## Intercomparison Tests of a NIST Primary Standard with a NRLM Transfer Standard for Small Mass Flow Rates of Nitrogen Gas

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### Abstract

Intercomparison tests of small gas flow measurements were carried out between the National Institute of Standards and Technology in the United States of America (NIST) and the National Research Laboratory of Metrology (NRLM) in Japan. A NRLM transfer standard package composed of sonic venturis, two pressure transducers, and two temperature sensors was calibrated using the NRLM primary standard for low gas flows (a gravimetric system) and was brought to the US in April of 1996. The NRLM transfer standard was compared to the NIST Fluid Flow Group piston provers (a volumetric system) using mass flow rates of nitrogen between 0.4 g/min and 11.5 g/min (0.3 L/min to 10 L/min). The mean differences between the NRLM transfer standard and the NIST Fluid Flow Group piston provers ranged from -0.09 % to +0.13 %.

Key words: intercomparison test, mass flow rate, piston prover, sonic venturi.

## 1. Introduction

Intercomparison tests between national laboratories are an important activity for finding and eliminating systematic errors that occur in calibration facilities and for verifying uncertainty analyses [1]. Interlaboratory comparisons determine the agreement between the primary flow standards of national laboratories and thereby enable efficient international trade. Intercomparisons in the field of gas flow measurements are particularly difficult because there are no "identity standards" of flow as there are for say length or mass, and as a result, flow standards are complex systems often involving numerous measurements (and calibrations) of length, mass, time, temperature, and pressure. In addition, the quality of the velocity profile provided for the meter under test (the approach condition) is an issue in flow calibration. Once such a complex system is put in order by thorough internal checks, comparison to another independently calibrated primary flow standard is useful to test what problems or systematic errors may remain. In this way, the international metrology community continuously refines its flow measurements.

Low mass flow rate measurements of gas are needed for semiconductor manufacturing, medical and chemical analyses, and for environmental measurements. The manufacturers and owners of flow meters used for small gas flows such as thermal mass flow meters, laminar flow elements, critical venturis, and turbines are continuously working to improve their design and accuracy [2]. Accurate primary flow standards are a necessary element for furthering the efforts to improve low gas flow rate measurements.

The Fluid Flow Group at NIST has a primary standard for low gas flows; a set of three mercury sealed piston provers which cover a flow range from 0.05 g/min to 33 g/min. The medium, and large piston provers used in this laboratory intercomparison have a flow relative uncertainty of  $\pm 0.09$  % with a 67 % confidence interval [3]. Relative standard uncertainties that provide an approximate level of confidence of 67 % will be used in this publication in accordance with current guidelines covering international intercomparisons. NIST developed a transfer standard based on a set of three redundant sonic nozzle flow measurements in 1992 that has a flow relative uncertainty of  $\pm 0.12$  %. This transfer standard has been used to perform intercomparisons between the Fluid Flow Group piston provers and other primary flow standards within the United States [4]. The Fluid Flow Group piston provers have also been compared to other primary flow standards within NIST.

In 1994, the Fluid Measurement Section of NRLM designed and built a gravimetric calibration facility and established a primary standard for small mass flow rates of gas with a maximum relative uncertainty of  $\pm 0.11$  % at a confidence level of 67 % [5]. Furthermore, NRLM has constructed a transfer standard with interchangeable sonic venturis, pressure transducers, and temperature sensors, which has been calibrated using the NRLM primary standard, and has a flow relative uncertainty of  $\pm 0.11$  % over the range of flows used in this intercomparison.

This report presents the results of an interlaboratory comparison performed in April, 1996 over mass flow rates from 0.4 g/min to 11.5 g/min (0.3 L/min to 10 L/min) using the NRLM transfer standard and the NIST Fluid Flow Group piston provers. (All volumetric flows are referenced to 20 °C and 101325 Pa conditions). The data show that the NRLM transfer standard and the NIST piston provers are in agreement within  $\pm$  0.13 %. This agreement is considered good when the uncertainties of the two facilities are taken into account, and indicates that the uncertainty estimates for the two facilities are reasonable.

## 2. The NRLM Primary Standard for Small Gas Flows

The NRLM gravimetric primary standard for small gas flows diverts flow into an evacuated cylinder, measures the mass of the cylinder before and after the collection, and divides the mass by the collection time interval to attain the mass flow rate. A description of the facility along with an uncertainty analysis has been presented previously [5]. Over the entire flow range of the facility, the worst case uncertainty of the NRLM primary standard is  $\pm 0.11$  %. The uncertainty is less than  $\pm 0.11$  % over certain flow ranges.

## 3. The NRLM Transfer Standard for Small Gas Flows

The NRLM transfer standard was designed to be easily transported to other laboratories for intercomparisons. The other design priorities were that it be rugged, have sensor redundancies, and maintain its calibration despite transportation. A photograph of the NRLM transfer standard is shown in figure 1.

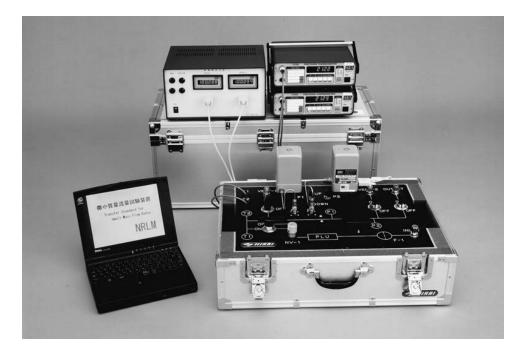


Fig. 1 The NRLM transfer standard.

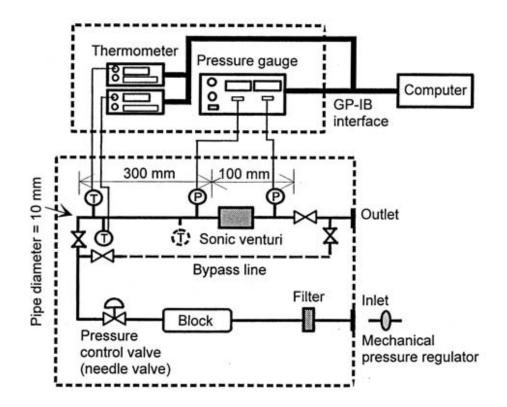


Fig. 2 A schematic of the NRLM transfer standard.

Figure 2 is a schematic representation of the NRLM transfer standard. The system includes a pressure regulator and filter, followed by a block to reduce pressure fluctuations and allow the gas to reach thermal equilibrium with the room. A needle valve is used to control the pressure upstream of the venturi and hence the flow rate. A pair of platinum resistance temperature devices (RTD's) is available to acquire the gas temperature upstream of the venturi. Two pressure transducers measure the pressure up and downstream of the venturi, allowing the user to measure the pressure ratio across the nozzle and assure critical flow through the venturi. The pressure transducers are removed for hand carrying during shipment of the transfer standard. A laptop computer uses a GPIB interface to read the pressure and temperature gauges, calculates the flow, and writes data to file. The pressure transducer and temperature sensor used in the venturi flow calculation can be changed by the user. A flowmeter or calibration facility can be connected upstream or downstream of the transfer standard depending on the measurement conditions.

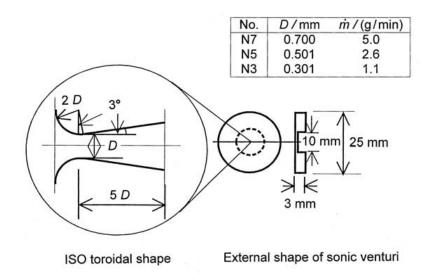


Fig 3 Schematic of the NRLM sonic venturis. ISO refers to the International Standards Organization.

Three sonic venturis have been machined into stainless steel discs that can be interchanged in a holder depending on the flow rate of interest. Figure 3 shows a drawing of the venturis as well as a table listing their throat diameters and nominal flow rates. The discharge coefficients of the sonic venturis have been fitted as a function of the theoretical Reynolds number based on calibrations with the NRLM primary standard. The theoretical Reynolds number,  $Re_{th}$ , is based on the theoretical mass flow rate,  $\dot{m}_{th}$ , calculated from the throat diameter, D, the viscosity at the stagnation conditions,  $\mu_o$ , and the throat temperature and pressure conditions. The throat pressure and temperature are calculated using stagnation pressure and temperature measurements and by assuming isentropic flow in the converging section of the venturi.

The theoretical mass flow rate is;

$$\dot{m}_{\rm th} = \left(P_0 \cdot A \cdot C^*\right) / \left(R \cdot T_0 \cdot Z\right)^{1/2} \qquad , \tag{1}$$

where *A* is the cross sectional area of the venturi throat, *R* is the gas constant, *P* is pressure, and *T* is temperature, with the subscript "0" referring to stagnation conditions. The variable *Z* is the compressibility factor and it is assumed to be unity, while  $C^*$  is the critical flow factor, calculated from the specific heat ratio,  $\gamma$ , as follows;

$$C^* = \gamma^{1/2} \cdot \left( \left( \gamma + 1 \right) / 2 \right)^{(\gamma+1)/(2-2\cdot\gamma)}$$
(2)

Using the theoretical mass flow, the theoretical Reynolds number can be calculated by:  $Re_{th} = (4 \cdot \dot{m}_{th})/(\pi \cdot D \cdot \mu_0)$ 

(3)

equation:

The discharge coefficients,  $C_d$ , for the three sonic venturis were determined from best fit equations with the theoretical Reynolds number as follows:

$$C_{\rm d} = 1.01417 - 3.72136 \cdot (Re_{\rm th})^{-1/2} \qquad \text{for N7}$$

$$C_{\rm d} = 1.00694 - 3.19550 \cdot (Re_{\rm th})^{-1/2} \qquad \text{for N5} \qquad (4)$$

$$C_{\rm d} = 1.00596 - 3.78300 \cdot (Re_{\rm th})^{-1/2} \qquad \text{for N3}$$

The mass flow rate measured by the sonic venturi, 
$$\dot{m}$$
, is calculated from the following

$$\dot{m} = C_{\rm d} \cdot \dot{m}_{\rm th} \tag{5}$$

A list of the uncertainty components for the NRLM transfer standard is given in Table 1. The uncertainty of the discharge coefficients of the sonic venturis depends upon the uncertainty of the NRLM primary standard, the repeatability of the measurements, the standard deviation of the discharge coefficient curve fit residuals, and the uncertainty of the theoretical Reynolds number. The uncertainty of the calibration facility listed in Table 1 is a 67 % confidence interval value calculated for the flow range utilized, and is not necessarily the maximum uncertainty for the NRLM primary standard. The pressure sensors used in the transfer standard were calibrated between 0 and 250 kPa using the standard piston gauge at NRLM. The relative uncertainty of the pressure measurements (including components due to the NRLM piston gauge and calibration residuals) was less than 0.04 %. The 100  $\Omega$  platinum RTD's were calibrated over the range of 15 °C to 35 °C and the uncertainties of the temperature measurements were less 0.1 °C. By analyzing the propagation of uncertainties through equations (1) and (3), the relative uncertainty of the theoretical Reynolds number calculated from the measurements of these sensors was  $\pm 0.043$  %. In the present intercomparison, three sonic venturis were operated at a pressure ratio of about 0.45 at three higher flow rates, 2.1 g/min, 3 g/min, and 11.5 g/min. However, these sonic venturis were calibrated at the pressure ratio of 0.4. Additional experiments about the relation between the discharge coefficient and the pressure ratio showed that there is a change of 0.03 % in the discharge coefficient at these two pressure ratios. This was included in the uncertainty analysis in the category labeled "operating condition". The values of the thermophysical properties used to calculate the mass flow rates were obtained from polynomial best fit functions fitted to tabulated reference data [6]. The polynomial functions fit the tabulated data to within  $\pm 0.01$  %.

Uncertainty Category	<b>Relative Standard Uncertainty</b>			
	N3	N5	N7	
Uncertainty of Calibration Facility	0.068 %	0.074 %	0.071 %	
Repeatability of Calibration Facility	0.030 %	0.030 %	0.030 %	
Uncertainty of Theoretical Reynolds Number	0.043 %	0.043 %	0.043 %	
Deviation from the Fitted Curve	0.041 %	0.051 %	0.044 %	
Operating Condition	0.015 %	0.015 %	0.015 %	
Uncertainty of Theoretical Mass Flow Rate	0.043 %	0.043 %	0.043 %	
Combined Relative Standard Uncertainty	0.11 %	0.11 %	0.11 %	

#### Table 1. Summary of Uncertainties, NRLM Transfer Standard

The combined relative standard uncertainty of the NRLM transfer standard mass flow rate is calculated by taking the root-sum-of-squares of the uncertainty components listed in Table 1. The combined relative uncertainties for the sonic venturis in the transfer standard are  $\pm 0.11$  % for all three venturis.

The transfer standard has two sets of temperature and pressure sensors to permit checks as to whether or not damage has occurred during shipping. In particular, after the flow rate is measured using one pair of sensors, a measurement of the flow rate using the other pair of sensors is repeated without changing the flow conditions. If the results of these two measurements do not agree, it indicates that some calibration drift has occurred, perhaps due to transport of the transfer standard. If both flow measurements are the same, the flow rates are considered to be measured correctly.

### 4. The NIST Fluid Flow Group Piston Provers

The NIST Fluid Flow Group operates a set of three piston provers which cover a flow range from 0.05 g/min to 33 g/min  $(3.7 \times 10^{-5} \text{ m}^3/\text{min} \text{ to } 3.0 \times 10^{-2} \text{ m}^3/\text{min})$  [3]. The three provers are mounted together in a console and connected by a manifold to a single inflow line. In the piston prover system (figure 4), the metered gas is diverted by valving into a glass cylinder to raise a mercury sealed piston. As the piston rises through the cylinder, it successively starts and stops a timer by blocking the light passing through machined slits at the ends of the collection volume. The temperature and pressure of the gas entering the collection volume are measured with a temperature sensor and an absolute pressure gauge. The temperature and pressure are used to calculate the density of the collected gas, and the density is used to convert the measured volumetric flow rate into a mass flow rate.

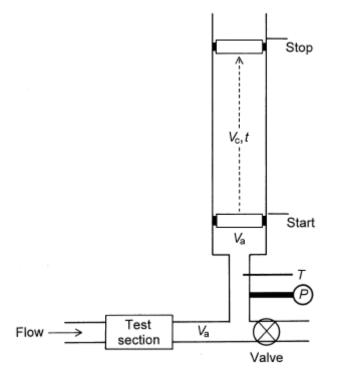


Fig. 4 A schematic of the NIST Fluid Flow Group piston provers.

The principles of mass conservation, as applied to the piston prover, can be put in the following form:

$$\dot{m} = \left(\rho_{\rm c} \cdot V_{\rm c}\right) / \Delta t + \left(\Delta \rho_{\rm a} \cdot V_{\rm a}\right) / \Delta t + \dot{m}_{\ell}$$
(6)

Here  $V_c$  is the collection volume generated by the piston displacement during the collection time interval,  $\Delta t$ . The quantity  $V_a$  is the remaining volume in the system; the volume of the flowmeter being tested, the approach piping connecting the meter under test to the cylinder, the tare volume in the prover, and tubing for pressure transducer connections. The mean gas density in the collection volume,  $\rho_c$ , is calculated from pressure and temperature measurements made during the run, and  $\Delta \rho_a$  is the change in the mean density of the gas in the approach piping that occurs during the collection interval.

The second term of (6) accounts for "storage effects" in the approach volume,  $V_a$ : if the density of the gas in  $V_a$  is increasing (due to decreasing temperature or increasing pressure), then gas is effectively "stored" in the connecting piping and the flow as measured by the piston prover is less than the flow through the meter under test during the collection interval. Conversely, if the gas within the connecting piping is expanding, then the flow determined by the piston prover is greater than the flow passing through the meter under test. The term  $\dot{m}_i$  is included to represent flows leaking from the system. Published in Metrologia, 35, pp. 211 – 221, 1998.

This term is zero or negligible due to leakage checks performed before calibrations are begun.

The uncertainty of the mass flow measurements determined by the piston prover can be analyzed by considering the uncertainties of the measured quantities in (6). The mass flow measurement is subject to uncertainties in the determination of the collection volume,  $V_c$ , the timing interval,  $\Delta t$ , and the density,  $\rho$ . The uncertainty of the gas density is due to uncertainties in the measurement of the temperature and pressure of the gas within the collection volume, as well as the goodness of fit of the best fit function used to calculate the density, and the quality of the experimental data used to determine the function. The uncertainties in temperature and pressure measurements are related to calibration quality, sampling errors, and sensor drift over time. The uncertainty of the collection volume is due to uncertainties in measuring the diameter of the cylinder and in measuring the separation between the start and stop location (the collection length), as well as the effects of thermal expansion due to room temperature variations. The uncertainty of the timing interval measurement can be traced to the uncertainty of the timer calibration, the uncertainties of its actuation by the start and stop switches, and any rocking of the piston as it passes the switches. The storage effects term of (6) becomes an error source if the change in density within the connecting piping is non-zero during the collection period due to changes in the temperature and pressure in the connecting piping.

	<b>Relative Standard Uncertainty</b>				
Uncertainty Category	Medium	Prover	Large Prover		
Collection Volume Density		0.061 %		0.053 %	
Temperature	0. 14 K	0.047 %	0. 11 K	0.037 %	
Pressure	0.022 kPa	0.022 %	0.022 kPa	0.022 %	
<b>Fitting Function</b>		0.029 %		0.029 %	
Experimental Data		0.012 %		0.012 %	
Collection Volume	0.078 cm <sup>3</sup>	0.011 %	2.448 cm <sup>3</sup>	0.033 %	
Cylinder Diameter	2.0 x 10 <sup>-4</sup> cm	0.009 %	2.3 x 10 <sup>-3</sup>	0.032 %	
•			cm		
Collection Length	5.0 x 10 <sup>-4</sup> cm	0.001 %	5.0 x 10 <sup>-4</sup>	0.001 %	
-			cm		
Thermal Expansion	$4.6 \text{ x } 10^{-2} \text{ cm}^3$	0.006 %	$4.8 \ge 10^{-1}$ cm <sup>3</sup>	0.006 %	
Collection Time	0.0102 s	0.058 %	0.0102 s	0.061 %	
Timer Calibration	$1.0 \ge 10^{-4} = s$	0.001 %	$1.0 \ge 10^{-4} = s$	0.001 %	
<b>Timer Actuation</b>	8.5 x 10 <sup>-3</sup> s	0.057 %	8.5 x 10 <sup>-3</sup> s	0.057 %	
Piston Rocking		0.012 %		0.023 %	
Storage Effects		0.007 %		0.001 %	
Combined Relative Standard Uncertainty		0.09 %		0.09 %	

Table 2. Summary of Uncertainties, NIST Fluid Flow Group Piston Provers

These uncertainties have been studied experimentally and quantified [3,7]. The results of the uncertainty analysis for the medium and large piston provers (used in the NIST - NRLM intercomparison) are given in Table 2, where it can be seen that the combined relative standard uncertainty of the flow measurement is 0.09 % for the two provers, with a 67 % confidence interval. The uncertainty values in Table 2 are combined type A and type B uncertainties, with the type B component generally much larger than the type A.

### 5. The NIST Fluid Flow Group Transfer Standard for Small Gas Flows

The NIST Fluid Flow Group transfer standard for small gas flows was used only to establish a constant flow during the present intercomparison and therefore it will only be briefly described [4]. The NIST transfer standard was designed to be portable and to redundantly measure the gas flow rate to ensure performance stability. Therefore, the design uses tandem sonic nozzles, temperature sensors, and pressure sensors with pertinent redundant checks, that permit diagnosis of sensor drift or damage. Tandem nozzles also permit checks of the stability of the flow through the transfer standard by monitoring the ratio of the up and downstream nozzle Reynolds numbers and by checking the correlation of the flow measurements made by the two nozzles.

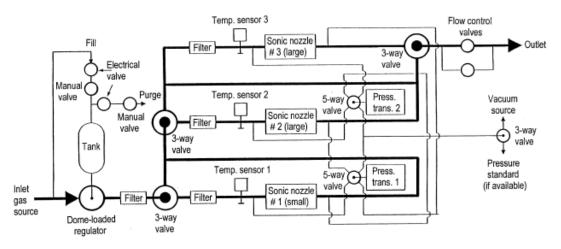


Fig. 5 A schematic of the NIST Fluid Flow Group transfer standard.

A schematic of the NIST transfer standard is shown in Figure 5. The transfer standard is comprised of three sonic nozzles, as well as three thermisters and two pressure transducers to measure the temperature and pressure of the gas upstream from each nozzle. Three-way valves permit the flow path through the nozzles to be varied, and five-way valves permit connection of the pressure transducers to various locations in the piping system up and downstream of the nozzles as well as to an external pressure calibration system. The sonic nozzles are calibrated and used at only two flow rates, 0.4 g/min and 1.0 g/min of nitrogen (0.34 L/min and 0.86 L/min, or nozzle 1 Reynolds numbers of 6700 and 16700).

## 6. Test Description

In April, 1996, comparison tests between the NRLM transfer standard and the NIST piston provers were carried out at five flow rates of nitrogen, 0.4 g/min, 1.0 g/min, 2.1 g/min, 6.0 g/min, and 11.5 g/min (0.34 L/min, 0.86 L/min, 1.8 L/min, 5.2 L/min, and 9.9 L/min). For the two smallest flow rates, the NRLM transfer standard and the NIST piston prover were set up in a parallel arrangement as shown in Figure 6a, and the NIST transfer standard was used to establish a metered, constant flow. A three-way valve was used to switch the flow between the NIST piston prover and the NRLM transfer standard. After a flow determination was made by the NRLM transfer standard, the flow was switched to the NIST piston provers to collect flow data, then the flow was switched back to the NRLM transfer standard and the process was repeated. For some tests, only one flow measurement per switch was collected by the piston prover, while for others, a set of five measurements were collected and averaged for comparison to the NRLM flows. The Reynolds number indicated by the NIST transfer standard was recorded so that corrections could be made to the comparison data if significant changes in the flow were observed over time. However, the largest change in Reynolds number observed between a change in the flow path was 0.01 % and hence corrections based on the NIST transfer standard Reynolds numbers were not made.

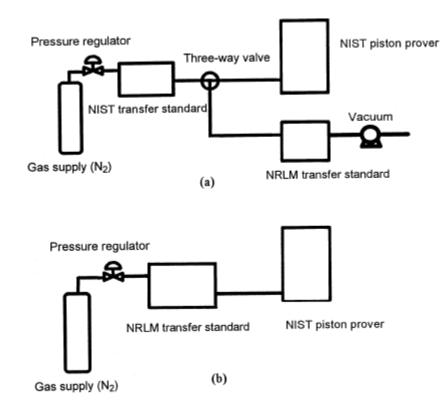


Fig. 6 Experimental setups for the intercomparison tests, a) parallel arrangement and b) series arrangement.

The flow test was set up in a parallel fashion because the NRLM venturi  $C_d$  curves had been characterized with near vacuum pressure conditions on the downstream side. Using the NRLM venturis with atmospheric pressure on the downstream side (as

needed by the mercury sealed piston prover) would result in Reynolds numbers greater than the range over which they had been calibrated. For flows greater than 1 g/min, the available vacuum pump was not large enough to maintain a critical pressure ratio across the NRLM venturis, hence the series arrangement shown in Figure 6b was used, with a pressure of about 220 kPa upstream of the venturi, and the  $C_d$  curves for the venturis were extrapolated beyond the range of the original calibration at NRLM. Upon return to Japan, the  $C_d$  values were checked over the range of Reynolds number used during the NIST intercomparison and the change was found negligible (< 0.02 %). Calibration checks of the temperature and pressure sensors used in the NRLM transfer standard were also performed upon return to Japan and the calibration drift was less than 0.01 % for all of the sensors.

The series flow arrangement allowed the measurements of the NRLM transfer standard and the NIST piston provers to be made over the same time interval for flows greater than 1 g/min. The piston prover provided one flow measurement over a 30 s or more collection interval and the NRLM transfer standard flows (updated every 2 s) were averaged over the piston prover collection interval. A set of five such flow measurements were made for each flow, and each flow was repeated on two or more occasions. Between flow repeats, a change of the sonic venturi in the holder was always made (and consequently the flow brought to zero), and usually repeats were collected on two different days.

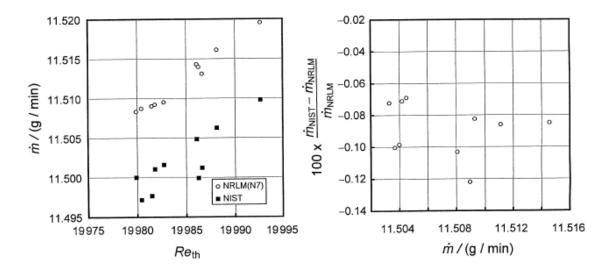


Fig. 7 Data from the 11.5 g/min case: NRLM and NIST flows versus theoretical Reynolds number, and the percent deviation between NRLM and NIST versus the NRLM mass flow rate.

### 7. Results

The results are shown in two different figures for each flow rate, one showing the mass flow rates measured by the piston prover and the NRLM transfer standard versus the NRLM transfer standard theoretical Reynolds number, and the other showing the deviation in percent of reading (using the NRLM data as the divisor), versus the mass flow rate measured by the transfer standard.

#### (1) Case of 11.5 g/min

The results for the highest flow rate tested are shown in Figure 7. The N7 sonic venturi and the large piston prover were used. The deviations between the two flow measuring systems range from -0.07 % to -0.12 %. Two sets of data were collected on two different days.

#### (2) Case of 6.0 g/min

The results at 6 g/min are shown in Figure 8. The N5 sonic venturi and the largest piston were used to collect three data sets on three different days. The flow measurements show good agreement with deviations less than 0.10 %.

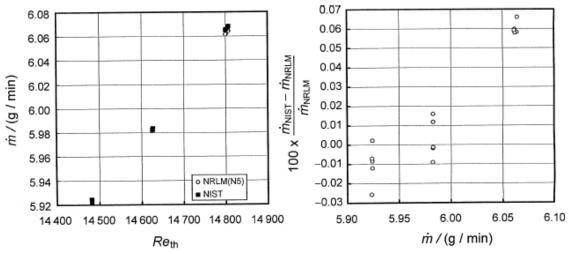
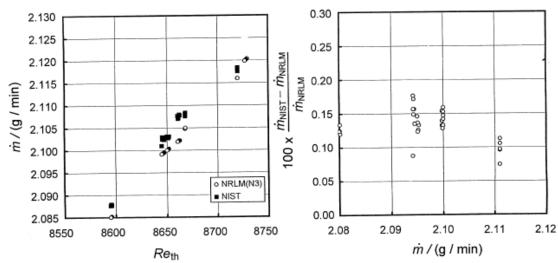


Fig. 8 Data from the 6.0 g/min case.

#### (3) Case of 2.1 g/min

The N3 sonic venturi and the medium piston were used to collect the six sets of data presented in Figure 9 which were collected on two different days. Four of the data sets involved switching between the two sets of temperature and pressure sensors available in the NRLM transfer standard while keeping the flow conditions essentially constant and observing what change occurred in the flow measured by the transfer standard. The differences in the flows measured with the two sensor sets was less than



0.02 %. The deviations between the NIST and NRLM flow measurements ranged from 0.07 % to 0.18 %.

Fig. 9 Data from the 2.1 g/min case.

#### (4) Case of 1.0 g/min

In this case, N3 and N5 were used on different occasions in the parallel arrangement along with the medium piston prover, and four data sets are plotted in Figure 10 (one set contains only two points). The data obtained using N5 are represented by the solid circles in the figure. The deviations between N3 and the piston prover vary from 0.05 % to 0.16 %, but all except two of the points fall between 0.11 % and 0.16 %. The deviations between N5 and the medium piston ranged from -0.01 % to 0.04 %.

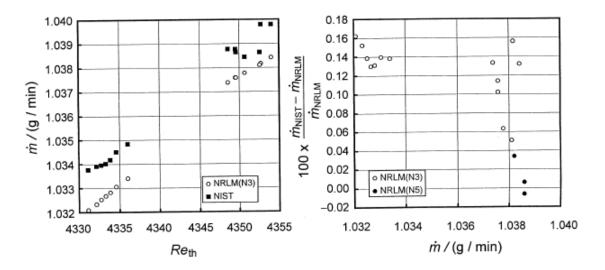


Fig. 10 Data from the 1.0 g/min case.

#### (5) Case of 0.4 g/min

Figure 11 presents the results of the comparison between sonic venturi N3 and the medium piston prover at 0.4 g/min. Three data sets collected on two days are shown (one set contains only three points). Differences between the NRLM transfer standard and the NIST piston prover are less than 0.13 %. The scatter in both this and the previous figure illustrate the increased difficulty of measuring smaller flows, or that greater scatter was introduced by the parallel flow arrangement.

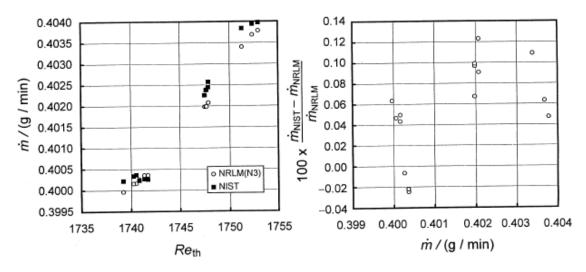


Fig. 11 Data from the 0.4 g/min case.

#### 8) Discussion and Conclusions

For the individual flow measurements gathered in this intercomparison (shown in Figures 7 - 11), the deviations ranged from -0.12 % to +0.18 %. The averages of the data sets at each flow tested are plotted in Figure 12, along with bars representing their standard deviation. The systematic differences between the two flow devices range from -0.09 % to +0.13 %, while the random differences (the standard deviation) varied between 0.016 % and 0.050 %. One can bound the differences that can be expected when the two flow systems are compared to each other by combining the uncertainties of each (taking the root-sum-of-square),  $[(0.11 \%)^2 + (0.09 \%^2)]^{1/2} = 0.14 \%$ . This 0.14 % figure bounds the deviations between the two systems expected during an intercomparison (at the 67 % confidence level). The 0.14 % bound is also shown in Figure 12, and one can see that all of the mean deviation points are within this 67 % confidence level bound. Expanding this uncertainty by a coverage factor of two indicates that with 95 % confidence, the deviations between the two facilities should be less than 0.28 %. Therefore, the deviations found during this intercomparison (which were always less than 0.18 %) show that the uncertainty analyses for the two facilities are reasonable.

There is a concave downward shape to the deviation plot in Figure 12, showing that there are systematic differences between the two facilities that seem to vary with the

flow rate. However, despite the correlation of the differences with flow rate, one cannot rigorously conclude that the differences *are a function of* the flow rate.

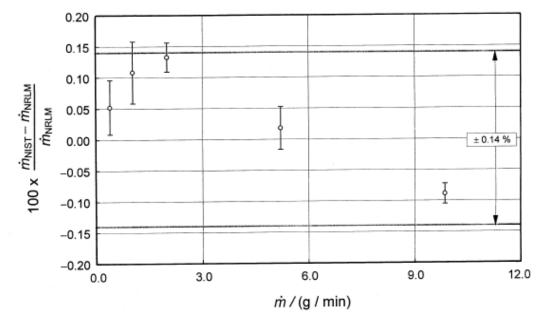


Fig. 12 The mean and standard deviation of the percent differences for each flow rate of the intercomparison. Also shown is the 67 % confidence uncertainty ( $\pm$  0.14 %) obtained from the root sum square of the NRLM transfer standard and NIST piston prover uncertainties.

The random differences are generally larger for the smaller flow rates. The standard deviations are 0.016 %, 0.034 %, 0.023 %, 0.050 %, and 0.044 % for the 11.5 g/min, 6 g/min, 2.1 g/min, 1.0 g/min, and 0.4 g/min flow rates respectively. Examining the data sets more closely, it is apparent that the scatter of deviations at each flow rate is often not truly random: there is correlation with time or the order in which the data points were collected. For some data sets the deviations are generally decreasing with time, while for others they are generally increasing. One explanation for the time correlation is storage effects, i. e., density changes (primarily due to temperature changes) in the connecting piping volume. Storage effects would be larger than normal for the parallel test arrangement due to the single collection method used which does not permit adequate temperature equilibration.

Tests conducted in March, 1996 with a laminar flow element [8] and the NIST piston provers showed 0.01 % agreement between the medium and large piston provers at a crossover flow of 1.2 g/min. Therefore, no significant discontinuity is expected in Figure 12 due to the fact that two different piston provers were used. In fact, the systematic differences appear to be a continuous function of the flow rate, this despite the fact that two different provers and three different sonic venturis were used during the intercomparison. The source of the systematic differences between the NIST Fluid Flow Group piston prover and the NRLM transfer standard is currently unclear, but will be the subject of further investigations by NIST and NRLM.

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# Nomenclature

Α	: throat area
$C_{\rm d}$	: discharge coefficient
$C^{*}$	: critical flow factor
D	: throat diameter of sonic venturi
Р	: pressure
'n	: mass flow rate
$\dot{m}_\ell$	: leakage mass flow rate
$\dot{m}_{ m th}$	: theoretical mass flow rate
Reth	: theoretical Reynolds number
R	: gas constant
Т	: temperature
Ζ	: compressibility factor
$ ho_{ m c}$	: density of the gas in the collection volume
$\Delta  ho$ <sub>a</sub>	: change in gas density in the approach volume
γ	: specific heat ratio
μ	: viscosity of gas
t	: collection time
Vc	: collection volume
Va	: approach volume, volume of connecting piping