Extending the Range of Gas Piston Provers

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Abstract **4**

1. Introduction **4**

2. Design Considerations **5**

2.1. Leakage 5

2.2. Piston-Cylinder Diametric Uncertainty 6

2.3. Piston Rocking 6-7

2.4. Thermal 7

2.5. Dynamic Pressure Compensation (From PVTt) 7-10

2.5.1 Derivation of Algorithm 8-9

2.5.2 Validation of PVTt Approach 9-10

3. Calibration Methodologies **11**

3.1. Gas Gravimetric 11

3.2. Liquid Gravimetric 11

4. Uncertainty Analyses **12-15**

4.1. 0.5-50 sccm Uncertainty Analysis 12-13

4.2. 5-500 slm Uncertainty Analysis 14-15

5. Design Optimization **15-16**

6. Conclustions **17**

7. References **17**

Figure 1 - 5 to 500 slm Prover (Definer 1020) **5**

Figure 2 - 0.5 to 50 sccm Prover (ML 800-3) **5**

Figure 3 - Optical Geometry **6**

Figure 4 - Piston Rocking **6**

Figure 5 - Prover with Standardization **7**

Figure 6 – Intra-Cycle Pressure, 500 slm Prover **8**

Figure 7 - Deviation From Standards, Pressure Corrected, RMS Deviation = 0.039% **10**

Figure 8 - Deviation From Standards, No Pressure Correction. RMS Deviation = 0.074% **10**

Figure 9 - Water-Gravimetric Calibration **11**

Figure 10 - Low Flow Prover Net Uncertainty 13

Figure 11 - High Flow Prover Net Uncertainty 15

Figure 12 - High-Flow Prover, Piston Rocking Eliminated 16

Figure 13 - High-Flow Prover, Piston Rocking Eliminated, Pressure Transducer Calibrated 0.02% 16

Figure 14 - High-Flow Prover, Piston Rocking And Diametric Uncertainty Eliminated, Pressure Transducer Calibrated 0.02% 17

Table 1 - Low Flow Prover Flow-Independent Uncertainties 12

Table 2 - Low Flow Prover Flow-Dependent Uncertainties 13

Table 3 - High Flow Prover Flow-Independent Uncertainties 14

Table 4 - High Flow Prover Flow-Dependent Uncertainties 14

Abstract

Traditionally, constant-pressure gas provers have been used in the range of 5 sccm to 50 slm. Our most recent viscous-sealed provers have extended the range upward to 500 slm and downward to 0.5 sccm.

Over the traditional range of 5 sccm to 50 slm, we have published uncertainty analyses yielding combined standard uncertainties of approximately 0.06% and have rated our commercial products at 0.15%. However, the new provers have greater uncertainties when calibrated by dimensional means. The 500 slm prover is subject to significant uncertainty from piston rocking, as the piston width of 152 mm and the piston-cylinder gap of approximately 0.1 mm create a significant uncertainty with respect to the measured path length of 200 mm. Both provers are also subject to significant uncertainty caused by the difference of piston and cylinder diameters being significant with respect to the absolute diameter. For these reasons, we specified a conservative 0.25% combined standard uncertainty for the highest and lowest range provers. In both cases, however, the uncertainty can be improved by using gravimetric gas calibration instead of direct dimensional or, in the case of the larger prover, water-gravimetric calibration.

Here, we present our uncertainty analyses for the extended-range provers using different calibration methodologies and we discuss the cost-uncertainty tradeoffs for each.

1. Introduction

Every technology possesses both fundamental and practical limitations. At the limits, all we can do is to trade one desirable parameter for another. Speed versus accuracy is a fundamental example. Any parameter versus cost is a practical example. So, the question arose: What are the practical limits of piston prover technology? Could previous limits be extended tenfold?

Traditionally, mercury-sealed provers operated in the range of approximately 5 sccm[1] to 50 slm at 0.15% accuracy[2]. Our goal was to extend the range one order of magnitude in each direction using our fast viscous-sealed prover technology. We sought at least 0.25% accuracy over a minimum dynamic range (turndown) of 100:1.

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 laboratory standards. Within the conventional piston prover range of 5 sccm to 50 slm, we were able to produce 0.15% primary instruments, while ourselves being accredited by NVLAP to ISO17025 to better than 0.07%.

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 10 and a 0.5 – 50 sccm prover (Figure 2). To-date progress was reported earlier [2,3]. Here, we present updated uncertainty analyses and a discussion of possible improvements.

We appear to have exceeded our original goals, as will be seen in the uncertainty analyses that follow. The low-flow production prover possesses a combined standard uncertainty close to 0.1% from 1 to 50 sccm, increasing to 0.15% at 0.5 sccm and to less than 0.5% at 0.1 sccm. The 5 – 500 slm prover has a combined standard uncertainty of approximately 0.17% at this time. While this meets our original goal, we would like to improve the uncertainty to a figure below 0.1%. We will explore this further, below.

[1] To be consistent with SI, we define 1 sccm = 10-6 m^3/min at 101.325 kPa and 0°C, and 1 slm as 1000 sccm

[2] We use the term "accuracy" to mean a combined standard uncertainty at 2X coverage factor.

Figure 1 – 5 to 500 slm Prover (Definer 1020)

Figure 2 – 0.5 to 50 sccm Prover (ML 800-3)

2. Design Considerations

We have previously published a comprehensive uncertainty analysis of this type of prover [4]. Therefore, here we will discuss only the primary metrological constraints upon our accuracy and their effect upon our design decisions.

2.1. Leakage

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 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.

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 for the low flow prover and 100 microns for the high flow prover 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, which was proven in later testing.

2.2. Piston-Cylinder Diametric Uncertainty

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, if we calibrate by measured volume, using either direct dimensions or water displacement gravimetric means, we must 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). We consider this a U-shaped distribution, dividing by the square root of 4.5 to obtain its estimated uncertainty.

In the case of the low-flow calibrator, 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. For this reason, we initially chose a gas-gravimetric calibration for the low flow cell.

We did not possess the requisite equipment or gas-venting means to use such a method for the high-flow system. Moreover, in the case of the high-flow instrument, there were more significant uncertainties arising from other sources.

2.3. Piston Rocking

Using (as we do) a simple optical means of sensing the piston's edge (Figure 3), 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.

The piston can rotate about a diametric line through its center until its diagonally opposed edges contact the cylinder walls, causing an uncertainty in the height of the measured edge with respect to the center of the piston (which determines actual volume).

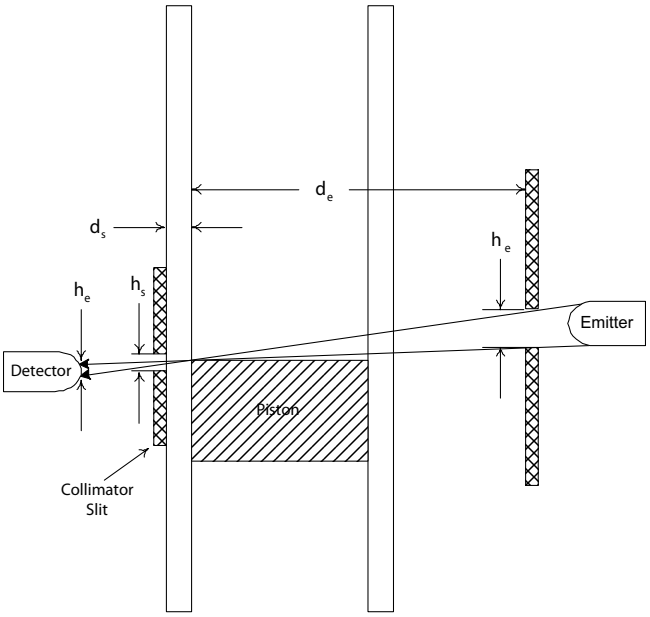

Figure 3 – Optical Geometry

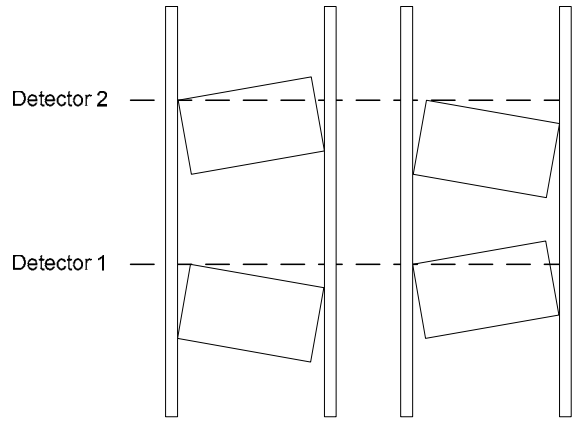

Figure 4 – Piston Rocking

Piston rocking caused only negligible uncertainty in our earlier designs. If we were willing to scale the height of the high flow prover to the flow, it might not present a problem in this design, either. However, our measuring cylinder would then be two meters in length and our measurement time several times as long as that of the smaller provers. In order to prevent our prover from being large and slow (and probably not possible to manufacture), we wished to reduce the measurement path length. However, piston rocking results in an error inversely proportional to the measured distance and thus becomes significant.

We used the cylinder diameter of 152 mm and assumed (for trial purposes) a timed path length of 140 mm. We performed straightforward geometric calculations to find the amount of vertical distance that the detected piston edge can move upward or downward from the height of the piston's center for a given piston-cylinder diametric difference, expressed as a ratio to the measured path length. For conservatism in assessing the resulting uncertainty, we then assumed a rectangular distribution, dividing by the square root of 3. We then multiplied the uncertainty by the square root of two, since the uncertainty affects both the upper and lower sensor positions. This became a major source of the prover's uncertainty.

2.4. Thermal

In order to create a maximally isothermal flow path, a fan forces air from the top of the measuring cell. The inlet air enters through the base vents, essentially holding the tube at the base temperature.

2.5. Dynamic Pressure Compensation (From PVTt)

Dynamic pressure measurement is extremely useful in achieving high speed and high accuracy in gas provers [5]. Since the pressure inside a prover is not exactly the same at the start and end of the timed interval (due to accelerative, oscillatory, turbulence and piston-jamming effects, see Figure 6), a simple calculation of volume divided by time does not yield the most accurate results. However, it is possible to accurately measure flow by measuring the internal pressure of the cylinder using a high-speed transducer and a PVTt methodology. Here is the derivation of the dynamic pressure equation:

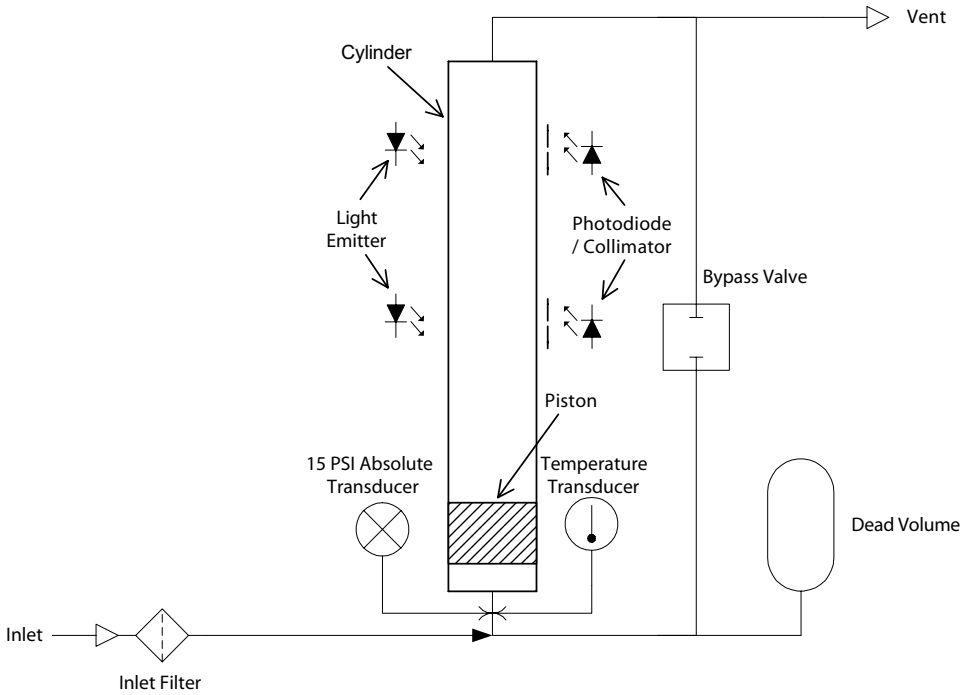

Figure 5 – Prover with Standardization

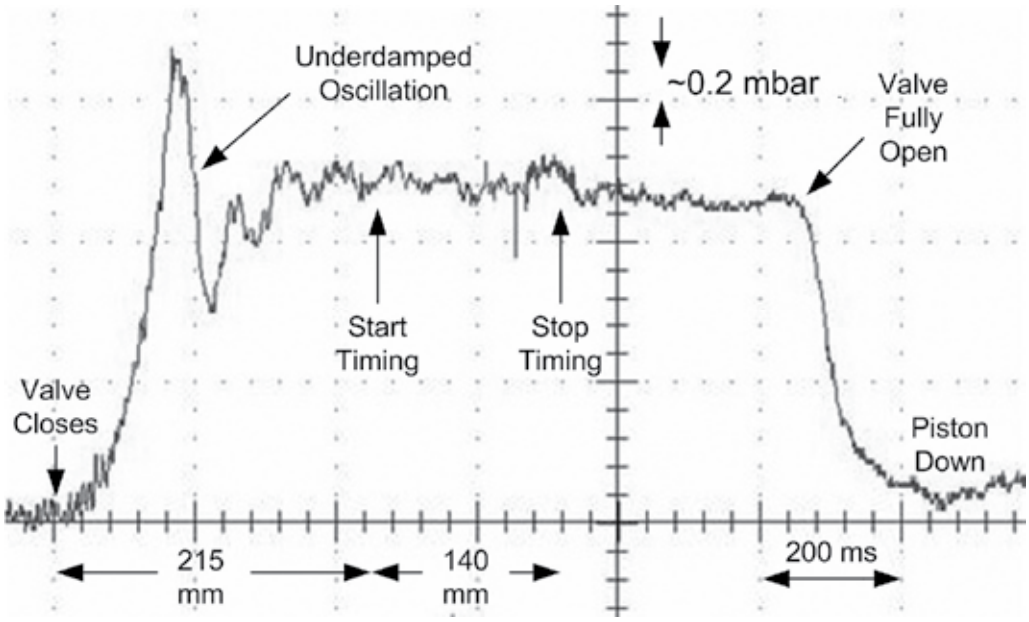

Figure 6 – Intra-Cycle Pressure, 500 slm Prover

In effect, any change of pressure that occurs between the start and end of timing must fill the measured volume and the inventory volume. Using the equations of state for the system at the start and end of timing, a simple equation can be derived to yield the correct flow [5].

2.5.1. Derivation of Algorithm

We begin with the state equations for the contained gas at the start (T_1) and end (T_2) of the timed interval:

$$P_1V_1 = n_1RZT_1 \; ; \; P_2V_2 = n_2RZT_2$$

We can restate these to yield the molar quantities (n_1 and n_2) at the start and end of the timed interval and as the change in molar volume (Δn):

$$\frac{P_1V_1}{RZT_1} = n_1 \; ; \; \frac{P_2V_2}{RZT_2} = n_2 \equiv n_2 + \Delta n$$

As previous experiments with a fast-response thermistor had shown that the temperature of the gas entering the measuring cylinder was within approximately 0.02 °C, we can replace T_1 and T_2 with a single temperature, T. Then, from the above:

$$\Delta n = \frac{P_2V_2 - P_1V_1}{RZT}$$

We can replace V_1 with the inventory volume V_{INV}, which is the total volume of the system at the start of timing, and V_2 with the sum of the inventory volume and the measured volume, V_M, such that:

$$V_1 = V_{INV} \; ; \; V_2 = V_{INV} + V_M$$

And:

$$\Delta n = \frac{(P_2 - P_1)V_{INV} + P_2 V_M}{RZT}$$

Dividing the above by the measurement interval, Δt, we can obtain the molar flow rate, $\dot{n}$:

$$\dot{n} = \frac{(P_2 - P_1)V_{INV} + P_2 V_M}{RZT\,\Delta t}$$

A naïve (traditional) expression of the molar flow would be:

$$\dot{n}_{naive} = \frac{P_2 V_M}{RZT\,\Delta t}$$

Now we can state that:

$$\frac{\dot{n}}{\dot{n}_{naive}} = \left[\frac{(P_2 - P_1)V_{INV} + P_2 V_M}{RZT\,\Delta t}\right] \Big/ \left(\frac{P_2 V_M}{RZT\,\Delta t}\right)$$

That simplifies to:

$$\dot{n} = \dot{n}_{naive} \bullet \left[1 + \frac{(P_2 - P_1)V_{INV} + P_2 V_M}{P_2 V_M}\right]$$

Or:

$$\dot{n} = \dot{n}_{naive} \bullet \left[1 + \frac{(P_2 - P_1)}{P_2} \bullet \frac{V_{INV}}{V_M}\right]$$

We can then infer the flow at the cylinder inlet (or outlet) as:

$$\dot{v} = \dot{v}_{naive} \bullet \left[1 + \frac{(P_2 - P_1)}{P_2} \bullet \frac{V_{INV}}{V_M}\right]$$

This can be thought of as correcting the traditional flow measurement by the relative difference in pressure between the end and the beginning of the measurement, multiplied by a "pneumatic lever" consisting of the inventory volume (which must be filled during the pressure change) divided by the measured volume.

2.5.2. Validation of PVTt Approach

Using sonic nozzles at NIST, we produced flows of 10, 25, 50, 90, 180, 240 and 500 slm and recorded approximately 10 readings at each flow while simultaneously integrating the flow data from NIST's nozzles. For each prover reading, we recorded the volumetric flow, the temperature and the instantaneous absolute pressure inside the cylinder at the start and end of timing.

In analyzing the data, we applied the PVTt equation described above. The internal volume of the instrument below the lower detector (obtained from its 3-D CAD drawings) was 5150 cc. Fittings and approximately 7.5 meters of 10 mm diameter copper tubing (used for temperature stabilization) were approximately 570 cc

in volume. The measured volume was 2548 cc. This resulted in an inventory to measured volume ratio of 2.24. The pressure equation was applied to the data using this actual volume ratio.

We then evaluated the linearity of our results with respect to the NIST nozzles by performing a leastsquares linear regression. The worst-case nonlinearity was 0.065% and RMS nonlinearity was reduced to 0.039% (Figure 7).

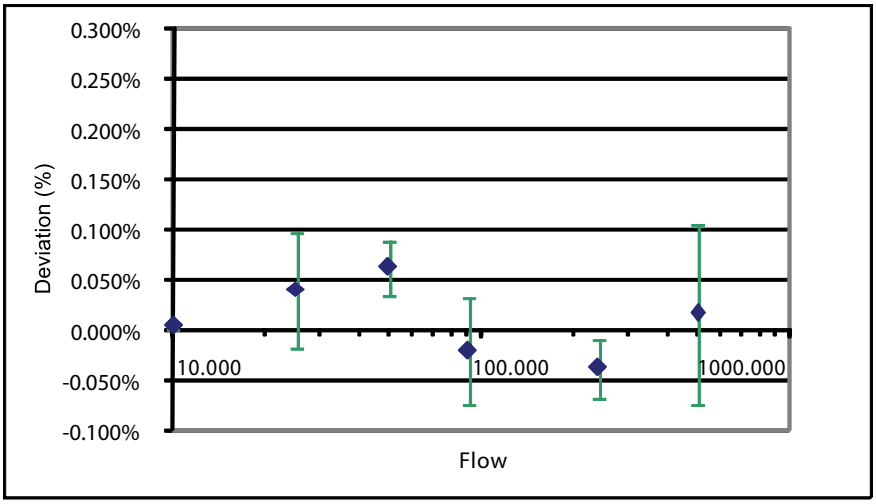

Figure 7 - Deviation From Standards, Pressure Corrected, RMS Deviation = 0.039%

To test the effectiveness of this approach, we then set the inventory volume to 0 in the above equation, yielding the traditional, simply-calculated flow. Our best fit showed a nonlinearity of 0.22% where expected, at the highest flow (Figure 8). Overall, the RMS (typical) non-correlation was 0.074%. This represented a threefold increase in worst-case non-linearity, proving the value of the PVTt approach.

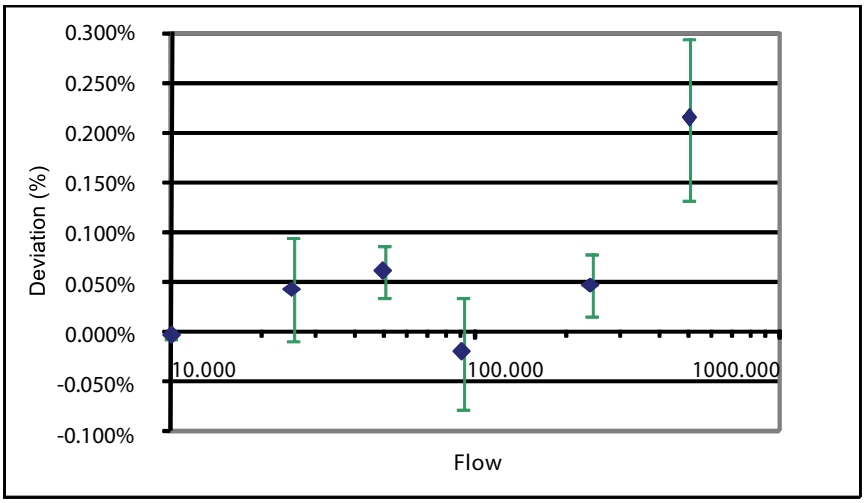

Figure 8 – Deviation From Standards, No Pressure Correction. RMS Deviation = 0.074%

3. Calibration Methodologies

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 product. For the low-flow prover, we chose gas-gravimetric calibration and for the high-flow prover, we used a liquid-gravimetric volume measurement.

3.1. Gas Gravimetric

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 double-substitution methodology, we can calibrate to an uncertainty of 0.07%. 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.

3.2. Liquid Gravimetric

Because of the large internal volume of the 500 SLM prover, gravimetric calibration using the mass of the volume of deionized water needed to fill the cylinder through the measurement interval provides more accurate calibration than the dimensional measurements used in the smaller Bios International prover.

The cylinder is fitted with a special piston that will float as water enters the bottom of the prover. The electronics detect when the piston reaches the bottom of the measurement interval and when the piston reaches the top of the measurement interval as the cylinder is filled. Knowing the water mass and temperature at these two points allows accurate determination of the measurement volume.

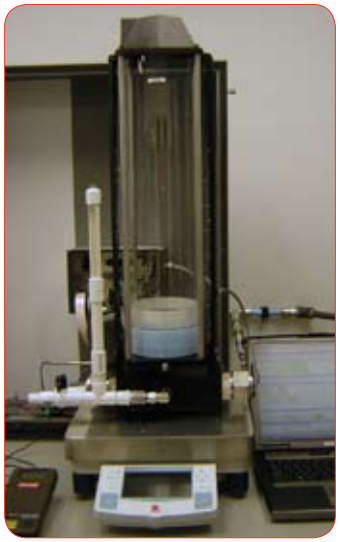

Figure 9 – Water-Gravimetric Calibration

4. Uncertainty Analyses

For these provers, we find it convenient to separate the analyses into the flow-independent and flowdependent constituents, later combining them to obtain the net accuracy versus flow rate. These analyses are for production instruments, and do not represent the limits of Bios' laboratory instruments.

4.1. 0.5-50 Sccm Uncertainty Analysis

The low-flow cell presented an adequate uncertainty, near ±0.1% over most of its range. Note that the uncertainties arising from gravimetric calibration and from pressure measurement are similar.

Flow-Independent Uncertainties	u (ppm)	Type	Sensitivity	Total (ppm)	% of RSS	Net (ppm)
Gravimetric Calibration Uncertainty						
Resolution (Start)	115	B	1	115	10%	
Repeatability	58	B	1	58	2%	
Resolution (Stop)	200	B	1	200	30%	
Start Timing Error	167	B	1	167	21%	
Stop Timing Error	167	B	1	167	21%	
Calibration Weight	24	B	1	24	0%	
Gas Density	50	B	1	50	2%	
Gas Purity	50	B	1	50	2%	
Timing Error	50	B	1	50	2%	**347**
Piston Optical Sensor Error:						
Upper Slit Width	72	B	1	72	4%	
Lower Slit Width	72	B	1	72	4%	
Piston Rocking	41	B	1	41	1%	**110**
Time Error:						
Time Base	29	B	1	29	1%	**29**
Pressure:						
Barometric Transducer	296	B	1	296	65%	
Differential Transducer	131	B	1	131	13%	**324**
Temperature:						
Reference Standard	59	B	1	59	3%	
Bath	10	B	1	10	0%	
Linearity of Sensor System	55	B	1	55	2%	
0.05c Difference, Sensor to Stream	98	B	1	98	7%	
Coefficient of Linear Expansion	12	B	3	36	1%	**132**
RSS (Net) u						**506**

Table 1 – Low Flow Prover Flow-Independent Uncertainties

	Flow-Dependent Uncertainties (ppm)			Combined Expanded Uncertainty		
Flow (sccm)	u (flow repeat.)	u (lkg effective)	Total F.D.	Combined u (ppm)	Expanded u (X2)	Average of 10
0.10	1200	2321	2613	2661	0.532%	0.481%
0.20	600	1160	1306	1401	0.280%	0.256%
0.28	500	829	968	1092	0.218%	0.197%
0.50	500	464	682	849	0.170%	0.141%
1.00	300	232	379	632	0.126%	0.113%
2.00	300	116	322	599	0.120%	0.105%
4.00	400	58	404	647	0.129%	0.105%
20.00	400	12	400	645	0.129%	0.104%
30.00	400	8	400	645	0.129%	0.104%
40.00	400	6	400	645	0.129%	0.104%
50.00	500	5	500	711	0.142%	0.106%

Table 2 – Low Flow Prover Flow-Dependent Uncertainties

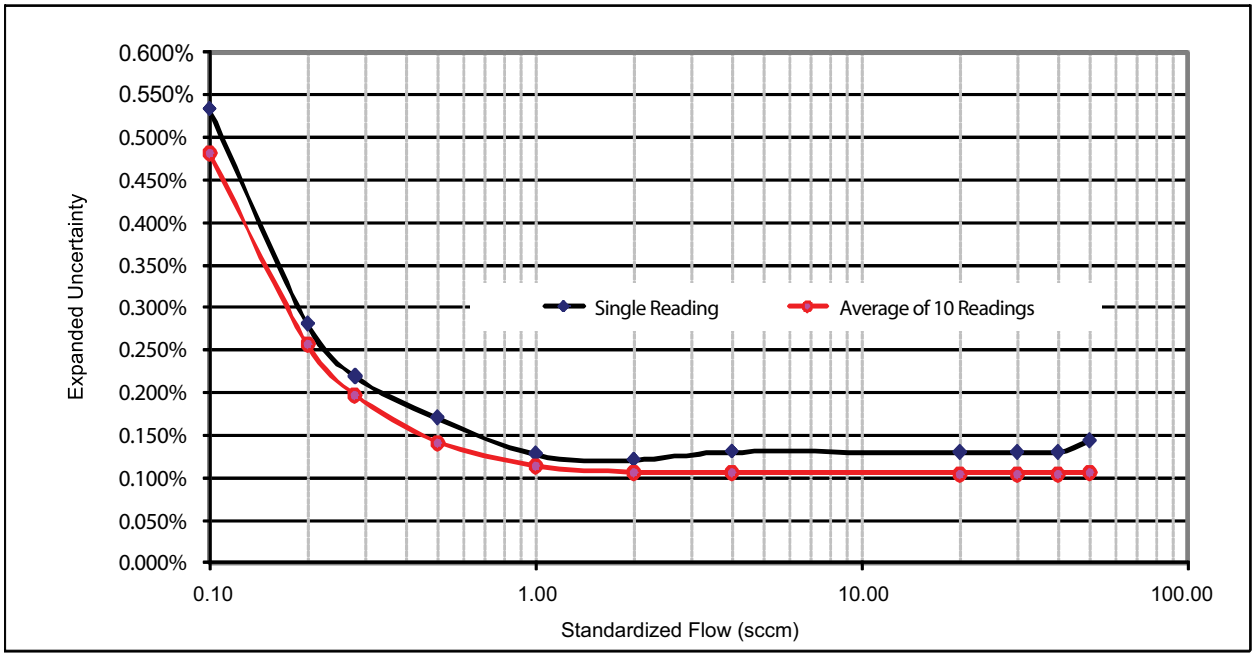

Figure 10 – Low Flow Prover Net Uncertainty

4.2. 5-500 slm Uncertainty Analysis

The high-flow prover met our goals, but did not possess our typical 0.1% accuracy, exhibiting close to ±0.17% over most of its range.

Flow-Independent Uncertainties	u (ppm)	Type	Sensitivity	u Total (ppm)	% of u RSS	u Net (ppm)
Calibration:						
Temperature Related Density u	21	B	1	21	0%	
Scale Measurement u	160	B	1	160	4%	
Reference Weight Accuracy	25	B	1	25	0%	
Down Tube Error	4	B	1	4	0%	**163**
Distance:						
Upper Slit Width u	169	B	1	169	4%	
Lower Slit Width u	169	B	1	169	4%	
Diametric Uncertainty	157	B	2	314	15%	
Piston Rocking	536	B	1	536	45%	**666**
Time:						
Timing	29	B	1	29	0%	**29**
Pressure:						
Pressure Transducer (Typical)	403	B	1	403	24%	**103**
Temperature:						
Reference Standard	59	B	1	59	1%	
Temperature Bath	10	B	1	10	0%	
Linearity of Sensor System	50	B	1	50	0%	
0.1c Difference, Sensor to Stream	195	B	1	195	6%	
Coefficient of Linear Expansion	12	B	3	36	0%	**213**
RSS (Net) u						**824**

Table 3– High Flow Prover Flow-Independent Uncertainties

	Flow-Dependent Uncertainties (ppm)			Combined Expanded Uncertainty		
Flow (sccm)	**u (flow repeat.)**	**u (lkg effective)**	**Total F.D.**	**Combined u (ppm)**	**Expanded u (X2)**	**Average of 10**
6	485	362	605	1022	0.204%	0.169%
10	150	217	264	865	0.173%	0.166%
25	341	87	352	896	0.179%	0.166%
50	156	43	162	840	0.168%	0.165%
90	308	24	309	880	0.176%	0.166%
240	143	9	143	836	0.167%	0.165%
502	440	4	440	934	0.187%	0.167%

Table 4 – High Flow Prover Flow-Dependent Uncertainties

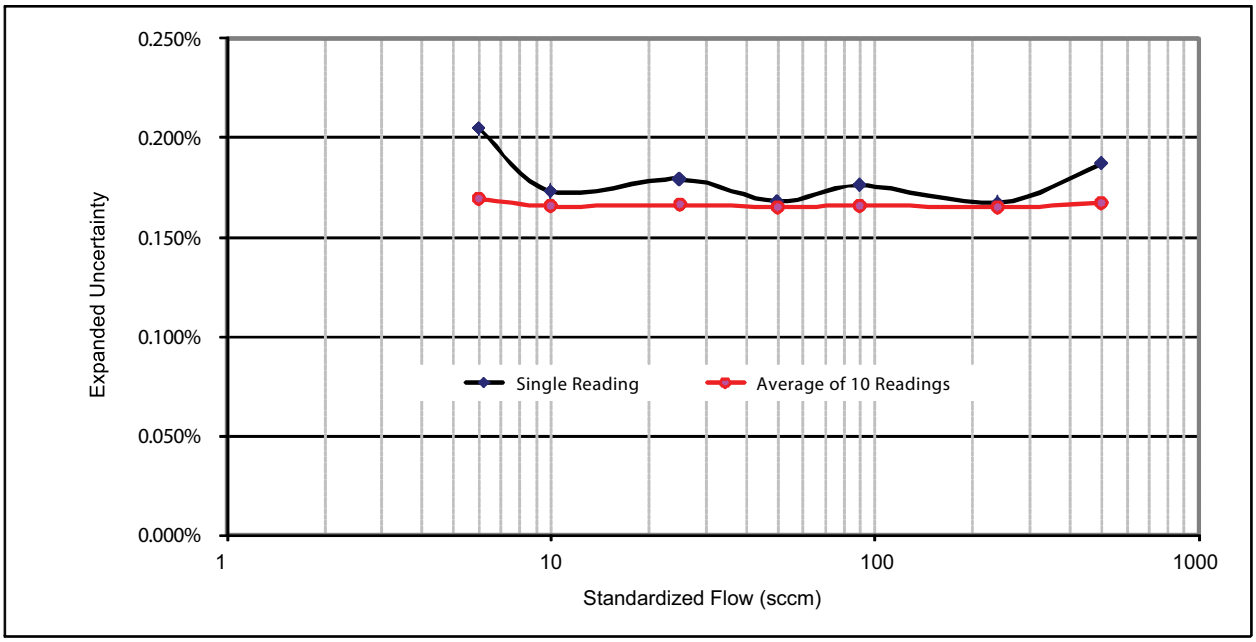

Figure 11 – High Flow Prover Net Uncertainty

5. Design Optimization

As we wish to improve the accuracy of the high-flow prover, we will examine its major sources of uncertainty. Referring again to Table 3, note the second-to-last column. Here, we have included each uncertainty's square as a percentage of the sum of all uncertainties' squares. This gives us a ready indication of the major contributors to our net uncertainty. We can immediately note that 45% of the net uncertainty is due to piston rocking. If we can eliminate piston rocking from being an uncertainty, we can improve accuracy to about ±0.13% (Figure 12). It is frustrating in that we do not even know if rocking is occurring, indeed, our earlier linearity comparisons would imply that it is not. However, until we can conclusively eliminate rocking as a factor, we must include it in our analysis.

There are a number of approaches to eliminating the rocking uncertainty, including:

- Proof of non-existence
- Opposed edge detectors, separately timed and averaged
- Optical detection of a target at the center of rotation
- Optical detection of a target at a fixed radius from the center of rotation

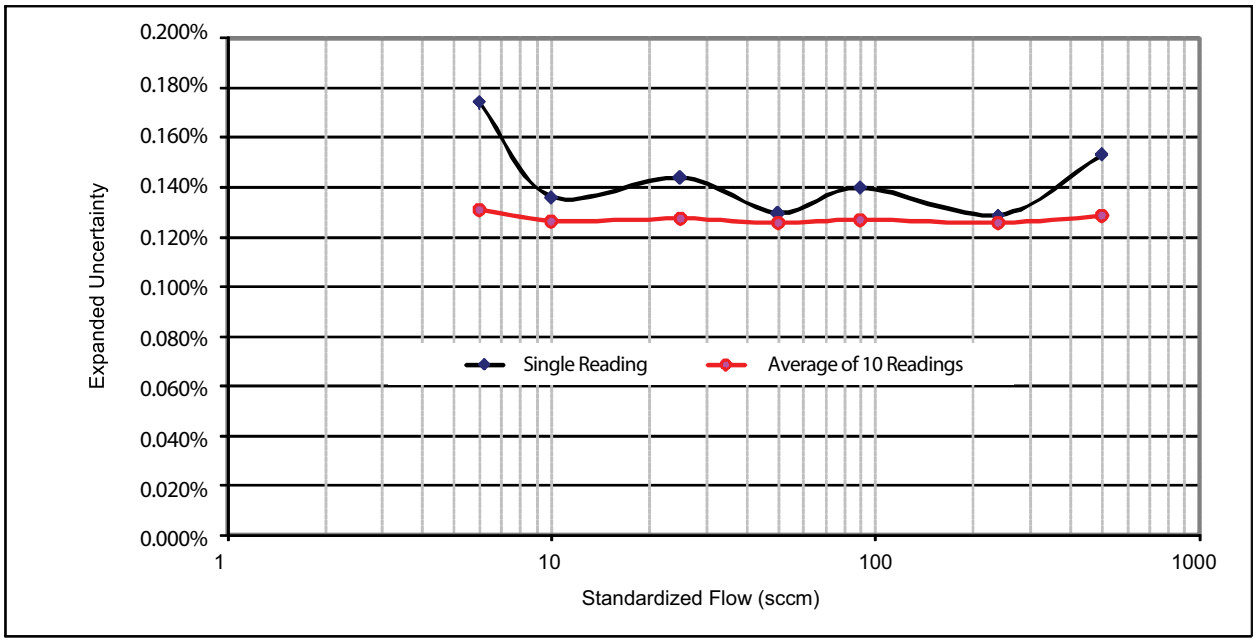

Figure 12 – High-Flow Prover, Piston Rocking Eliminated

At that point, the largest remaining uncertainty would be caused by the absolute pressure transducer. Improving the transducer to ±0.02% (or calibrating it to that level before measurements) would improve accuracy to ±0.1% (Figure 13).

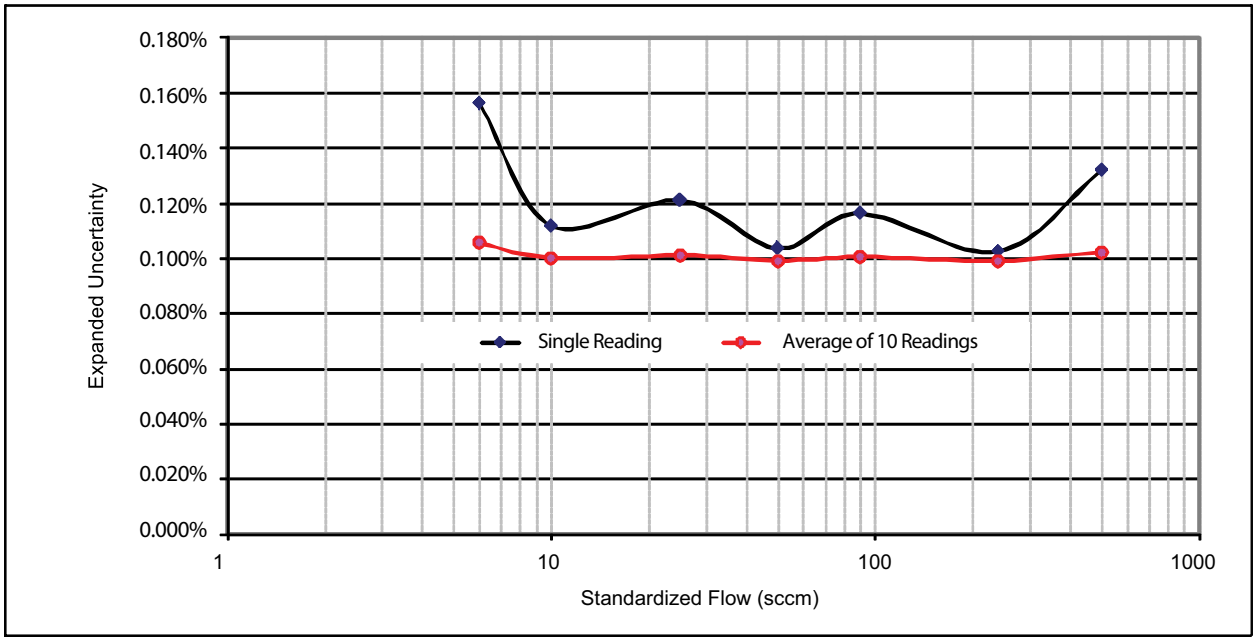

Figure 13 – High-Flow Prover, Piston Rocking Eliminated, Pressure Transducer Calibrated 0.02%

Continuing further, the largest remaining uncertainty is caused by diametric uncertainty due to the unknown effects of the piston-cylinder gap. If we performed a gas calibration, that could be eliminated, resulting in approximately ±0.08% accuracy (Figure 14)

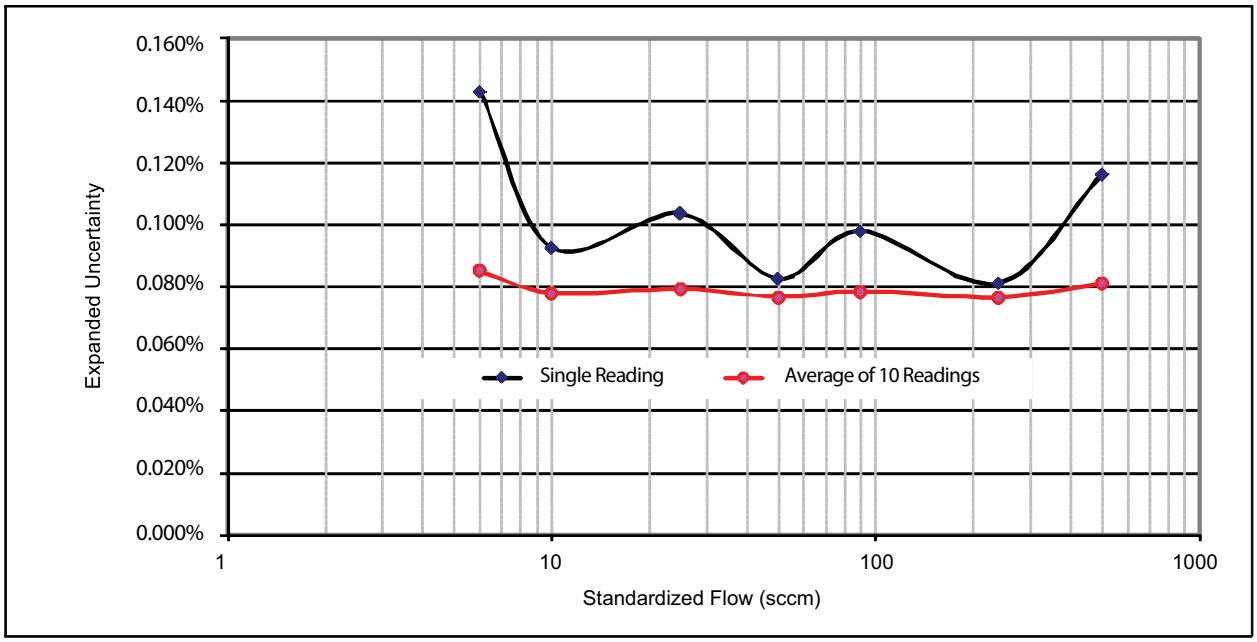

Figure 14 – High-Flow Prover, Piston Rocking And Diametric Uncertainty Eliminated, Pressure Transducer Calibrated 0.02%

6. Conclusions

It appears that we have reached our goal. We presently have production prover designs of

- 0.1% from 1 to 50 sccm, increasing to 0.15% at 0.5 sccm and to less than 0.5% at 0.1 sccm
- 0.17% from 5 – 500 slm with possibility of improvement to better than 0.1%

We have also empirically proven the value of the PVTt approach in piston prover design.

7. References

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

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

[3] Padden, Ultra Low Flow Fast Primary Gas Prover, Measurement Science Conference 2009, Anaheim, California

[4] Padden, Uncertainty Analysis of a High-Speed Dry Piston Flow Prover, Measurement Science Conference 2002

[5] 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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