Constant pressure primary flow standard for gas flows from $0.01\ cm^3/min$ to $100\ cm^3/min$ (0.007 μmol/s to 74 μmol/s)

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Abstract
We describe a flow standard for gas flows in the range from 0.01 sccm to 100 sccm with a relative standard uncertainty (68 % confidence) of 0.03 % at 1 sccm. (1 sccm $\equiv$ 1 cm^3/min of an ideal gas at 101325 Pa and 0 °C ≈ 0.74358 μmol/s.) The flow standard calibrates a secondary meter by withdrawing a piston from a cylinder held at constant pressure P while gas flows from the secondary meter into the cylinder. The flow standard can operate anywhere in the range 10 kPa < P < 300 kPa, and it can act as a flow source as well as a flow receiver. The flow standard incorporated features that improved its convenience and lowered its cost without sacrificing accuracy, specifically (1) dry sliding seals made with commercially available, easily replaced, o-rings, (2) a compact design based on a commercially available, hollow piston, and (3) a linear encoder with a small Abbe error.

keywords: constant pressure flow meter; gas flow; primary gas flow standard; piston prover

1 Introduction

Two ranges of industrial gas flow meet at 1 sccm; chemical process flows are typically larger, while leak rates are usually smaller. (1 cm^3/min of an ideal gas at 101325 Pa and 0 °C = 7.4358 × 10^{-6} mol/s.) The present flow standard straddles a range around 1 sccm in support of a program to detect leaks into gas masks. Its range and accuracy also make it suitable for calibrating spinning rotor gauges [1] and measuring dilute gas viscosities [2]. Its accurate performance is due in part to elements adapted from previous flow standards that were designed for smaller flows, and its simple construction and maintenance are due to novel features.

The flow standard operates by moving a piston out of a cylinder held at constant temperature T and pressure P while gas flows into the cylinder at an unknown rate. The associated molar flow rate is approximately

$$\dot{n} \cong \frac{d}{dt}\left[\frac{PV}{RT}\right] \cong \frac{P}{RT}\frac{dV}{dt}, \tag{1}$$

where t is time, R is the gas constant and V is the volume of the cylinder. Hayward and Jepsen [3] were the first to use this simple principle to generate a small gas flow into a vacuum system. Peggs' 1976 review [4] mentions their work and that of Smetana [5], who varied the volume with a sealed bellows instead of a piston. Table 1 lists more recent primary flow standards that operated at constant pressure and somewhere in the range $0.001 < \dot{n} < 100$ sccm.

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Table 1. Primary gas flow standards that operated in the range $0.001 < \dot{n} < 100$ sccm by changing a gas-filled volume held at constant pressure. (Not included are gravimetric methods (e.g. [6]) and standards whose seal is liquid mercury (e.g. [7]) or gas lubrication (e.g. [8]).) Most acted only as a source (s) while some could act as either a source or a receiver (s/r). Here, *D* is the effective diameter of the piston or calibrated bellows, $\dot{n}_{\min}$ and $\dot{n}_{\max}$ are the minimum and maximum flow rates, and *u* is the flow standard's best relative standard (*k* = 1) uncertainty.

reference	s/r	type	*D* mm	$\dot{n}_{\min}$ sccm	$\dot{n}_{\max}$ sccm	*u* %
[9] 1987 McCulloh	s	piston + cylinder + custom PTFE seal	10,25	1×10^{-4}	0.13	0.1
[9] 1987 McCulloh	s	piston + bellows + oil-filled cylinder	10,25	1×10^{-5}	0.13	0.1
[10] 1988 Thornberg	s	piston + sliding seal + lubricated o-ring	13	1×10^{-4}	0.13	1.1
[11] 1992 Hufnagel	s	piston + lubricated o-rings	25	8×10^{-4}	80	1.
[12] 1993 Jousten	s	calibrated bellows	12	5×10^{-6}	0.05	0.15
[13] 1995 Jitschin	s	piston + lubricated o-rings	25	5×10^{-4}	50	0.1
[14] 1996 Li	s/r	piston + bellows + oil-filled cylinder	10,25	5×10^{-6}	13	2.
[15] 1999 Jousten	s	calibrated bellows	12	5×10^{-7}	13	0.2
[16] 2002 Jousten	s	calibrated bellows	12	5×10^{-7}	13	0.14
[17] 2003 Calcatelli	s	piston + cylinder + custom PTFE seal	5,20	1×10^{-5}	0.7	0.2
[18] 2004 Berg	s/r	piston + bellows + oil-filled cylinder	102	1×10^{-1}	270	0.02
[19] 2005 Cignolo	s/r	piston + lubricated o-ring	120	9×10^{-2}	1900	0.01
[20] 2006 Kramer	s/r	double piston	16	1×10^{-3}	1.3	0.03
[21] 2007 Cheng	s	piston + bellows + oil-filled cylinder	?	5×10^{-6}	0.05	0.57
[22] 2008 Gronych	s	calibrated bellows	9,30	3×10^{-4}	55	1.
this work	s/r	piston + FEP encapsulated o-ring	25	1×10^{-2}	100	0.03

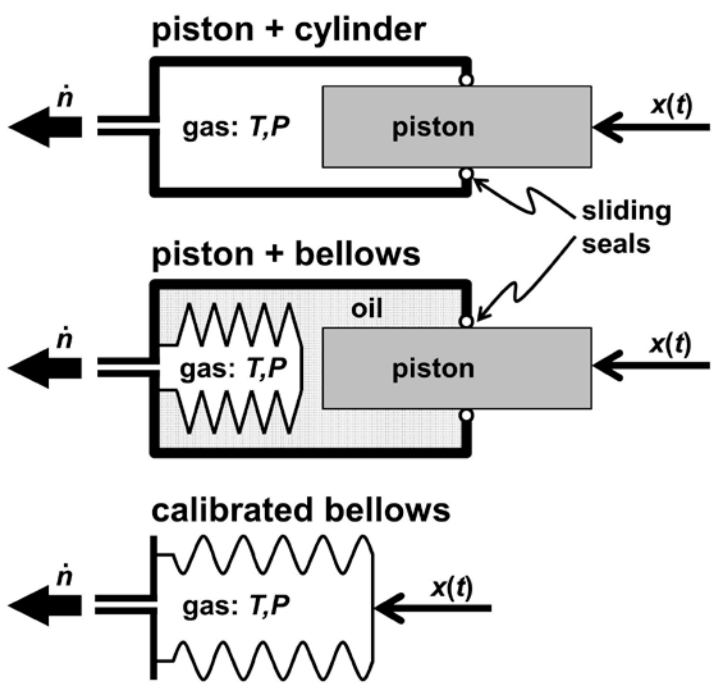

Figure 1. Three types of constant-pressure gas flow standards.

Figure 1 illustrates the three types of flow standard listed in Table 1, all of which operate by changing a gas-filled volume held at constant pressure. The first type, piston + cylinder, [9-11,13,17,19] uses a piston that is inserted into a gas-filled cylinder through a sliding seal. Its chief advantage is simplicity: the volume change is determined from the piston's cross-section area and displacement. The sliding seal is its chief disadvantage; outgassing from a polymer seal is a concern, and the pressure difference across the seal must be kept small to prevent a significant leak.

The second type, piston + bellows, [9,14,18,21] contains the gas in a metal (welded diaphragm) bellows, thereby eliminating the sliding seal and its potential for outgassing or a leak. Both the bellows and the piston are contained in an oil-filled volume so that the volume displacement of the bellows is known from the displacement of the piston. The oil is effectively incompressible, but only if it excludes gas bubbles; the flow standard built by McCulloh et al. [9] does so by attaching a weight to the bellows so that the oil pressure is always greater than one atmosphere. The additional complication of a long thermal time constant occurs if the oil volume is large [18].

The third type of flow standard, calibrated bellows, [12,15,16,22] also has no sliding seal, and it eliminates the complications due to the oil by driving the bellows directly. Its chief disadvantage is that the bellows volume $V(x)$ must be calibrated as a function of its linear displacement x. Jousten et al. [12] found that $V(x)$ was more linear for a formed bellows than for a welded diaphragm bellows, although Gronych et al. [22] later were able to use a welded diaphragm bellows despite its nonlinearity.

Most of the devices in Table 1 were not flow receivers that measured an unknown flow directly but instead were flow sources that produced an accurately known gas flow. A common use of the flow source was to generate a known pressure to calibrate vacuum gauges. A known flow source can be used also to measure an unknown flow, but doing so requires adding a device (with a corresponding uncertainty contribution) to compare the known and unknown flows. The present flow standard can act as both a flow source and a flow receiver.

The present flow standard is a piston + cylinder device, and it incorporated several advances:

- The sliding seals were made with commercially available, easily replaced, o-rings. No lubrication was needed because the o-rings were encapsulated in FEP (fluorinated ethylene propylene), which has a coefficient of friction almost as low as PTFE (polytetrafluoroethylene). Previous flow standards with dry sliding seals used custom PTFE rings backed by elastic o-rings [9,17].
- The piston was a commercially available design with a hollow center that allowed an efficient, compact design.
- The displacement was measured simply and accurately by a linear encoder. Previous flow standards used either a micrometer drive screw, which is less accurate, or a laser interferometer, which is more complicated and expensive.

2 Theory of operation

See Figure 2 and Figure 3. The heart of the flow standard is a 25 mm diameter piston, which is withdrawn from the cylinder while gas flows into the cylinder. Proportional-integral (PI) feedback from a differential pressure gauge adjusts the rate of displacement so that the pressure in the cylinder is held constant. The resulting molar flow rate into the cylinder is determined from the pressure P, the temperature T, the piston cross sectional area A, and the time derivative of the piston's displacement x as follows.

$$\dot{n} = \frac{d}{dt}\left[\frac{P\left(V_{\text{out}} - Ax\right)}{RT\left(1 + B_P P\right)}\right] \cong -\frac{PA}{RT}\frac{dx}{dt}. \tag{2}$$

Here B_P is the second pressure virial coefficient that characterizes small deviations from ideal gas behavior, and V_{out} is the volume of the cylinder when the piston is at its outermost position (x = 0). The flow standard can operate also as a flow source by driving the piston into the cylinder.

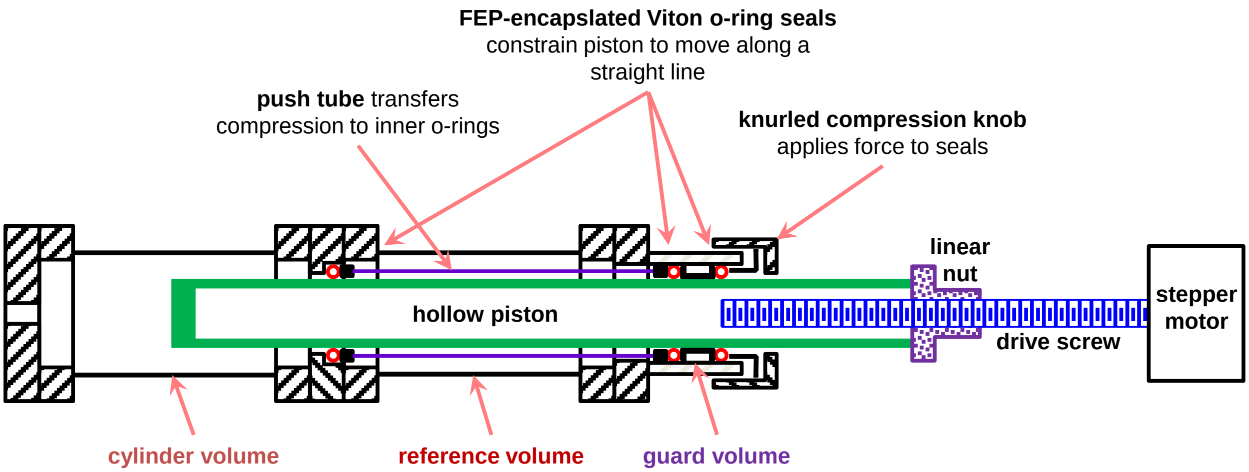

Figure 2. Cross section of the piston-and-cylinder assembly.

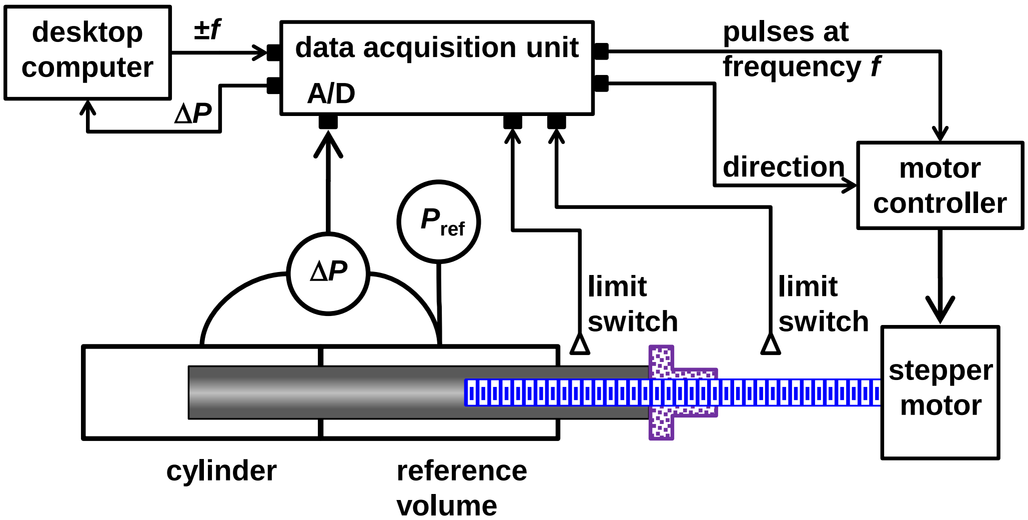

Figure 3. Feedback adjusts the speed of the stepper motor so that the differential pressure ΔP between the cylinder and the reference volume stays constant while gas flows into the cylinder.

The cylinder pressure P is not measured directly. Instead, it is computed as the sum $P = P_{ref} + \Delta P$, where P_{ref} is the pressure in the adjacent reference volume and ΔP is the differential pressure between the two volumes. Keeping ΔP small makes negligible the leak across the sliding seal that separates the two volumes. This indirect method minimizes the cylinder volume by excluding the volume of the reference pressure gauge. It also prevents errors caused by variations of the temperature of the gas contained in the reference pressure gauge, which sits outside the thermal enclosure. The reference volume is sufficiently long that the portion of the piston that is inserted into the cylinder is never exposed to humid air outside the cylinder; this minimizes outgassing caused by desorption of water from the piston. The use of a reference volume, sliding seals, and feedback control of pressure are based on NIST experience with flow standards that have operated at flow rates from 10^{-5} sccm to 10^{3} sccm [9,18].

3 Components

The flow standard comprises a thermal enclosure, the piston-and-cylinder assembly, the gas manifold, and the electronic instrumentation.

3.1 Thermal enclosure

Figure 4 shows the thermal enclosure that maintains the temperature of the piston-and-cylinder assembly near 25 °C. Its height and area are respectively 0.4 m and (1.1×0.5) m^2. The walls and top are rigid sheets of 25 mm polyisocyanurate insulation glued to 4 mm sheets of corrugated polypropylene (Coroplast [23]). The bottom is similar except that the insulation is sandwiched between two sheets of 6 mm plywood. Hook-and-loop fasteners (Velcro [23]) ensure tight seals while allowing quick access to the interior of the enclosure.

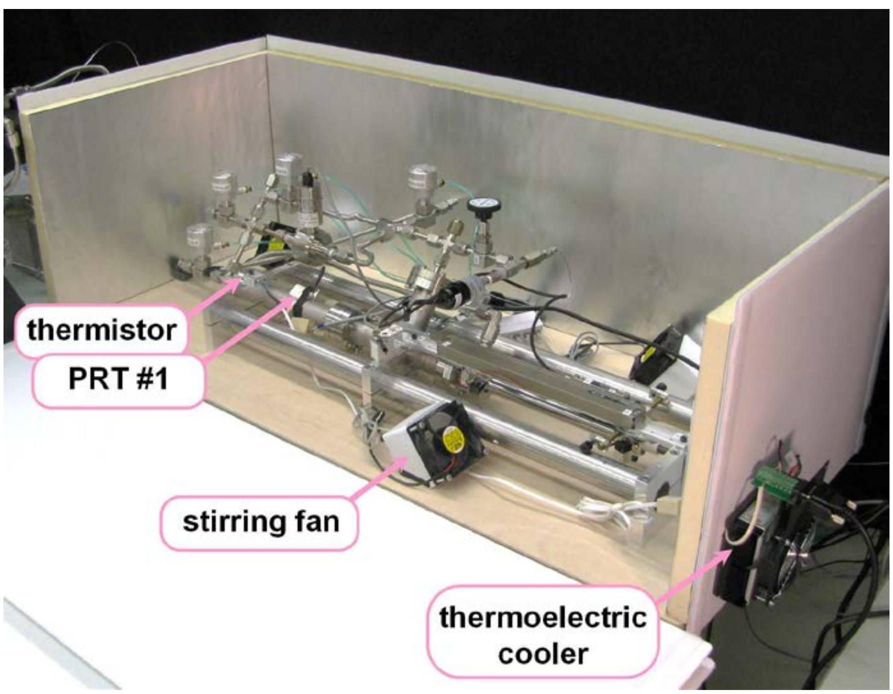

Figure 4. The thermal enclosure with the top and front side removed.

Temperature uniformity is maintained by four small stirring fans. Temperature stability is maintained by a controller that monitors a thermistor bolted to one of the strut clamps and drives a thermoelectric cooler. Cooling is required because the stirring fans dissipate 10 W and the enclosure has a conductance of only 2 W/K; with no cooling the enclosure temperature would be 5 K warmer than ambient. The only other significant heat source is the 0.5 W light located inside the encoder carriage; its influence was minimized by constructing the carriage mount plate from rigid plastic (Bakelite).

3.2 Piston-and-cylinder assembly

Figure 5 shows the piston-and-cylinder assembly. Its central part is the 500 mm long piston that penetrates the cylinder, reference, and guard volumes. Two support rails parallel to the piston hold the assembly together, and, as shown in Figure 2, three internal o-rings seal the volumes and define the axis of the piston's sliding motion. Stainless steel tubing connects the volumes to valves and the gauge that measures the differential pressure between the cylinder and reference volumes.

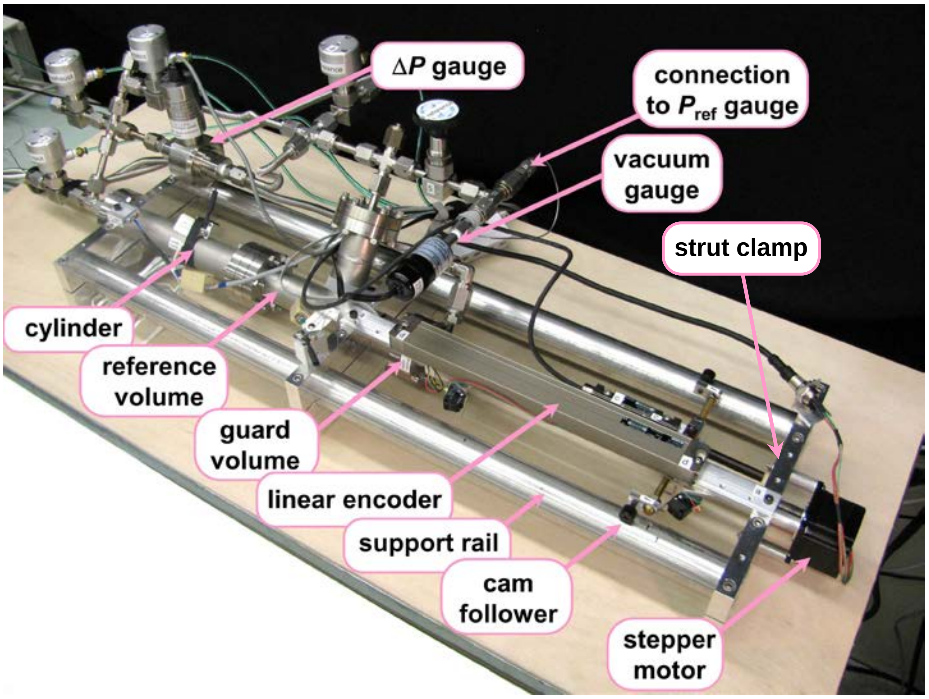

Figure 5. The piston-and-cylinder assembly. Three strut clamps hold the stepper motor, the cylinder volume, and the reference volume between the two support rails.

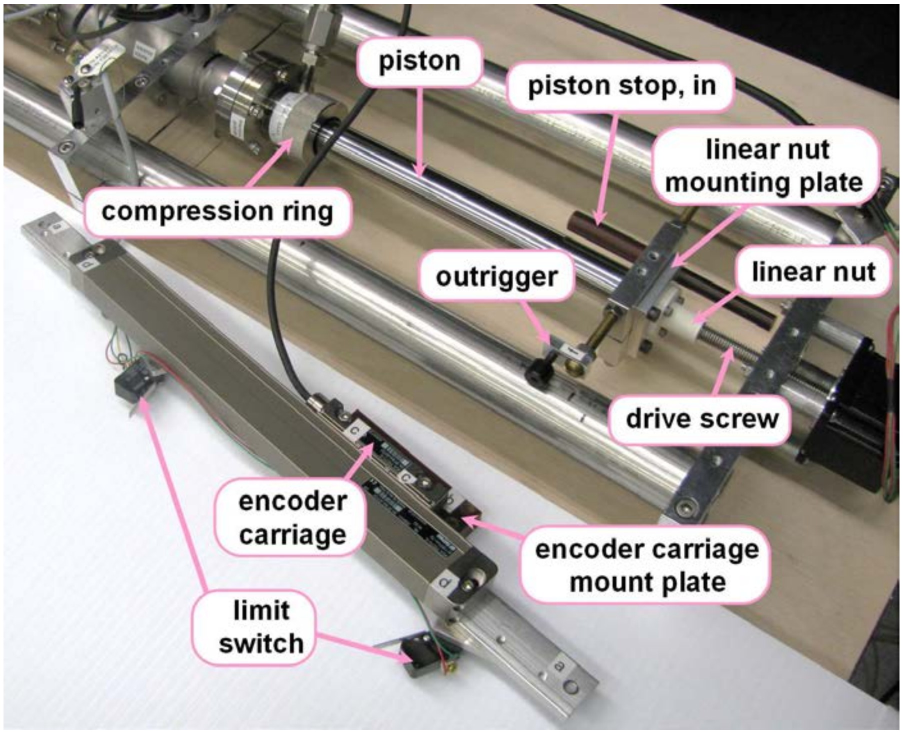

Figure 6. View of the piston with the linear encoder removed. Parts attached to the base of the piston normally include the linear nut, the two outriggers, the two piston stops, and the encoder carriage.

The piston is driven by an Acme screw that is rotated by a size 23 stepper motor, and the available force exceeds 500 N at all speeds, which is sufficient to overcome the forces due to friction at the sliding seal and a cylinder pressure as large as 300 kPa. The screw mates with a linear nut attached to the base of the piston. Rotating the screw moves the hollow piston out of the cylinder and pulls it over the screw. (See Figure 2.) Using a hollow piston makes the flow meter's drive train more compact, which reduces the flow standard's footprint and tightens the connection from the motor to the piston. Rotation of the piston is prevented by a pair of outriggers attached to the piston base. At the tip of each outrigger is a (cam follower) roller bearing that touches the support rail.

The piston (Misumi PSPJT25-500-M20-DKC-VC-K20 [23]) was purchased as a "hollow linear shaft" with a hard chrome plating, an internal thread at one end, and a diameter specified to be (25.000 + 0.000 – 0.009) mm. A micrometer referenced to a gauge block was used to measure diameters at 18 locations along the shaft; the average value and standard deviation were

$$D = \left(24.9977 \pm 0.0022\right)\ \mathrm{mm}, \tag{3}$$

which is consistent with the specification. The tip of the piston is sealed by an o-ring held between the lip of the hollow piston and a stainless steel bolt threaded into the piston; the bolt's hexagonal head was machined to the same diameter as the piston.

The piston's motion is read by a linear encoder. The encoder body is attached to the middle and right strut clamps, and the encoder carriage is attached to the moving base of the piston. The plate that holds the encoder and limit switches can be attached and detached from the assembly without disturbing the alignment between the encoder and the piston.

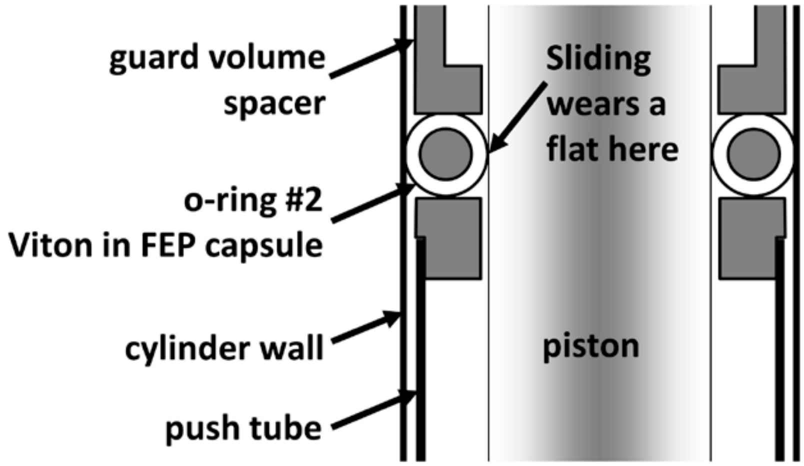

Figure 7. The push tube, which is a stainless steel tube with Delrin ends, transmits the compression force to the innermost o-rings. The Viton o-ring is contained in an FEP capsule whose wall is sufficiently thick that the seal can be improved significantly by wearing a 1 mm flat onto the inner diameter.

Figure 2 shows how the three o-rings separate the cylinder, reference, and guard volumes. They are made of Viton [23] encapsulated in FEP (fluorinated ethylene propylene) with inner and minor diameters of 25.0 mm and 2.6 mm (Marco Rubber T1001-214 [23]). The wall of the FEP capsule is 0.3 mm thick, and deliberately wearing a 1 mm flat onto the inner diameter, as shown by Figure 7, significantly improves the seal against the piston. A small tunnel in the double Conflat flange that holds o-ring #1 vents the volume behind the o-ring. Tightening the knurled compression knob increases the o-ring compression force, which is distributed among all three o-rings by the tubular guard volume and push tube indicated in Figure 2 and Figure 7.

3.3 Gas manifold

Figure 8 is a schematic of the gas manifold, which can be configured to allow the following:

- Vacuum pumping to remove unwanted gases such as air and water.
- Pressurizing or evacuating selected volumes to detect leaks and outgassing.
- Setting up a run by allowing flow through the device under test without piston motion.
- Operating the flow meter at a pressure in the range from 10 kPa to 300 kPa.

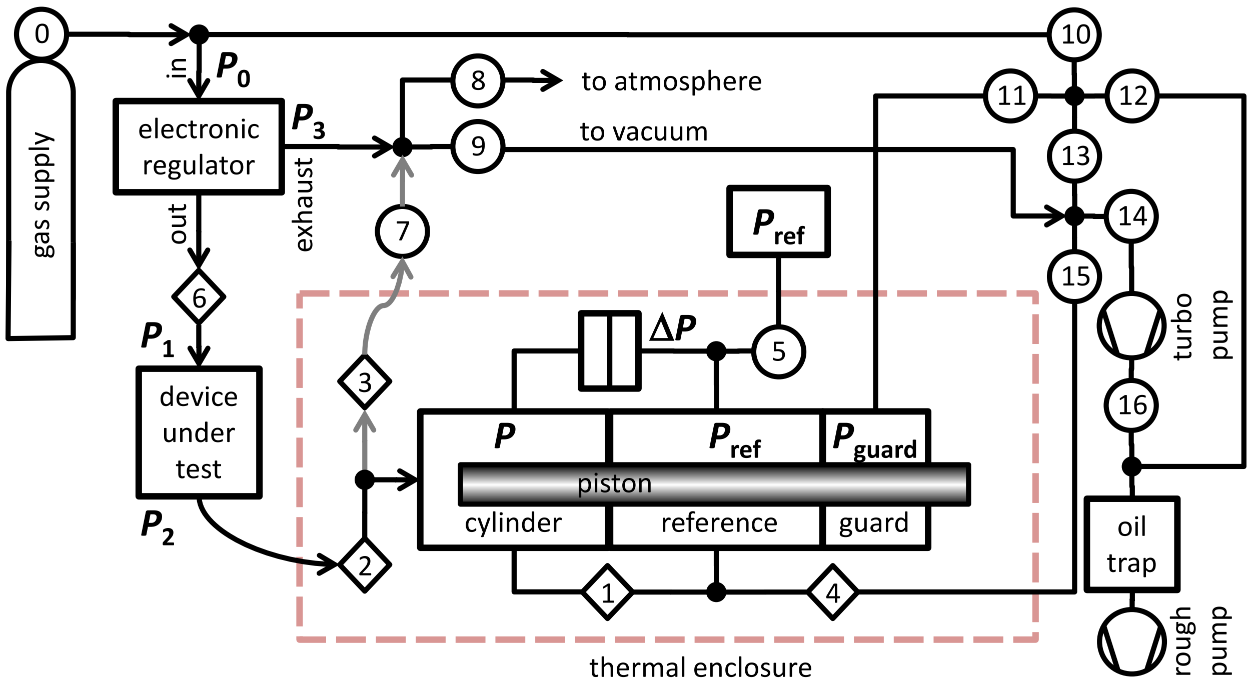

Figure 8. Schematic of the gas manifold. Pneumatic valves are indicated by diamonds. The curved connections to valves 2 and 3 indicate steel capillaries. In typical operation as a flow receiver, gas initially flows through the regulator, the device under test, and then to atmosphere through valve 3. Closing valve 3 and controlling $\Delta P = 0$ causes the piston to withdraw from the cylinder. Operating the instrument as a flow source requires rearranging the connections to the regulator and the device under test.

Valves 1 through 4, which are attached to the piston-and-cylinder assembly, are pneumatically operated to allow automatic operation and to avoid the temperature disturbance caused by opening the thermal enclosure. The remaining valves are located in a small rack that also holds an electronic pressure regulator and a turbo pump. The main pressure gauge and a thermocouple vacuum gauge monitor the reference volume, and a second vacuum gauge monitors the turbo pump.

3.4 Electronics

All of the electronic instruments were obtained commercially, two of which are attached to the piston-and-cylinder assembly:

- Enclosed linear encoder, 200 mm length, 0.5 um resolution, 5 um/m accuracy.
- Differential pressure gauge, ±1200 Pa scale, 2.4 Pa accuracy, 3.5 MPa proof pressure.

Some of the other instruments were:

- Pressure gauge, 0-310 kPa, 31 Pa accuracy.
- Thermoelectric temperature controller, 60 W, 12 V, ±5 A, 0.01 K 24 h stability.
- Stepper motor controller and power supply, 52 V, 5.8 A, 300 W.
- USB data acquisition unit (Measurement Computing USB-1208HS [23]) with:
  - digital I/O (used to monitor the limit switches)
  - ±10 V analog input (used to measure ΔP)
  - timer output (used to generate stepper motor pulses)
- electronic pressure controller, 0-310 kPa range, 0.1 kPa resolution.

4 Control software

The flow standard is controlled through a LabVIEW [23] program that is a stacked sequence structure containing three code blocks. The first and last blocks handle general functions such as initializing communications and creating and releasing data structures needed for user events. The middle block contains nine parallel loops that perform the functions listed in Table 2; the loops run simultaneously at rates that are appropriate to the individual functions. The fastest loop handles the PI (proportional-integral) feedback loop.

Table 2. Monitoring and control loops in the control program. The loops run simultaneously with the indicated periods.

loop function	period (s)	note
control piston speed	0.01	update PI feedback loop based on $\Delta P(t)$
monitor piston position	0.05	
monitor limit switches	0.05	can generate "piston limit" event
monitor differential pressure	0.1	average 5×10^4 samples of A/D converter
monitor reference pressure	0.3	can generate "overpressure" event
monitor device under test	1.	get pressure, temperature, and reported flow rate
monitor enclosure temperature	1.	
monitor cylinder temperature	10.	
handle events		see Table 3

The event handler loop handles events, which can be generated by the user (e.g. a mouse click), automatically by the program, or both. Table 3 lists important events that are handled by the event handler loop.

Table 3. Events handled by the event handler loop. Depending on its function, the event can be generated by the user, automatically by the control program, or both.

event	user	auto	note
set regulator pressure	✓		
set up run	✓		move piston to start position, set valves, read pressures
open valve *N*	✓	✓	
start run	✓		
stop run	✓	✓	
save data on timeout		✓	save data at rate specified by user
overpressure		✓	protect pressure gauge
piston limit		✓	reverse piston motion until limit switch is disengaged
piston control on	✓	✓	turn on PI feedback loop
set motor pulse rate	✓	✓	output of PI feedback loop
move piston in	✓	✓	
move piston out	✓	✓	

The combination of the stepper motor, motor driver, and drive screw caused the piston to move with a step of 4 μm per motor pulse, and the PI loop sent pulses to the stepper motor driver at the frequency

$$f(t) = k_P\left(\Delta P(t) - \Delta P_0\right) + k_I \int_0^t \left(\Delta P(t') - \Delta P_0\right) dt', \tag{4}$$

where ΔP_0 is the offset of the differential pressure gauge. The coefficient values k_P = 1 Hz Pa^{-1} and k_I = 0.2 Hz Pa^{-2} gave satisfactory behavior over the full range of flow rates.

5 Set up

Setting up the flow standard includes seating the o-rings, aligning various parts, measuring the cylinder volume, and checking for leaks.

New o-rings must be installed and seated by wearing a 1 mm circumferential flat onto their inner diameters, as shown in Figure 7. This is done by sliding the o-rings and spacers onto the piston, inserting the piston into the cylinder-and-piston assembly, lightly tightening the compression knob, and then stroking the piston by hand. The compression knob is then tightened to its final position, as shown in Figure 9.

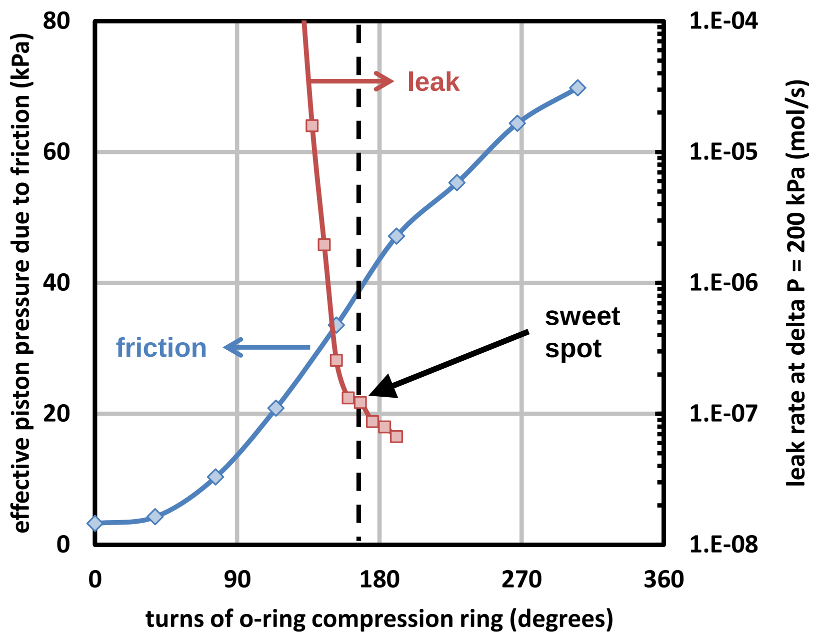

Figure 9. Typical dependence of leak rate and friction on the angle of rotation of the compression knob. Tightening the compression knob to about 160° past initial contact brings it to a "sweet spot" at which both the sliding friction and the leak rate across o-ring #1 are sufficiently small.

The motor must be centered so that its force on the piston is purely longitudinal.Otherwise, the drive screw will bend slightly and create a transverse force that may cause a leak past an o-ring. The transverse force may also cause the angle between the piston and the encoder to change during the piston stroke, thereby creating Abbe error, as shown in Figure 10. The encoder body must be aligned parallel to the piston to eliminate cosine error.

See Figure 6. The outriggers limit the rotation of the piston about its axis, and the heights of their roller bearings must be adjusted to prevent binding of the encoder and allow small rotations of the piston. Typically, one bearing is about 1 mm above its support rail when the other bearing touches its support rail. This “play” prevents the outriggers from overconstraining the piston, which could apply transverse force on the o-rings.

Ideally, the gas in the cylinder is held at constant temperature and pressure, but in practice, small changes in those quantities occur that may affect the flow measurement. For example, a pressure drift of dP/dt = 1 Pa/s and a cylinder volume of $V_{\text{out}} \approx 200\ \text{cm}^3$ will create an apparent molar flow of

$$\dot{n}_{dP/dt} = \frac{V_{\text{out}}}{RT}\frac{dP}{dt} \simeq 0.1\ \text{sccm}, \tag{5}$$

when the piston is stopped at $x = 0$. An accurate value of V_{out} in Eq. (2) will eliminate errors due to pressure and temperature drifts, and it can be

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measured to within 0.3 % by creating a small pressure drift by increasing the pressure in the guard volume above that in the cylinder. One then measures a small flow with and without the pressure drift and chooses the value of V_{out} that minimizes the error caused by the pressure drift. This method is indirect but useful because it includes the effective volume contained between the flow standard and the device under test.

Three types of leak can cause a significant error: a leak across o-ring #1, a leak to atmosphere, and outgassing. Each of these leaks can be measured by setting up an appropriate test condition and observing the drift of either P or ΔP at steady temperature. Determining the leak across o-ring #1 at the operating condition $\Delta P < 10$ Pa requires an extrapolation from measurements made at large ΔP.

6 Operation

Varying the cylinder pressure P is useful for determining the pressure dependence of a flow impedance [2], and the cylinder pressure P can be set anywhere in the range 10 kPa < P < 300 kPa. Pressures outside this range are not practical; below 10 kPa the required piston speed is too fast for larger molar flow rates, and above 300 kPa the piston force can be too large for the stepper motor.

See the gas manifold, Figure 8. The configuration of the valves depends on whether the flow standard will operate below, near, or above atmospheric pressure P_{atm}. The rough pump is needed to obtain $P < P_{\text{atm}}$ in the cylinder and, when regulating at input pressure $P_1 < P_{\text{atm}}$, at the exhaust port of the pressure regulator. The turbo pump is useful for removing adsorbed water from the walls of the cylinder. Note the capillary connections to valves 2 and 3; they reduce the volume outside the thermal enclosure, which minimizes the error caused by variations of room temperature.

In typical operation, the flow standard is used to calibrate a device under test (DUT) that receives gas at P_1 and exhausts gas into the flow standard at $P_2 \approx 1$ atmosphere. A calibration run then comprises the following steps.

1. The user sets up the run. The control program moves the piston to its innermost position and then measures atmospheric pressure P_{atm}, the pressure tare of the device under test, and the offset ΔP_0 of the differential pressure gauge. Valves 2, 3, and 6 are opened to allow flow through the DUT, past the connection to the cylinder, and out to atmosphere.
2. The user defines the input flow rate, usually by setting P_1.
3. The user starts the run. The program then closes valves 1 (bypass) and 3 (flow exhaust) and starts the PI feedback loop.
4. As gas flows into the cylinder, the program withdraws the piston at a rate that keeps $\Delta P = \Delta P_0$, and it periodically records P, T, x, and the values reported by the DUT.
5. The run ends when the piston reaches the outer limit switch.

Unlike a typical DUT, the flow standard does not measure the flow rate directly; instead it periodically measures the amount of gas that has accumulated in the cylinder at time t,

$$n(t) = \frac{P\left(V_{\text{out}} - Ax(t)\right)}{RT\left(1 + B_p P\right)}. \tag{6}$$

The subsequent analysis obtains the difference between the flow rates reported by the DUT and the flow standard as follows:

$$\dot{n}_{\text{DUT}} - \dot{n} = \frac{d}{dt}\left[\int_0^t \dot{n}_{\text{DUT}}(t')\,dt' - n(t) + n(0)\right]. \tag{7}$$

Plotting the molar difference n_{DUT} - n as a function of time is useful because the slope of that difference is a direct and sensitive indication of the difference in flow rates.

7 Uncertainty

When the flow standard is used for a calibration, the total relative uncertainty of the calibration u_{cal} is the quadrature sum of the Type A (statistical) uncertainty u_{DUT} of the device under test (DUT), the Type A uncertainty u_{A} of the flow standard, the Type B u_{B} uncertainty of the flow standard, and the uncertainty u_{C} caused by the connection between the DUT and the standard:

$$u_{\text{cal}} = \left(u_{\text{DUT}}^2 + u_{\text{A}}^2 + u_{\text{B}}^2 + u_{\text{C}}^2\right)^{1/2}. \tag{8}$$

Often, a DUT that operates near 1 sccm is a device that measures P_1 and P_2, the pressures at the entrance and exit of a laminar flow impedance. The DUT's Type A uncertainty is then typically due to the precision of the pressure tare, which is the difference P_1 - P_2 measured at zero flow rate.

The flow standard's Type A uncertainty is defined as the normalized uncertainty of the time derivative of the number of moles contained in the cylinder, $u_A = u(\dot{n}) / \dot{n}$.

Table 4 lists the contributions to the Type B uncertainty of the flow standard. The largest component is due to the average piston diameter D; further improvement would require characterizing the diameter as a function of displacement.

The second largest component is due to the angle θ between the piston and the encoder axes. Cosine error is negligible, but Abbe error can be significant because θ can depend on the piston's displacement x. The relative Abbe error is

$$u_{\text{Abbe}} = \frac{d}{L_{\text{stroke}}} \tan(\Delta\theta), \tag{9}$$

where d is the separation between the piston and encoder axes, L_{stroke} is the length of the piston stroke, and $\Delta\theta$ is the change of $\theta(x)$ that occurs during the stroke. Ideally, $\Delta\theta = 0$ because the piston's axis is defined by the o-rings and therefore fixed with respect to the encoder. However, as mentioned earlier, a misalignment of the motor can bend the drive screw and apply a transverse force to the piston. The bending and the resulting transverse force will depend on the piston's position x, which will vary the orientation of the piston axis and cause $\Delta\theta \neq 0$. The value $\Delta\theta = 0.6$ mrad was calculated by estimating the deviation of the piston axis from the center of o-ring #3 to be no more than 0.3 mm and dividing it by the 500 mm length of the piston.

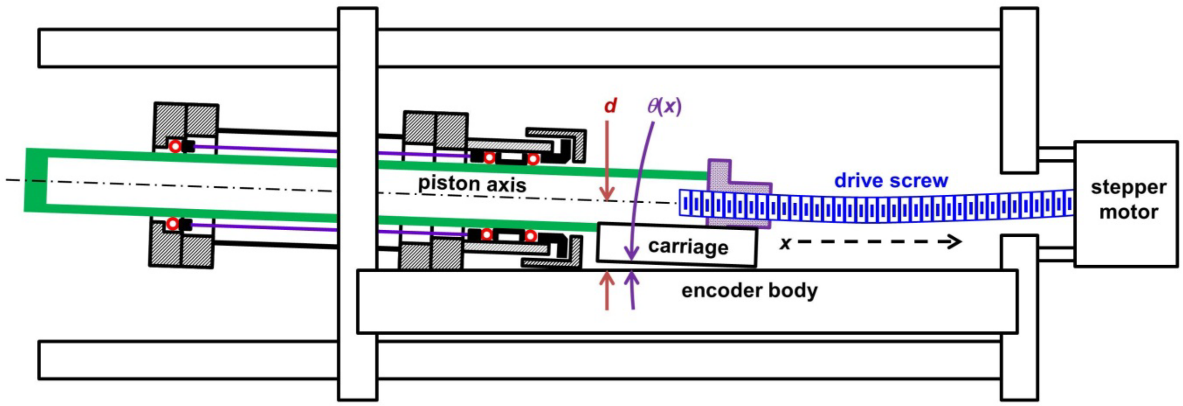

Figure 10. Abbe error is due to the separation *d* between the piston and encoder axes and the change of the angle $\theta(x)$ between the piston and the encoder that occurs with the displacement *x* of the piston stroke. No Abbe error occurs if $\theta(x)$ does not depend on *x*.

Table 4. Type B uncertainty contributions (those independent of the number of flow measurements) for a cylinder pressure near atmospheric pressure and a flow rate of 1 sccm. The last three components, due to leaks and outgassing, are inversely proportional to the flow rate. The last column is the relative standard (k = 1) uncertainty *u*.

component	value		$10^5 u$
PRT accuracy	0.01	K	3
temperature stability	0.002	K	1
temperature uniformity	0.02	K	7
pressure accuracy	10	Pa	3
piston diameter $u(D)$	0.0022	mm	18
encoder precision	0.0005	mm	0
encoder accuracy	0.003	mm	2
cosine error	0.004	rad	1
Abbe error	0.0006	rad	14
clock accuracy	10	s/d	12

clock resolution	0.001	s	1
leak to atmosphere	4×10^{-5}	sccm	4
leak across o-ring #1	1×10^{-5}	sccm	1
outgassing	6×10^{-5}	sccm	6
Sum in quadrature			**28**

The connection between the DUT and the flow standard will cause an error if its temperature T_C drifts significantly during the flow measurement, thereby causing the connection to act as a virtual source or sink of flow. This error can be reduced by surrounding the DUT with thermal insulation and using a small capillary to connect it to the flow standard. For a room temperature drift rate of dT_C/dt = 0.3 K/h and a 2 m capillary with an inner diameter of 1.3 mm, the resulting error will be

$$\delta \dot{n}_C = \frac{PV_C}{RT_C}\frac{1}{T_C}\frac{dT_C}{dt} \simeq 3\times10^{-5}\ \text{sccm}\,, \tag{10}$$

where V_C and T_C denote the volume and temperature of the connection. For a flow of 1 sccm, the corresponding value of u_C is negligible in comparison to the Type B uncertainty estimated in Table 4. The value of u_C can be reduced by another factor of 100 by placing the DUT inside the thermal enclosure.

8 Performance

The maximum flow rate at P = 100 kPa is 107 sccm, which corresponds to a motor pulse rate of 1000 Hz. The motor likely could be driven faster by using a power supply with a larger capacity, but the run time would then be less than 40 s, which is too short to allow settling of the PI loop. The minimum flow rate is limited by outgassing to approximately10^{-2} sccm.

The flow standard was built and tested at National Institute of Standards and Technology (NIST), taken apart, shipped to the US Army Primary Standards Laboratory (APSL), then rebuilt and tested. Figure 11 shows the results of tests, made at the two locations, in which the flow standard was compared with a capillary flow meter that had been calibrated by an independent primary flow meter [18]. The error bars show the standard uncertainty calculated by Eq. (8); the large uncertainties seen below 1 sccm are due to the Type A uncertainty of the DUT, specifically the uncertainty of the pressure tare.

Note the relatively large deviation of the point at 0.57 sccm. This occurred because the temperature of the DUT and its connecting tubing decreased at the unusually large rate of 0.4 K during the 18 min measurement, which caused the associated volume of roughly 10 cm^3 to act as a sink for gas flow. The deviation could have been reduced by minimizing the volume of the connection to the DUT and by either controlling the laboratory temperature better or placing the DUT inside the thermal enclosure.

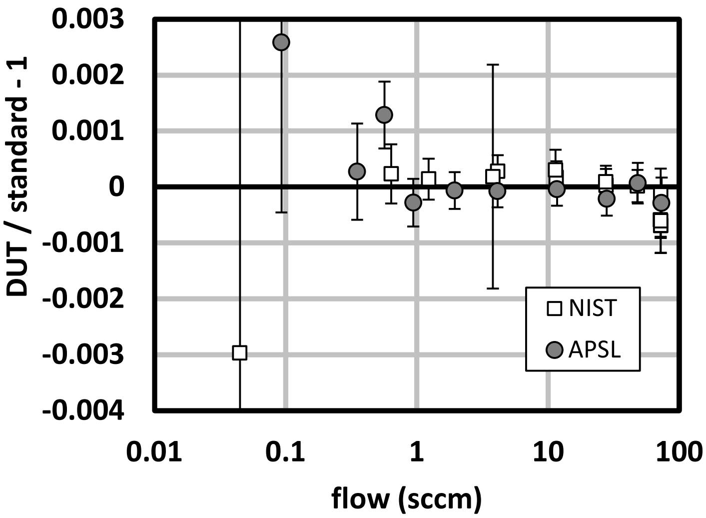

Figure 11. Results of tests made at NIST and APSL, in which the flow standard was compared with a capillary flow meter that had been calibrated by an independent flow meter. The error bars show the standard uncertainty calculated by Eq. (8); the large uncertainties seen below 1 sccm are due to the Type A uncertainty of the DUT, specifically the uncertainty of the pressure tare.

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23. Certain commercial equipment or materials are identified in order to specify the measurement procedure adequately. Such identification is not intended to imply recommendation or endorsement by the National Institute of Standards and Technology, nor is it intended to imply that the equipment or materials identified are necessarily the best available for the purpose.

11 Short biographies of the authors

Robert Berg is a physicist in the Sensor Science Division of NIST. His past work includes measurements of viscosity near critical points and standards for gas flow below 1 liter per minute. His present work supports measurements of the degradation of optical mirrors caused by photoresists intended for extreme ultraviolet lithography.

Tim Gooding worked in the Sensor Science division of NIST, where he designed, constructed, and programmed instruments that measure gas flow and pressure. He now is a museum specialist in the analytical laboratories of the Smithsonian National Museum of Natural History. He provides support for microscopy of geological samples and devises instruments for field work, such as a radio-synchronized camera array that was used to create a 3D model of a growing volcano dome.

Rob Vest came on staff at NIST in 1991 in the far and extreme ultraviolet detector calibration program, making calibration measurements and performing research in the development and characterization of detectors. In 1998 he assumed the leadership of the far and extreme ultraviolet detector program and of the near ultraviolet calibration program in 2013. He continues to perform research in EUV and UV radiometry techniques, technology, and applications and to provide calibration and metrology support to various user community. He has experience in control system design and programing for numerous calibration system and particle accelerator applications.