evaluate the uncertainty associated with reproducibility of the measurement.

The term $u(q_{m,th})$ can be evaluated based on equation (2) and the method of propagation of uncertainties. Uncertainties related to the determination of P_0 , T_0 , C^* and M have been considered and attributed to $u_1(q_{m,th})$. Temperature and critical flow effects as well as leaks have also been taken into account and attributed to $u_2(q_{m,th})$. The sources and magnitudes of these seven components are elucidated below.

- (1) Pressure: The pressure at the inlet of the CFV's was measured using the participants' own pressure gauge. Contributions from traceability, curve-fitting and drifting were considered. As a result, the relative standard uncertainties of the pressure measurement, with reference to an operation pressure of 450 kPa / 300 kPa for nozzles SN-003 / SN-010 & SN-060, are 0.006 % / 0.008 % and 0.011 % / 0.015 % for CMS and NIMT, respectively.
- (2) Temperature: Both CMS and NIMT used the same temperature sensors in the comparison and thus calibration biases are correlated. Observations showed that the temperature variation during a single calibration was well within 0.05 . Taking this value as the effective resolution and applying a rectangular distribution result in a relative standard uncertainty of 0.01 % with reference to the room temperature of 296.15 K. The sensitivity coefficient c_i is 0.5 since temperature appears under the square root in equation (2).
- (3) Critical Flow Function: The real critical flow function was calculated according to the international standard ISO 9300 [6] and was used in this comparison for both participants. However, as a conservative measure, the value 0.008 % from [2] which was obtained by analyzing the differences between the real and ideal critical flow function values was adopted in this comparison as the relative standard uncertainty.

- (4) Molecular Weight: The value 0.011 % from [2] was adopted in this comparison. This value was obtained by analyzing the agreement of M between the reported values and those calculated from the dew point temperature among the CCM.FF-K6 comparison participants. Appearance under the square root in equation (2) gives M a sensitivity coefficient of 0.5.
- (5) Temperature Effects: The uncertainty due to temperature effects for the CVF's used in K6 was deliberately studied in [7] and was incorporated in the K6 report [2]. The values 0.016 % and 0.006 % for the small and large CVF's, respectively, were given in the K6 report. In light of the facts that (a) the observed maximum differences between the room and CVF gas temperature at CMS and NIMT were both smaller than 1 K, which is smaller than that of 1.6 K found in the K6 report, (b) the CVF's SN-003 and SN-010 used in the bilateral comparison are smaller than the small ones used in K6, indicating that they would be more sensitive to temperature effects, and (c) the CVF's used in this comparison were installed by o-ring sealing without direct contact with the holder, meaning that they could be less susceptible to environmental temperature change, the value 0.016 % was used for all the CVF's in this comparison.
- (6) Critical Flow Effects: A preliminary study at CMS showed that the critical back pressure ratio (CBPR), defined as the downstream to upstream pressure ratio at which the discharge coefficient is 0.1 % below the initial value in the choking condition, of the CVF's SN-003, SN-010 and SN-060 were, as shown in Fig. 5, approximately 0.34, 0.57 and 0.47, respectively, under an inlet pressure of about 300 kPa. At a back pressure ratio larger than CBPR, the nozzles will no longer be critical. In this comparison, as can be derived from Table 1, the maximum back pressure ratio was about 0.22 for SN-003 and 0.33 for SN-010 and SN-060, which were well below their corresponding CBPR. Therefore, the uncertainty due to breakdown of choke condition was considered negligible.
- (7) Leaks: Leak check on the CVF's was performed prior to the comparison. The design of the CVF holder allows minimum rearrangement of the TS piping and gives us the confidence that the leaks were controlled to less than 0.01 % of all flows tested.

Since both NIMT and CMS used the same values of the universal gas constant and CVF throat diameter (cross-sectional area at throat) in the calculation of C_d , uncertainties related to these components can be ignored.

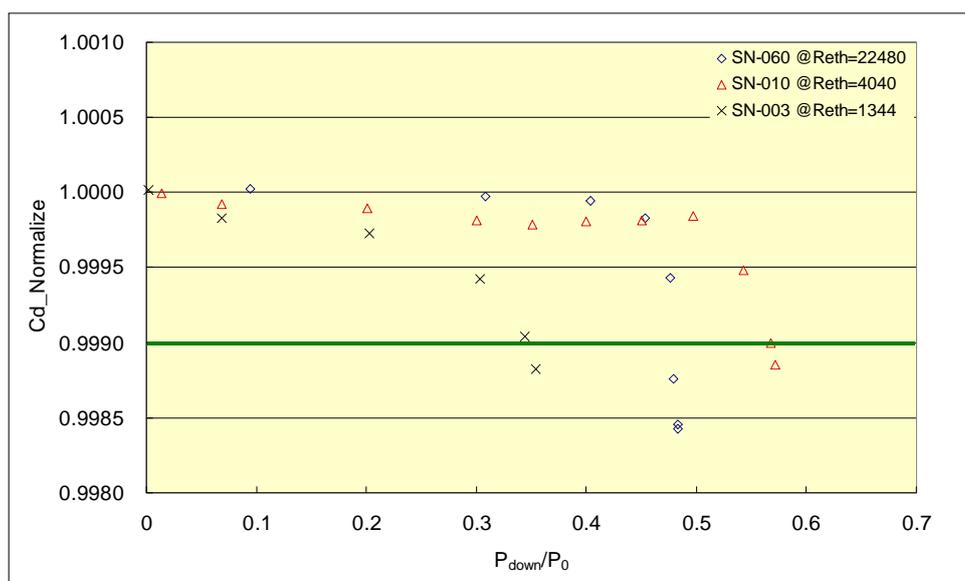


Fig. 5 Normalized C_d vs. back pressure ratio at 300 kPa inlet pressure for the three CVF's

Taking the root-sum-square of the aforementioned components and sources, as depicted in equation (3) and (4), gives the relative standard uncertainty of the measurement of C_d . Table 4 summarizes the evaluation results for the three CFV's calibrated by CMS and NIMT. The relative standard uncertainties for C_d measurements by CMS were all 0.056 %, and they were 0.068 %, 0.062 % and 0.061 % for SN-003, SN-010 and SN-060, respectively, for measurements by NIMT.

Table 4 Uncertainty budget for C_d measurement of the three CFV's

Source (sensitivity coeff.)	CMS (%)	NIMT (%)
	SN-003/010/060	SN-003/010/060
1 Mass flow (1)	0.05	0.063/0.055/0.054
2 Reproducibility (1)	0.009/0.006/0.009	0.007/0.003/0.003
3 Pressure (1)	0.006	0.011/0.015/0.015
4 Temperature (1/2)	0.005	0.005
5 Critical flow function (1)	0.008	0.008
6 Molecular weight (1/2)	0.006	0.006
7 T effects (1)	0.016	0.016
8 Critical flow effects (1)	-	-
9 Leak (1)	0.01	0.01
Combined [$c_i \cdot u(x_i)$, $k = 1$]	0.056	0.068/0.062/0.061
Expanded ($k = 2$)	0.112	0.136/0.124/0.122

3. Measurement Results

Figure 6, Fig. 7 and Fig. 8 show the C_d measurement results for nozzle SN-003, SN-010 and SN-060, respectively, plotted versus the inverse square root of the theoretical Reynolds number, Re_{th} , which is defined as follows.

$$Re_{th} = \frac{4q_{m,th}}{\pi d \mu_0} \quad (5)$$

Where d is the throat diameter of the sonic nozzle, and μ_0 is the viscosity of gas upstream of the sonic nozzles at stagnation temperature and pressure. In these figures, the first and second measurements by CMS are marked as blue triangle (CMS-#1) and blue circle (CMS-#2), respectively, and the measurement by NIMT is represented by red crosses.

The results show that, in general, the first measurement carried out at CMS, has the lowest C_d value, where as the measurement by NIMT has the highest and is relatively close to the second measurement conducted at CMS. Differences between the two measurements by CMS are within 0.05 %, showing that the TS was stable during the circulation. Since extra care was taken for temperature equilibrium and measurement in the CMS-#2 calibrations, this set of data was considered more reliable and thus was used as the reference values in this comparison.

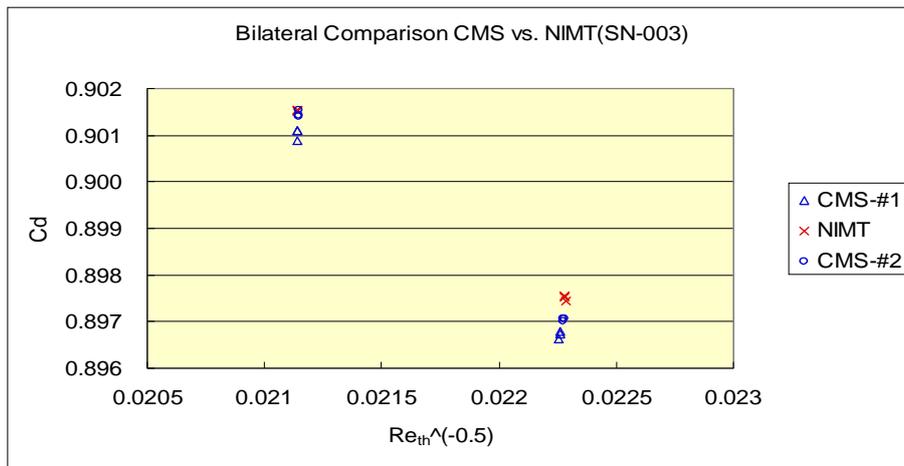


Fig. 6 Results of C_d vs. $Re_{th}^{-0.5}$ for nozzle SN-003

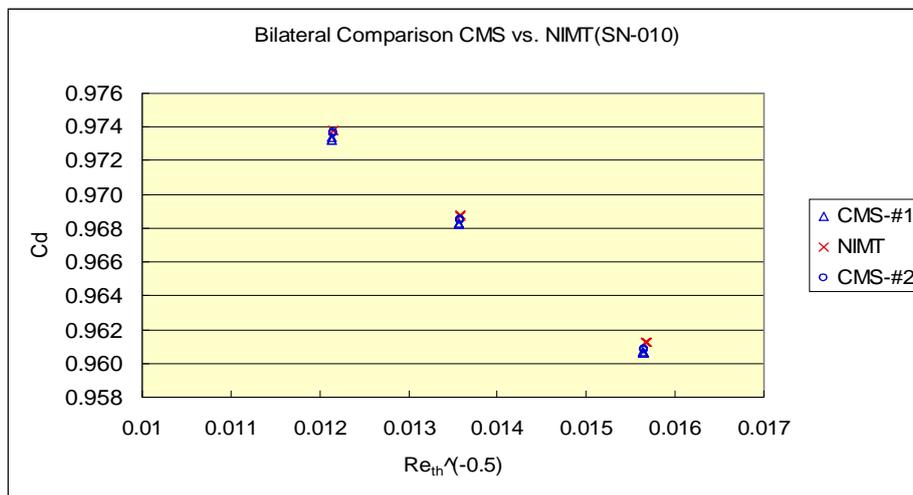


Fig. 7 Results of C_d vs. $Re_{th}^{-0.5}$ for nozzle SN-010

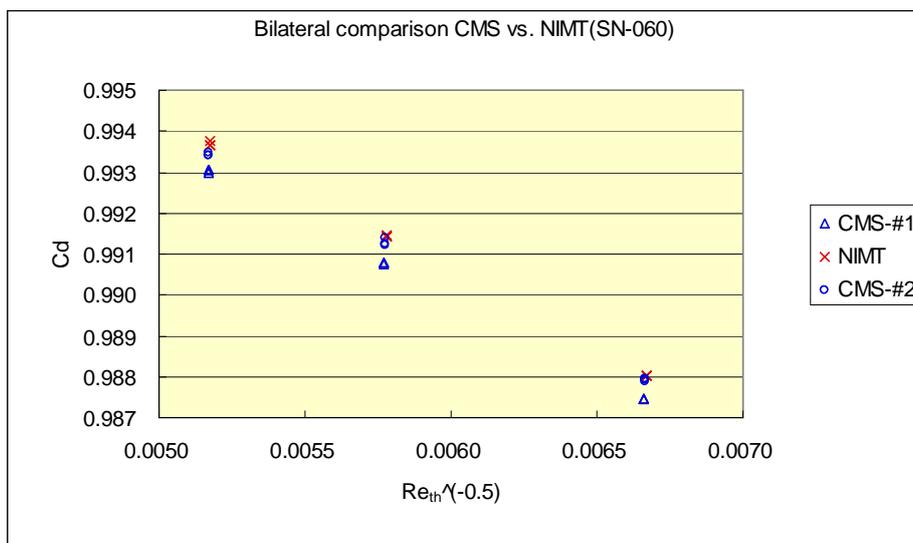


Fig. 8 Results of C_d vs. $Re_{th}^{-0.5}$ for nozzle SN-060

4. Comparison Results

As mentioned earlier, the comparison was made between the averaged discharge coefficient \bar{C}_d at each flow and the reference \bar{C}_d value based on CMS-#2 data set. Since the operational conditions among the three sets of measurement differed slightly, NIMT's \bar{C}_d values were adjusted to align with those of the CMS-#2 data at the same $Re^{-0.5}$ values. The slope $d\bar{C}_d/d(Re^{-0.5})$ used for the adjustment was based on NIMT's data. The \bar{C}_d values were then normalized by the reference values. The degree of equivalence for NIMT with reference to CMS, denoted as D_{NIMT} , was calculated as follows.

$$D_{NIMT} = \frac{\bar{C}_{d,NIMT} |_{Re_{th}(CMS-#2)} - \bar{C}_{d,CMS-#2}}{\bar{C}_{d,CMS-#2}} = \frac{\bar{C}_{d,NIMT} + \delta\bar{C}_d}{\bar{C}_{d,CMS-#2}} - 1 \quad (6)$$

Where $Re_{th}(CMS-#2)$ is the Reynolds number corresponding to the actual operational condition of the CMS-#2 data set; $\delta\bar{C}_d$ is the correction applied to make the aforementioned adjustment. The approximate 95 % confidence level ($k = 2$) uncertainty of D_{NIMT} , $U(D_{NIMT})$, can be expressed in terms of other components by the following equation.

$$U(D_{NIMT}) = \sqrt{\left(\frac{\bar{C}_{d,NIMT}}{\bar{C}_{d,CMS-#2}}\right)^2 U^2(\bar{C}_{d,NIMT}) + \left(\frac{\delta\bar{C}_d}{\bar{C}_{d,CMS-#2}}\right)^2 U^2(\delta\bar{C}_d) + U^2(\bar{C}_{d,CMS-#2})} \quad (7)$$

The degree of equivalence of NIMT to the reference was further examined by the En value, a dimensionless parameter defined as the difference between the participant's result and the reference value divided by the expanded uncertainty of the difference. The En values for NIMT's measurement results were calculated according to equation (8). Table 5 summarizes the bilateral comparison results at each flow.

$$En = \frac{|D_{NIMT}|}{U(D_{NIMT})} \quad (8)$$

Table 5 Summary of the comparison results between NIMT and CMS

Flow rate (mL/min)	$\bar{C}_{d,NIMT}$	$\bar{C}_{d,CMS-#2}$	$\delta\bar{C}_d$	D_{NIMT} (%)	$U(\bar{C}_{d,NIMT})$ (%)	$U(\delta\bar{C}_d)$ (%)	$U(\bar{C}_{d,CMS-#2})$ (%)	En
42	0.89750	0.89705	0.00003	0.054	0.136	1.6	0.112	0.30
47	0.90153	0.90145	0.00002	0.006	0.136	1.6	0.112	0.03
278	0.96125	0.96083	0.00006	0.049	0.124	0.5	0.112	0.29
374	0.96876	0.96848	0.00001	0.030	0.124	0.5	0.112	0.18
470	0.97375	0.97364	0.00001	0.011	0.124	0.5	0.112	0.06
8700	0.98803	0.98791	0.00002	0.014	0.122	1.2	0.112	0.09
11600	0.99144	0.99128	0.00002	0.019	0.122	1.2	0.112	0.11
14500	0.99370	0.99344	0.00001	0.027	0.122	1.2	0.112	0.16

In Table 5, the flows specified are approximate values at standard condition of 0 and 101.325 kPa. $U(\bar{C}_{d_NIMT})$ and $U(\bar{C}_{d_CMS\#2})$ were quoted from Table 4, and $U(\delta C_d)$ was obtained through statistic analysis of those data for the linear regression. As can be seen, all the values of D_{NIMT} are less than 0.06 % and well within the stated uncertainty. All the En values are smaller than 0.4.

5. Conclusions

A bilateral comparison between NIMT and CMS on the primary low-pressure gas flow standards was conducted to verify the measurement capability of the system, a piston prover capable of measuring flow from 5 mL/min to 24 L/min at the reference condition of 296.15 K and 101.325 kPa, at NIMT. With the use of a set of three CFV's as the transfer standard, the comparison was carried out based on calibrations of eight flows covering the range of 42 mL/min to 14.5 L/min. Discharge coefficient (C_d) was used as the parameter for the comparison and the measurement uncertainties for both NIMT and CMS were evaluated. The relative expanded uncertainty for C_d measurements by CMS was 0.112 % for all three CFV's, and were 0.136 %, 0.124 % and 0.122 % for nozzles SN-003, SN-010 and SN-060, respectively, for those by NIMT.

The difference, denoted as D_{NIMT} , between NIMT's averaged discharge coefficient at each flow and the corresponding reference value based on CMS's second calibration data set (CMS-#2), both normalized by the reference value, and the En values were analyzed to evaluate the degree of equivalence. Results showed that the difference was less than 0.06 % throughout the flow range tested, and the En values at all flows were well smaller than unity. Flow measurements by both participants of this comparison are in good agreement and the measurement capabilities claimed are thus considered valid.

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