Ultra Low Flow Fast Primary Gas Prover

Measurement Science Conference 2009

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Abstract

Abstract - Traditionally, constant-pressure gas provers have been used in the range of 5 sccm to 50 slm. Our recent work with viscous-sealed provers has extended the range upward to 500 slm. However, there has been a growing demand for primary standards in the sub – 1 sccm range.

We have built prototype provers designed for flows of approximately 0.1 to 20 sccm. Using a quartz tube and piston of 2.5 mm diameter and 5 micron diametric difference, we are initially experimenting with a measured piston path of 5 cm. This will allow 1 sccm flows to be measured in approximately 21 seconds.

Our initial experiments show a standard deviation (repeatability) of about 0.02% and a piston leakage (tare) of about 0.0002 sccm. Using a target combined standard uncertainty of 0.25% or better, our pending uncertainty analysis will determine whether we can shorten the piston travel to affect faster readings.

We present data to date, along with preliminary production designs.

1. Introduction

Over the last seventeen years, we have introduced a variety of viscous-sealed piston provers. These provers differ from conventional designs in that the piston is sealed by the viscosity of the gas under test, eliminating other sealing methods such as mercury rings [1]. This allows the instrument to be small, automatic and portable, yet with accuracy suitable for standards. Within the conventional piston prover range of 5 sccm to 50 slm, we were able to produce 0.15% primary instruments (Figure 1, left), while ourselves being accredited by NVLAP to ISO17025 to better than 0.07%.

Figure 1 – Drycal Ml-800 (Attached To A Mass Flow Controller) and Definer 1020 (500 slm)

However, as is usual in the instrumentation field, we found demand for instruments that extended our range by at least an order of magnitude at each end. In response, we developed a 5 slm – 500 slm prover (Figure 1, right), which we have already reported upon [2]. This paper describes our efforts to go an order of magnitude (or more) lower than before. Our initial target flow range is 0.2 sccm to 20 sccm at ±0.25% combined standard uncertainty, with extended operation to 0.1 sccm at 0.5% uncertainty. Of course, we would be happy to exceed these minimum specifications.

2. Design Considerations

In this section, we discuss the considerations leading to a practical design.

2.1. Materials

We typically use borosilicate glass cylinders and pistons because they have excellent dimensional stability and wear characteristics combined with a very low coefficient of thermal expansion. We employed borosilicate, but we also experimented with a black quartz piston combined with a clear quartz cylinder to avoid the necessity for coating the piston in order to make it opaque to the optical detectors.

2.2. Piston-Cylinder Diametric and Gap

The viscous-sealed prover necessarily has a gap between the piston and cylinder, and this is the primary design constraint. The gas that leaks past the cylinder forms a constant flow [3] that runs counter to the flow of the gas being measured. The prover's internal computer adds the leakage (tare) into the reading to correct for it, but the deviation of the mean (measured) leakage tare determines the lowest flow that can be measured with a given uncertainty.

2.2.1. Piston Tipping Considerations

Using (as we do) a simple optical means of sensing the piston's edge, there is an uncertainty associated with the geometric freedom of the piston to tip upward or downward at the edge being detected. This creates an uncertainty at all flow rates. However, piston tipping causes only negligible uncertainty in this design due to the small ratios of piston diameter and gap to piston height and detection path length.

2.2.2. Leakage Considerations

To maintain accuracy at low flows, we must tare out the leakage between the piston and cylinder. Thus, the uncertainty at the lower limit of flow is primarily limited by the standard deviation of the leakage tare.

Assuming that the uncertainty of the leakage scales with the leakage, we would like our leakage value for the 0.2 to 20 sccm prover to be no higher than the leakage for a 500 sccm prover divided by 25. This represents a typical leakage of 0.004 sccm.

In scaling the piston-cylinder design, we must consider that leakage does not scale in a linear manner. If the leakage between the piston and cylinder (typically 7 to 11 microns apart) were laminar, conventional (Hagen-Poiseuille) calculations would result in leakage increasing as the third power of the gap. Our own observations of the pistons in current use showed an approximate 2.4 power relationship, possibly because the flow is somewhat turbulent due to the (necessary) roughness of the piston's surface.

The limits of available fabrication methods allow a piston-cylinder gap of approximately 5 microns to be achieved in practice. Based upon our prior designs' results and the scaling considerations discussed above, it appeared that the required leakage could be achieved.

2.3. Optical Path Length and Measurement Distance

The earlier Bios provers used LED emitters in combination with photodiodes collimated by a fine slit. Since the gap between the emitters and detects is much smaller than previous designs, the higher light level allowed the beam to be collimated with 2 slots, one on either side of the cylinder, instead of one. In addition, the slit width was reduced to 0.0008 cm.

Calculations based upon these dimensions indicate that a measured path length above approximately 2 cm would provide low enough uncertainty of the detected path length. However, repeatability and calibration considerations led us to use 5 cm as a nominal path length pending empirical measurement of repeatability.

2.4. Acceleration Distance

The piston must be allowed to accelerate to the speed of the flow stream before measurement is begun. We arbitrarily chose an acceleration distance to the measurement initiation sensor of 2.5 cm. Later observation of the device's internal pressure at its highest flows, along with other linearity testing, would empirically yield the final acceleration distance.

2.5. Valve

At this design's low flows, the primary considerations in valve selection were insignificant leakage, low inventory volume and minimal heating of the flow stream.

2.6. Thermal

In order to create a maximally isothermal flow path, a fan forces air entering from the top of the measuring cell over the electronics and valve solenoid, exiting at the bottom. The electronics are located far from the gas flow path and during measurements and the gas flow path bypasses the valve solenoid.

The reasoning behind the use of a downdraft is that heat generated by the bypass solenoid can be directed away from the flow path. However, if room temperature changes, the measuring tube will be at a different temperature that the gas entering from the base, which has a much longer time constant than the tube.

We may find, again empirically, that reversing the fan provides better results. If the fan volume is high enough, solenoid heat can be removed effectively enough to reduce its significance. The inlet air would enter through the base vents, essentially holding the tube at the base temperature.

2.7. Calibration

Any instrument must have calibration methodology at the core of the design process. This device creates interesting tradeoffs in calibration methodology. Calibration of a primary flow device is done by one of two methods, dimensional or gravimetric. Each method results in different specifications for the end product.

2.7.1. Direct Dimensional Calibration

The difference in diameter between the piston and the cylinder creates a direct dimensional uncertainty. Intuitively, the effective diameter would be an average of the two dimensions. However, slippage of the gas from either surface can cause the effective diameter to vary. Therefore, we include in our analysis an uncertainty resulting from the diametric difference between the piston and cylinder with a sensitivity of 2 (since the diameter is squared to calculate volume) and consider it a U-shaped distribution, dividing by the square root of 4.5 to obtain its estimated uncertainty.

In this case, using a diameter of .25 cm and a diametric difference of 5 microns, we obtain an uncertainty due to diametric difference of 0.089%, implying a standard uncertainty contribution (at k=2) of 0.18%. Adding other calibration uncertainties, we would be limited to a 0.2% to 0.25% overall combined standard uncertainty using dimensional calibration..

2.7.2. Gravimetric Calibration

A gravimetric calibration procedure would consist of measuring the weight depletion of a canister of gas. As an example, one readily available canister, commonly used for propane, weighs 450 grams and contains 20 grams of N_2 at 1.4 mPag (200 psig). With a scale resolution of 10 mg and using a doublesubstitution methodology, we could calibrate to an uncertainty of 0.06%. Using a more expensive scale (such as 1 mg resolution) or more expensive gas (such as xenon) would allow higher calibration accuracy.

Because of the low flow rate, the calibration time will be long. Approximately 16 liters of nitrogen and a calibration flow rate of 6 sccm would result in a two day calibration time. This would then be doubled because a leak test of similar duration would be needed before calibration.

Calibration time at the same uncertainty could be reduced by using the more accurate scale or denser gas. Hence, gravimetric calibration appears practical for achieving instrument accuracy that is not limited by its calibration methodology.

2.7.3. Uncertainties Common to Both Methodologies

The other uncertainties for this prover are similar to those of our other provers. As our analyses show 0.07% or better for the other provers, we expect this design to be limited in its accuracy by:

- Calibration methodology, overall
- Leakage repeatability at the lower limit
- Acceleration distance and linearity at the upper limit

3. Experimental Prototypes

Because so much remained to be determined empirically, there was little point to further analysis until the design's feasibility was established. Pistons, nominally 2.5 mm diameter by 4 mm high, were fabricated with an actual diameter approximately 5 microns smaller than that of their corresponding cylinders. One set was fabricated from borosilicate glass, while the other used a black quartz piston and clear quartz cylinder.

The fabrication time for the pistons and cylinders was approximately six weeks. Since the program could not proceed until experimental feasibility was established, the intervening time was used to create 3-D production designs in Solidworks.

Modular measuring cells similar to those used in our ML-800 product line were designed. Our intention was for these cells to be interoperable with the other cells on the same base assembly.

Figure 2 is a rendering of the flow cell's internal construction. Gas enters the lower fitting and passes through the rear-mounted bypass valve through the passage at the cell's rear and exits through the upper fitting. When a reading is called for, the bypass valve closes, forcing the gas through the piston-cylinder assembly.

Printed circuit boards on either side of the measuring tube contain light emitters and detectors for piston position. The lowermost sensor set is simply used to detect proper reset of the piston between readings. The two upper sets of sensors and collimators are used for the actual timing of the piston's movement.

The piston accelerates until it is at the gas flow rate. Then, it is timed from its passage of the center sensor until it reaches the upper sensor. The time interval is used to calculate the flow rate. When the piston is detected by the upper sensor, the bypass valve is opened, allowing the piston to fall until detected by the lowermost (reset) sensor.

Figure 3 shows front and rear quarter views of the complete cell assembly, detailing the fan, enclosure and venting system.

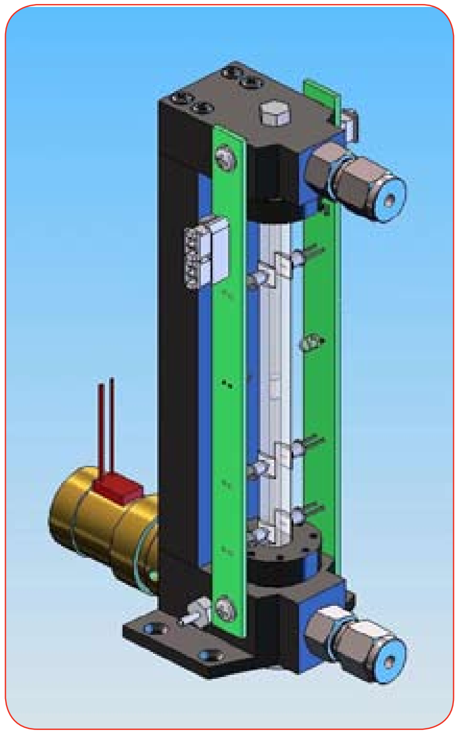

Figure 2 - Internal Cell Construction, Showing Tube and Optical Detectors

Figure 3 - Front and Rear Quarter Views of Measuring Cell

4. Feasibility Studies

Experimental prototypes were fabricated using both borosilicate and quartz. We desired to assess:

- Materials selection
- Piston leak test methodology (for setting tare)
- Leakage and its standard deviation (determines lower flow accuracy limit)
- Repeatability (determines individual-reading accuracy)
- Linearity (determines upper flow limit)

Much of this work is not rigorous: It is meant to prove the feasibility of a design that will fulfill our desired goals, with rigorous uncertainty analyses and additional comparisons to be performed later upon production prototypes.

4.1. Materials Selection

Our material selection made itself obvious. Using the quartz assembly, as we took repeated readings at a relatively high flow (~15 sccm); we noticed a steadily increasing but trendless scatter of our data points. In 70 minutes, our standard deviation had increased to almost 2% (Figure 4).

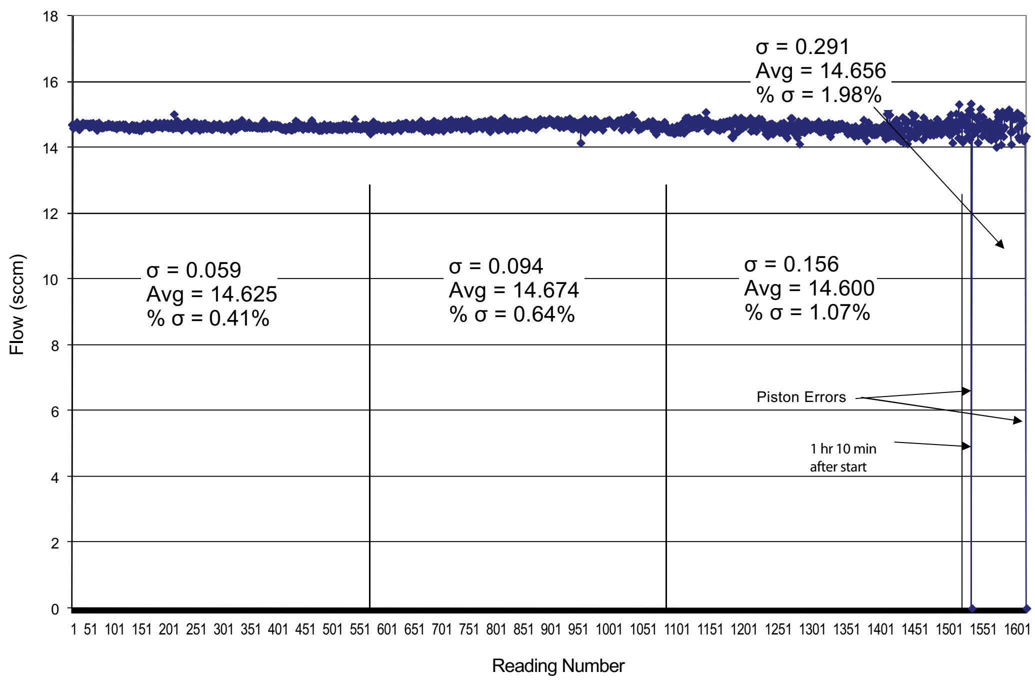

Figure 4 – Quartz Tube Assembly Readings at ~15 sccm

While we would like to claim that we immediately recognized the problem, such was not the case. With the high skirt area to weight ratio of this piston and the close tolerances (5 micron gap), sticking of the piston was an obvious suspicion. However, it took some time to discover that static charge was building on the cylinder walls. Forcing humid air into the cylinder remedied the problem, only to have it recur with repeated dry-gas usage. Apparently, the dissimilarity between the black quartz piston and the clear glass cylinder was causing buildup of static charge.

Since no such effect was observed with the all-borosilicate glass configuration, our materials choice became obvious.

4.2. Piston Leak Test Methodology

We use two methods to determine the tare value of piston leakage. The summation method is readily automatable, using valves, while the inversion method is not. However, the summation method can be affected by valve leakage, while the inversion method rejects any non-piston leakage because of its intrinsically differential approach.

4.2.1. Summation Method

The summation method determines the leakage by measuring the flow from source A, then Source B, and finally the combined flow is measured. Subtracting Flow A and Flow B from the combined flow yields the piston tare value. Below, A, B, and A+B are the total flows from the source. The reading taken is the actual flow rate minus the leakage.

$$[(A+B) - \lambda] - (A - \lambda) - (B - \lambda) = \lambda$$

4.2.2. Inversion Method

The inversion method determines the leakage by taking one measurement in the normal manner and then a second measurement with the cell inverted. The normal measurement is the actual flow minus the leakage and the inverted flow is the actual flow plus the leakage.

$$M_I - M_N = (A + \lambda) - (A - \lambda) = 2\lambda$$

4.3. Leakage and Its Standard Deviation

Using the summation method for three runs of ten readings each, we obtained the results in Table 1. Leakage was 0.013 ccm and its standard deviation was approximately 0.001 ccm.

Leakage	Uncertainty
0.01317	0.00139
0.01293	0.00101
0.01338	0.00072

Table 1 - A, B, A+B Leak Test

Using the inversion method for two runs of ten readings each, we obtained the results in Table 2. While leakage was also 0.013 ccm, higher than our original goal, its standard deviation was less than 0.0005 ccm. The improved results may have been from elimination of valve artifacts and the differential nature of the method, but that is not relevant at this time. More importantly, we observe a consistent leakage tare value confirmed by both methods and with a standard deviation that we may be able to improve from 0.0005 ccm with further refinement of our testing.

Leakage	Uncertainty
0.01305	0.00023
0.01328	0.00046

Table 2 - Inversion Leak Test

Remembering that the deviation of a mean decreases by the square root of the number of readings in the mean, the worst of the two inversion runs, 0.00046 ccm, improves to 0.00015 ccm. Setting a leakage tare error of 0.2% at the lower flow limit implies a flow of 0.075 ccm at 0.2% tare-related uncertainty. This implies that a total instrument combined standard uncertainty (k=2) of 0.2% is feasible at 0.2 ccm, or 0.07% at 0.5 sccm.

4.4. Repeatability

A constant flow of approximately 0.21 sccm was generated using porous plugs and precision pressure regulators. Flow readings were recorded over a working day (Figure 5). As is typical at low flows, we can observe an initial period of about 15 minutes of declining readings as thermal equilibrium was reached. Over the day, flow showed a linear increase of about 0.25%. This is probably a result of thermal conditions in the laboratory affecting the flow generating system.

We fitted straight lines to 100-reading segments and found that the repeatability (as deviation from a straight line) was approximately 0.1%. This is consistent with the standard deviation we observed during the leakage tests and confirms the feasibility of a 0.2 sccm prover. It should be remembered that the repeatability could be improved by averaging multiple readings, as well.

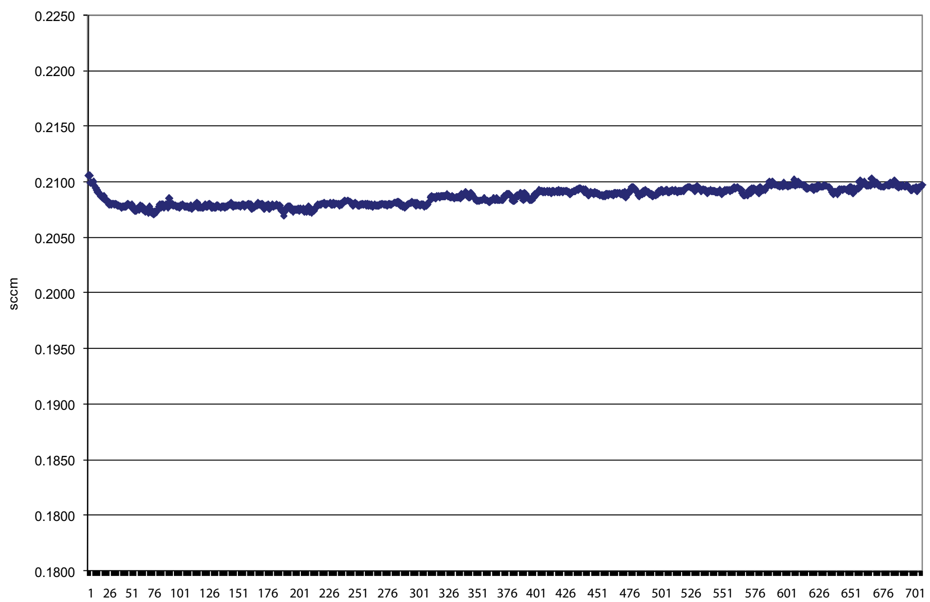

Figure 5 – Flow Readings Over an 8 Hour Day, ~0.21 sccm

4.5. Linearity

Ultimately, we will prove linearity by a binary, laddered test. Stable flows will be generated by two restrictors for each of a binary series of flows. The restrictors will be kept at constant temperature in a controlled bath if necessary. Then, we will measure each individual flow and the flow of the Y-connected pair. We will attribute the difference between the sum of the two readings and the combined reading to nonlinearity. At these low flows, we anticipate no significant error from dynamic pressure effects. Nonlinearity will be cumulatively plotted against flow to determine the linear range before maximum accurate flow is achieved.

For feasibility purposes, though it is sufficient to observe the cell's internal pressure.

4.5.1. Pressure Observations

A typical recording of internal pressure vs. time is shown in Figure 6. As expected, it is very similar to those seen with our earlier provers. At the closing of the bypass valve, the piston begins accelerating. It overshoots with a declining oscillation typical of an underdamped mechanical oscillator. In fact, that is exactly what it is: The mass of the piston and the spring constant of the contained gas are damped by the viscous friction of the gas that forms the seal.

We note that, at our desired maximum flow of 20 sccm, the interior pressure has stabilized to the point of residual oscillations of about ±0.1 mbar (or 0.1%) when our timing period begins. As we measure pressure at the start and again at the finish of timing and apply a PVTt correction [3], we envision no problems in achieving flows to 20 sccm with this design.

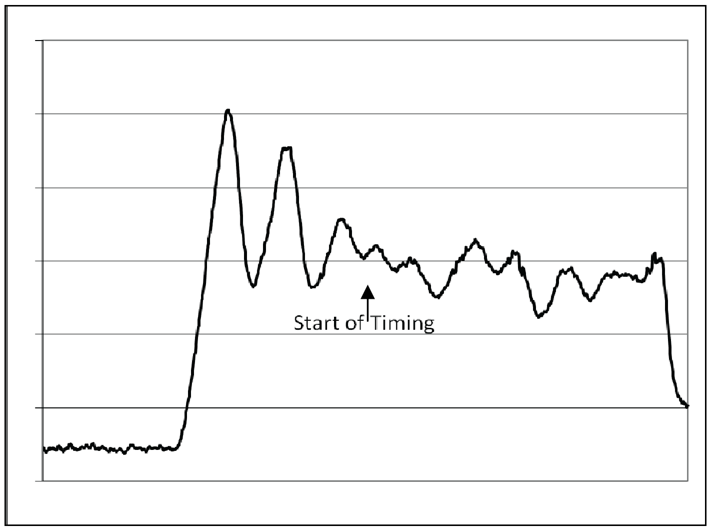

Figure 6 – Pressure Vs. Time at 20 sccm (Unscaled)

Moreover, the cylinder under test possessed imperfections resulting in visible sticking, or hesitation, during the acceleration interval. We expect that future production will be capable of higher flows, perhaps to 50 sccm.

5. Conclusions and Future Work

From this feasibility study, we conclude that we can build an instrument with a specification of ±0.25% at 0.2 to 20 sccm and 0.15% above 0.5 sccm for instruments that are calibrated gravimetrically. We also observe that flows to 50 sccm may be possible. Black quartz pistons have proven unfeasible for static electricity reasons, so we will concentrate on an all-borosilicate glass design.

Production can proceed, but before release as a commercial instrument, we must:

- Measure repeatability vs. timed path length to determine the shortest (therefore, the fastest) length
- Measure linearity by the binary summation method to determine the acceleration distance required for flows of 20 sccm and for flows to 50 sccm. This could be done by polishing out the surface imperfections in the acceleration region of the present prototype or by waiting for fabrication of a new prototype.
- Compare dimensional and gravimetric calibration results
- Perform a rigorous uncertainty analysis

6. References

[1] Padden et al., Flow calibrator, United States Patent 5,440,925

[2] Padden, Development of a 500 slm High Speed Piston Prover, 2008, Anaheim, California

[3] Padden, Comparisons of Viscous-Sealed Provers with LNE and Studies of Piston-Cylinder Leakage, Sixth International Symposium on Fluid Flow Measurement, 2006, Queretaro, Mexico

[4] Padden, Dynamic error correcting positive displacement piston flow meter and method of measuring gas flow in a piston flow meter, United States Patent 6,763,731

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