Uncertainties and Inter-Laboratory Comparisons of Dry Piston Gas Flow Provers

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Abstract

Dry piston provers are similar in nature to conventional mercury-sealed flow provers, with the exception that the viscosity of the gas forms the seal across a very small (< 10 u) section. As a result, very small, portable provers can be produced. Calibrations can be performed with the accuracy, primacy and large dynamic range of the earlier mercury-sealed provers, but far more rapidly. The transportable nature of these primary instruments also makes them very useful for the inexpensive, ongoing harmonization of laboratories.

Our latest uncertainty analysis shows a combined expanded uncertainty of less than 0.08% over the range of 5 to 50,000 sccm. Although we made every effort to perform an accurate analysis, empirical verification of any uncertainty analysis is necessary. At this level, the only means of verification is through peer-to-peer inter-laboratory comparisons.

As a result, we have performed informal comparisons of a single pair of provers with a number of national and private laboratories on three continents. We compared for reproducibility with respect to different national laboratories and over time, with transportation and between overlapping cell ranges. In addition, and we compared newly manufactured provers with the original pair to establish reproducibility with manufacture. Typically, the provers exhibited discrepancies (within their specified range) of less than 0.1% in comparison with critical flow venturis at NIST and NMIJ, with the possible exception of the 50000 sccm point. At the highest flow, the original prover exhibited a discrepancy of 0.15% to 0.2%. A second prover later showed a discrepancy of 0.035%. We must conduct further investigations to determine the linearity of the design for flows above 30,000 sccm.

Here, we will present an introductory summary of the uncertainty analyses, details of the comparison methodology, data on potential experimental error sources (such as inventory volume), the comparative data, and the methods of data analyses used. We will also discuss our ongoing research at the lower limit of this design's useful range (approximately 1 sccm), where leakage is not necessarily constant and conventional viscosity may not apply.

Keywords: Gas flow calibration, primary prover, inter laboratory comparison

1.0 Introduction

Field-rugged primary gas flow measurement devices in the 1 sccm to 50,000 sccm flow range have long been needed for diverse applications, from calibration of environmental air samplers at 1% accuracy to the manufacture or field service of mass flow meters and flow controllers at 0.2% accuracy and for national-level calibration laboratories at 0.1%.

Historically, primary flow calibrators had the advantages of direct traceability and minimal drift mechanisms; however, they were large, delicate and slow to operate (a practical limit to dynamic range). More portable and user-friendly primary provers were highly desirable.

Viscous-sealed (dry) piston provers reduce the primacy/portability tradeoff. Using a virtually frictionless piston, such provers achieve uncertainties below 0.1% while remaining small, fast and portable.

2.0 Piston Prover Design

Constant-displacement systems are, perhaps, the simplest and most intuitive flow measurement devices. They have the extremely desirable characteristic of being characterized by the most basic of quantities: length and time. As flow is necessarily a derived unit, a dimensionally - characterized system would be as close as possible to direct traceability from national dimensional standards.

An idealized piston prover would consist of a massless, frictionless, leak proof, shape-invariant and impermeable piston inserted within the flow stream and enclosed by a perfect cylinder (Figure 1). The time that the piston takes to move a known distance (which implies a known volume) then yields the volumetric flow as:

$$F = V / T = \pi r^2 h / T$$

Such a device would be as accurate as its physical dimensions and its clock, with almost insignificant drift mechanisms. Of course, such idealized devices do not exist. Historically, three basic practical versions of piston provers have been employed.

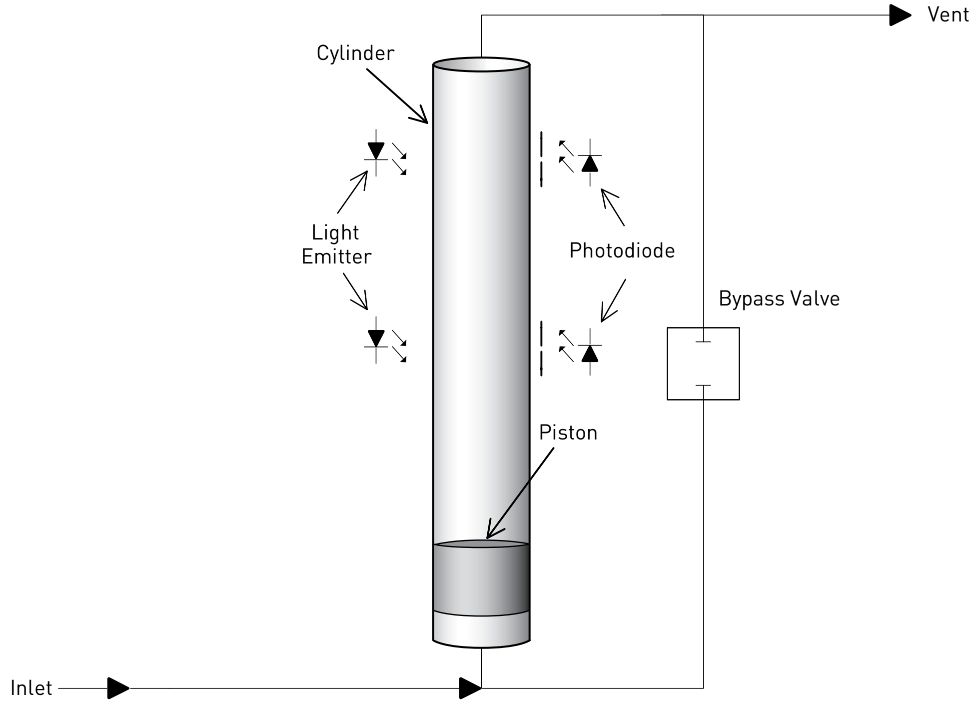

Figure 1 – Idealized Automatic Piston Prover

2.1 Viscous-Sealed Provers

The DryCal viscous-sealed prover uses a piston and cylinder fitted so closely that the viscosity of the gas under test results in a leakage small enough to be insignificant. For reasonable leakage rates, such a gap must be approximately 10 microns. As a practical matter, the piston and cylinder are made of graphite and borosilicate glass because of their low, matched temperature coefficients of expansion and low friction (Figure 2).

Figure 2 – Viscous-sealed piston and cylinder

Such a device comes close to the ideal. The piston is shape invariant, impermeable and virtually frictionless. There is no vapor pressure from a soap film or sealant. The instrument can utilize high piston speeds, resulting in a measurement repetition rate rapid enough to be considered quasi-continuous.

An uncertainty analysis for such an instrument has unique considerations. The static uncertainties can be evaluated in a manner similar to that used for conventional provers. Indeed, the design reduces piston rocking and temperature stratification errors. However, dynamic uncertainties resulting from significant underdamped piston mass, the effects of inventory volume and leakage must be assessed. Finally, as with all provers, standardization must be applied for most applications, with particular attention paid to dynamic accuracy of the standardizing parameters (Figure 3).

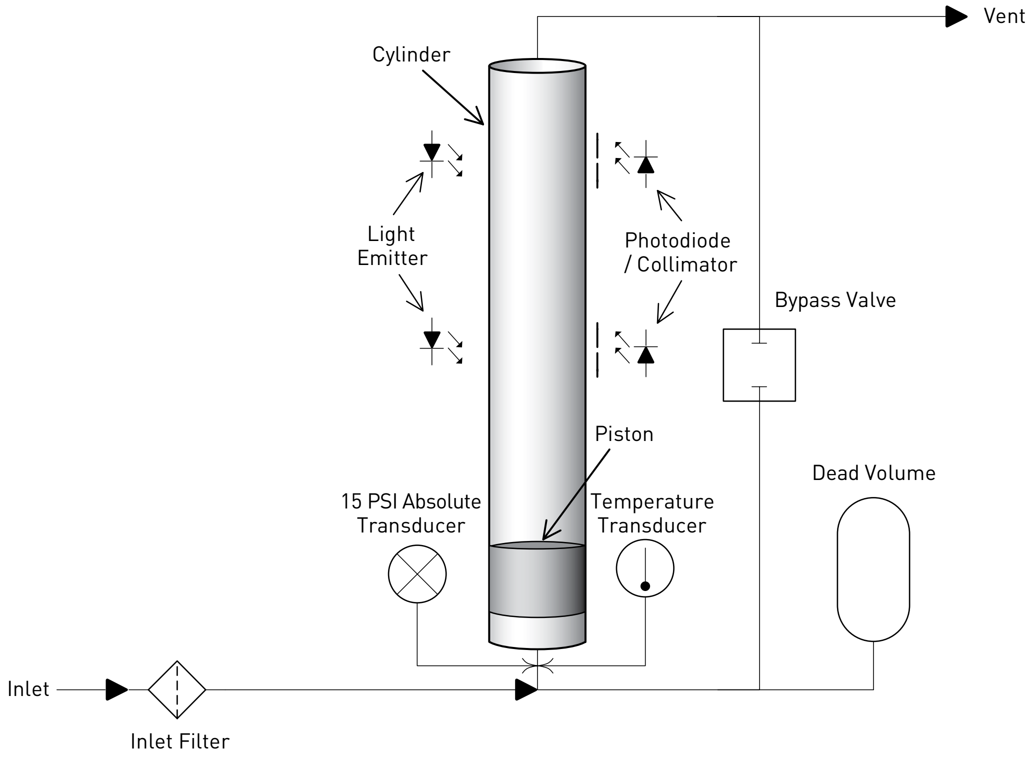

Figure 3 – Practical Piston Prover

2.2 Experimental Test Articles, DryCal ML-800

The DryCal ML-800 is a portable commercial prover of approximately 0.1% accuracy (Figure 4). The instrument consists of a base unit with three sizes of interchangeable flow cells Flow cells are available in Low (10), Medium (24) and High (44).

Each cell consists of a machined base containing the inlet and outlet fittings, bypass valve, temperature sensor and pressure tap. The bypass valve is of a self-relieving, low pressure, large area design. It latches in either the open or closed position to minimize introduction of heat into the flow stream. The pressure tap is located at the entrance to the measuring cylinder to maximize accuracy.

A support structure is attached to the cell base. It holds the measuring cylinder, infrared light emitters and detectors, as well as the cell's electronic circuitry. Detector slits are mounted directly to the cylinder's outer surface for maximum repeatability. A high-accuracy, high-speed transducer system allows dynamic standardization techniques to be used, enhancing accuracy. Each interchangeable cell contains its own signal processing circuitry, A/D conversion and an EEROM for calibration data.

Figure 4 – DryCal ML-800 (shown with MFC)

3.0 Summary of Uncertainty Analysis

Since we wish to characterize the ML-800 over its range, we first calculate the flow-independent uncertainty and then calculate the total uncertainty over a range of tested flows for each cell size.

3.1 Flow-Independent Uncertainty

The flow-independent sources are summarized in Table 1.

Table 1 – Flow-independent Uncertainties (Type B)

Flow-Independent Uncertainties (Type B)	Low u (PPM)	Medium u (PPM)	High u (PPM)
Diameter			
Micrometer resolution (piston)	32	12	66
Micrometer resolution (plug gauge)	12	12	66
Plug gauge	32	12	13
Annular gap	134	91	69
Distance			
Upper slit width	78	86	99
Lower slit width	78	86	99
Upper detector measurement	19	25	25
Lower detector measurement	24	29	29
Piston rocking	24	82	84
Time			
Time base	29	29	29
Pressure:			
Barometric transducer	205	205	205
Differential transducer	22	22	22
Temperature			
Reference standard	59	59	59
Bath	10	10	10
Linearity of sensor system	55	55	55
0.1C Difference, sensor to stream	195	195	195
Coefficient of linear expansion	36	36	36
U, (RSS of above components, ppm)	351	348	362

3.2 Flow-Dependent Uncertainty

Flow-dependent uncertainties consist of the statistical data from the repeatability root-sum-squared with the leakage uncertainty. The total flow-dependent uncertainties are shown in Table 2.

Table 2 – Flow-Dependent Uncertainty (Type A)

Low		Medium		High	
Flow (~sccm)	u (ppm)	Flow (~sccm)	u (ppm)	Flow (~sccm)	u (ppm)
1	1045	50	97	300	108
2.5	524	150	104	500	99
5.0	319	400	81	1500	83
15	270	900	81	2000	44
40	294	2100	75	4000	56
90	272	5000	162	9000	74
210	213			21000	180
500	170			50000	335

3.3 Total Uncertainty

Finally, we can combine (root-sum-square) the flow-independent uncertainties of Table 1 with the flow-dependent uncertainties of Table 2 and multiply by 2 to obtain the expanded uncertainties of Table 3.

Table 3 – ML-800 Prover Expanded Uncertainty (2X, Standardized)

Low		Medium		High	
Flow	**u**	**Flow**	**u**	**Flow**	**u**
1	0.220%	50	0.072%	300	0.076%
2.5	0.126%	150	0.073%	500	0.075%
5.0	0.095%	400	0.071%	1500	0.074%
15	0.089%	900	0.071%	2000	0.073%
40	0.092%	2100	0.071%	4000	0.073%
90	0.089%	5000	0.077%	9000	0.074%
210	0.082%			21000	0.081%
500	0.078%			50000	0.099%

The expanded uncertainties can be visualized more easily in graphical form in Figure 5, Figure 6 and Figure 7 We have added a line representing the average of ten readings to each, derived by dividing the repeatability by the square root of ten before inclusion with the other errors.

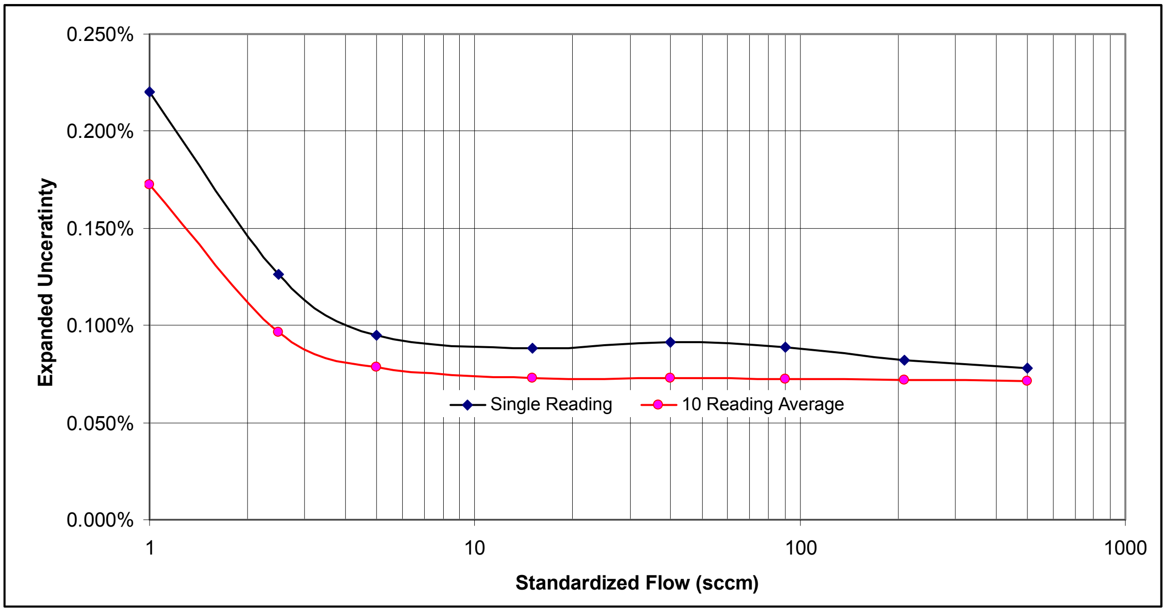

Figure 5 – ML-800 Low Cell Expanded Uncertainty (2X)

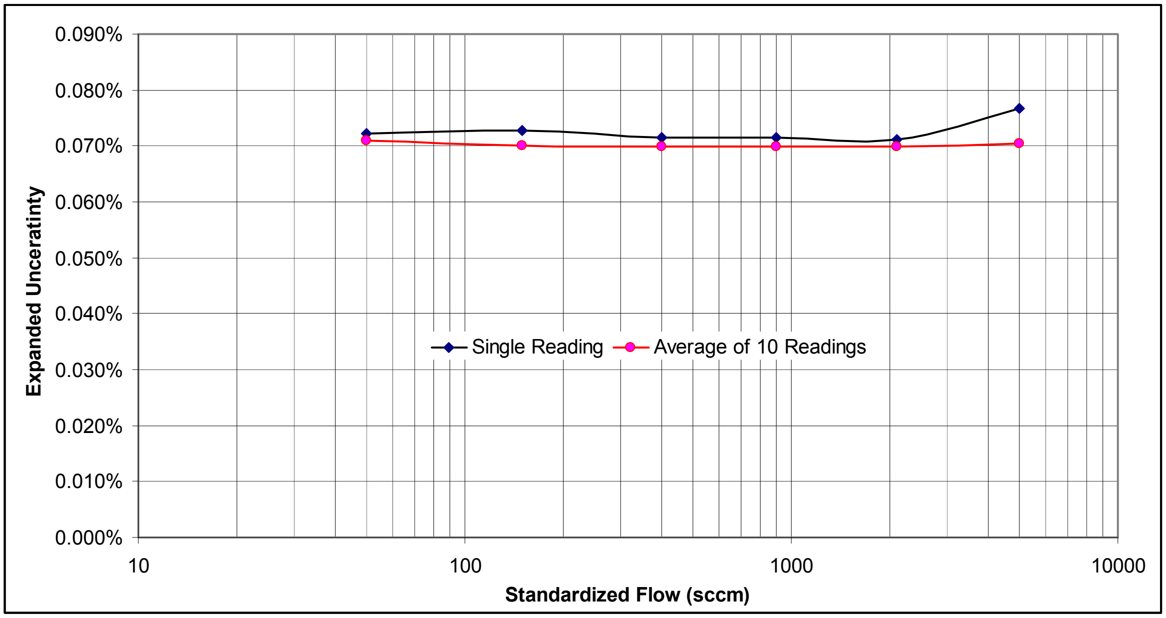

Figure 6 – ML-800 Medium Cell Expanded Uncertainty (2X)

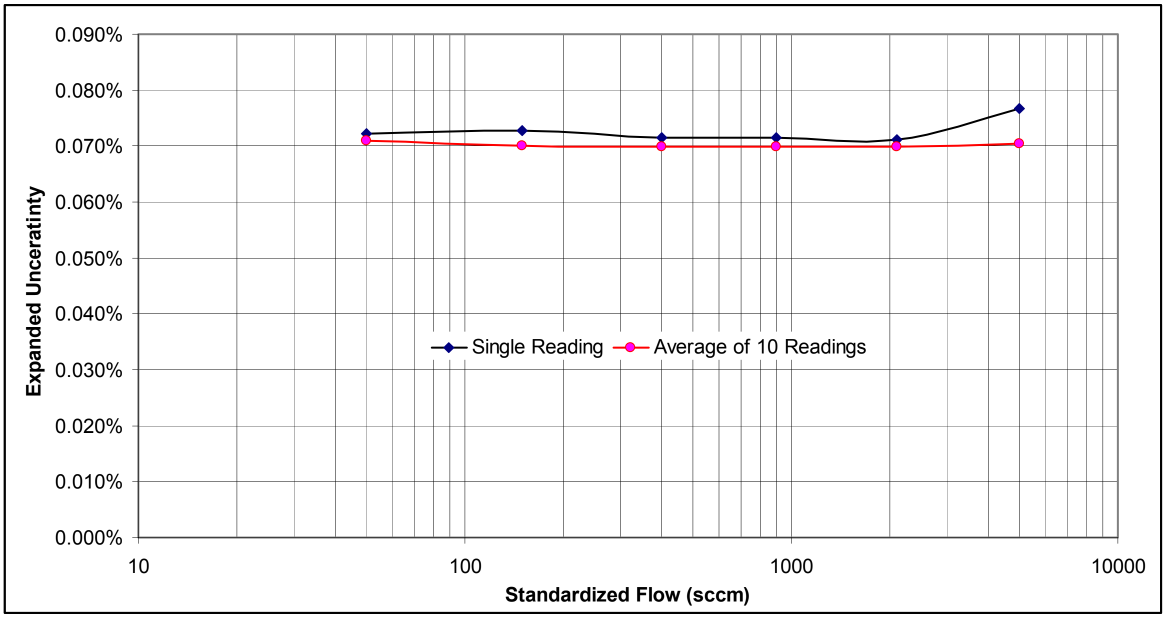

Figure 7 – ML-800 High Cell Expanded Uncertainty (2X)

3.4 Need for Inter-Laboratory Comparison Program

Uncertainty analyses of the ML-800, currently our most accurate prover, showed a theoretical uncertainty of approximately 0.075%. The uncertainty analyses were based, as much as possible, on empirical (type A) evidence. The high speed and automation of the provers was helpful, as large amounts of data could be acquired quickly and automatically for such variables as reproducibility.

However, uncertainty analyses are not sufficient to assure instrument accuracy. Factors may be omitted or miscalculated, and it is necessary to provide empirical validation of the analysis. Therefore, a program was needed comparing the provers to other laboratory instruments. At this level of accuracy, though, there are no instruments capable of providing a direct measure of the DryCal's accuracy. Rather, it was necessary to formulate a program of peer-to-peer comparisons.

First, we needed to design experimental configurations that minimized device interaction. We also studied the effects of inventory volume on DryCal accuracy to prevent errors from that source.

4.0 Application Uncertainties and Configurations of Experiments

Although the DryCal's dynamic pressure effects are very small, in some circumstances they may affect the measurement or interact with the device under test. For the above reasons, certain precautions should be observed when using a DryCal.

4.1 Device Interactions

Any measurement interacts with the device being calibrated to some degree. Often, these interactions are negligible. However, sometimes device interactions can seriously affect measurement accuracy. Here we will explain what happens during a DryCal measurement to aid in using the instrument appropriately.

In its inactive state, the DryCal, like any device, will exhibit a constant insertion pressure drop. At all but the highest flows, the pressure drop is very small. In the inactive state, gas flows from the inlet to the outlet through the bypass valve (Figure 3). When a measurement cycle begins, the bypass valve closes, and the gas is directed into the cylinder, effectively inserting the piston in series with the gas flow, allowing measurement. Timing commences after the piston has accelerated to the flow stream's speed. At the end of the timed cycle, the valve opens and the piston falls to its inactive position at the bottom of the cylinder.

4.2 Initial Pressure Pulse

Figure 8 is an illustration of a typical DryCal's internal pressure during a measurement cycle. A near-maximum flow rate is illustrated to accentuate the pressure variations. At the beginning of a cycle, pressure rises rapidly until the piston accelerates to the speed of the flow stream. The initial pressure pulse, lasting some tens of milliseconds, reaches a peak of about 0.5 kPa, or 0.5% of its working, near-atmospheric pressure. The pressure settles out to about 0.1 kPa (0.1% of working pressure) during the timed period. This pressure represents the added pressure due to the weight of the piston. The underdamped piston exhibits declining oscillations about an asymptotic increase toward the final pressure. Very small oscillations continue due to the piston's under-damped nature.

The initial pressure pulse is small, about 0.5% of an atmosphere. However, even so small an increase may affect some very sensitive transducers (such as the resonant transducers used in LFE systems) for several seconds. For this reason, an LFE instrument may not be accurate for a number of seconds after the start and the end of a DryCal measurement cycle. When calibrating such systems, a stable flow source should be used and the LFE read before and after the DryCal cycle.

4.3 Intra-Cycle Pressure Change

After the initial brief pressure pulse, the change in insertion pressure is typically 0.1 kPa (10-3 bar). This is usually insignificant. For example, flow from a 100 kPa gauge source will change by 0.1%. However, very low-pressure sources will show larger flow change during a DryCal cycle and may require compensating calculations to achieve DryCal's best applied accuracy.

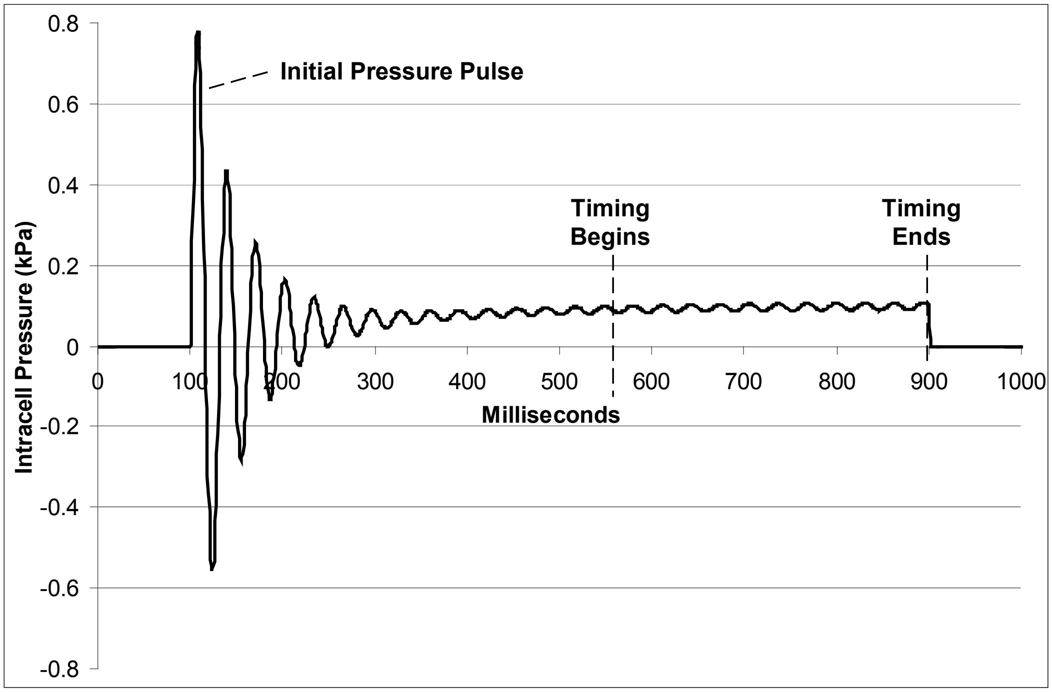

Figure 8 – DryCal Internal Pressure

4.4 Inventory (Dead) Volume

Inventory volume consists of all the space contained between the flow source's point of restriction and the timed portion of the cylinder. This includes tubing, empty space within the DryCal base, the lower portion of the measuring cylinder and any other space contained within the test configuration.

It is important to keep inventory volume to a minimum. Excess inventory volume amplifies the effects of the pressure variations within the DryCal cell. In extreme cases, the excess volume also prevents gas pressure from accelerating the piston properly, causing significant errors in readings.

4.5 Inventory Volume Studies

In order to assess the effect of inventory volume, an empirical study was conducted. Each DryCal cell was connected to a constant flow source and its reading recorded. Subsequently, a roughly binary volume progression of empty vessels (accumulators) was connected to the instrument inlet.

Without varying the flow source, the subsequent reading was recorded and compared to that originally obtained. The result was recorded as an inventory volume-increase (over the volume intrinsic to the cell) – related error as a function of vessel capacity, with flow as a parameter. The results obtained for the ML-800 devices discussed herein are shown in Figure 9, Figure 10 and Figure 11.

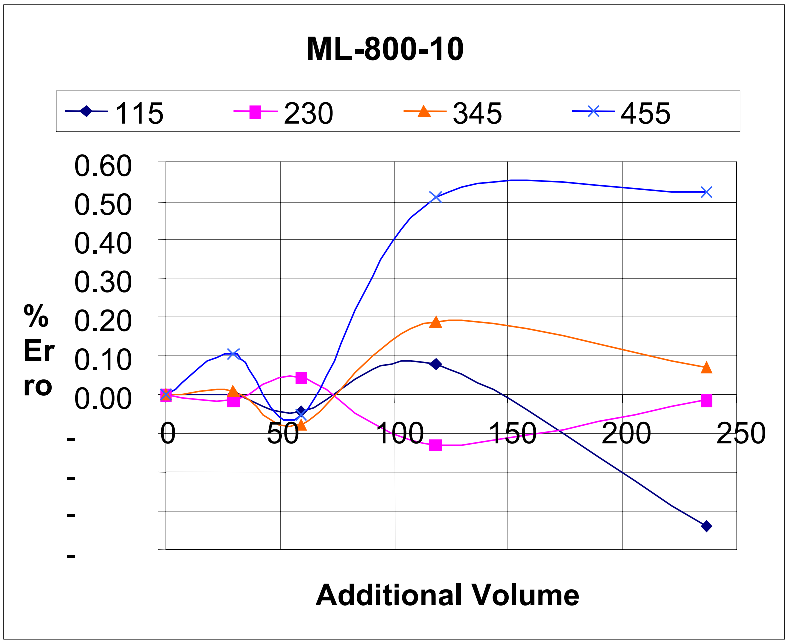

Figure 9 – ML-800-10 (Low) error vs. added inventory volume

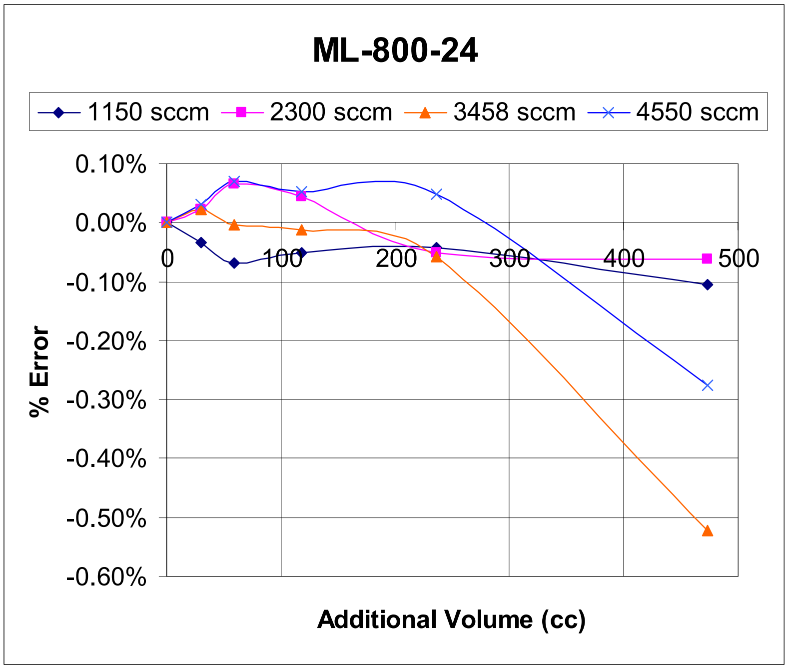

Figure 10 – ML-800-24 (Medium) error vs. added inventory volume

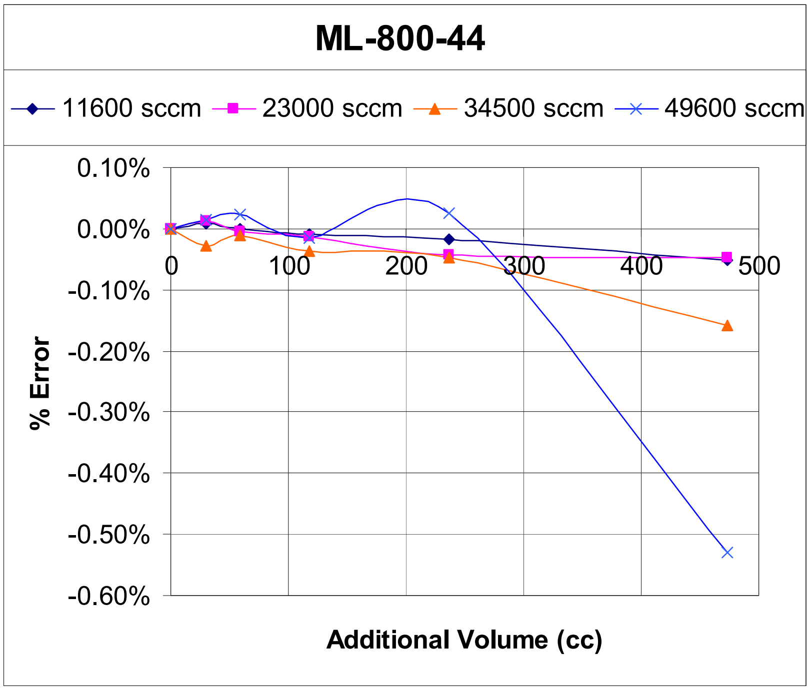

Figure 11 – ML-800-44 (High) error vs. added inventory volume

The volume contained between the cell and the flow source should be no more than that shown in Table 4, which also shows the volume as an equivalent length of tubing.

Table 4 – ML-800 Maximum External Volume and Tubing Lengths

	Recommended Maximum Volume (cc)	**Recommended Maximum Length (meters)**		
Tubing Inside Diameter	**N/A**	**3mm (1/8 inch)**	**6mm (1/4 inch)**	**9mm (3/8 inch)**
Low Cell	30	4.2	1.1	0.5
Medium Cell	100	14	3.5	1.6
High Cell	300	42	10	4.7

4.7 Comparison Methodologies

Comparison of DryCal with Piston or Bell Provers

Piston or bell provers have a much longer measurement time than DryCals. For this reason, it is possible to compare them simultaneously, but certain precautions must be observed. When the DryCal begins its cycle, the piston's weight causes the internal pressure to rise by about 0.001 atmospheres (~0.1 kPa). If a simple pressure regulator feeds the test chain, we are simply using the resistance of the entire flow chain to set our flow rate. The rate will then change significantly when the DryCal is in its measurement cycle. This will cause the actual flow measured during the DryCal cycles to be less than the average flow seen by the piston or bell prover.

To render this effect insignificant, the flow must not be affected significantly by the DryCal's cyclic pressure increase. This can be achieved by use of a critical flow venturi as the stable flow source, or by feeding a fixed restrictor with a precisely regulated pressure of more than 200 kPa, as Bios' MFS Flow Bench. Note that at 200 kPa (30 PSI), the dynamic flow decrease of a simple restrictor caused by the piston's weight will be about 0.05%.

For this type of calibration, we can use the configuration shown in Figure 12. The adjustable regulator is used to set the flow rate within the range of a properly sized flow restrictor. A piston or bell prover cycle is instituted. The DryCal and the prover can then be alternately measured using the fixed flow source.

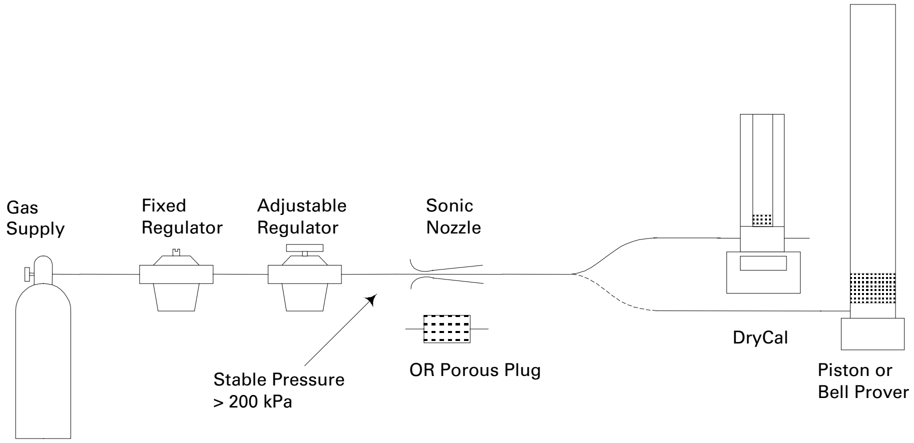

Figure 12 – Configuration for Piston or Bell Prover

An alternative approach can be used with piston provers, as shown in Figure 13. A cycle is initiated on the prover, which is much slower than a DryCal. The DryCal is then started in a cyclical mode, averaging its flow. Before the prover ends its cycle, the DryCal is stopped and the average flow read.

The DryCal can be set for sufficient cycles in its average to allow interruption by the Stop button, or smaller averages, such as 5 or 10 readings, can be taken during the prover cycle. It should be noted that the periodic pressure pulses might cause oscillations in bell provers, reducing the bell proverb's accuracy somewhat.

In certain circumstances, the critical flow venturi or porous plug flow generator may be replaced with a mass flow controller (MFC). However, due to MFC instability, very misleading results may be obtained, especially if the MFC is turned down to less than 10% of full flow. Because MFC stability is typically less than DryCal accuracy, Bios does not recommend the use of an MFC as a calibration flow generator.

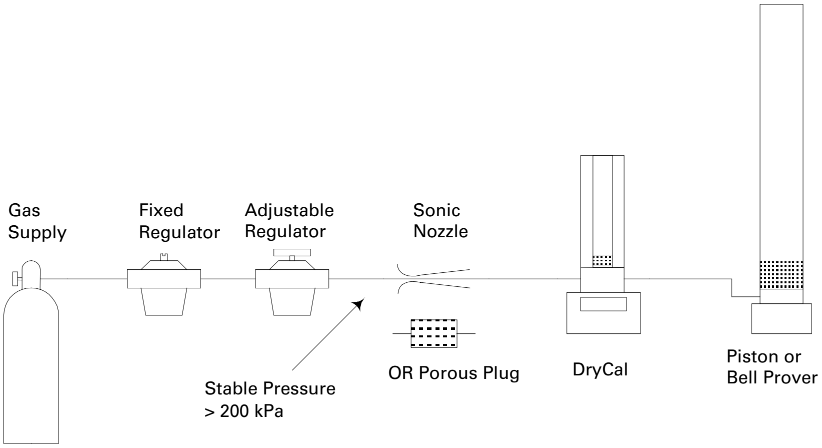

Figure 13 – Alternative Configuration for Piston Provers

Comparison of DryCal with Critical Flow Venturi Transfer Standards

A high quality critical flow venturi used above its critical pressure ratio will supply a constant flow despite changes in its outlet pressure. For this reason, a calibrated critical flow venturi can be compared to a DryCal by simply connecting its outlet to the DryCal's inlet as shown in Figure 14.

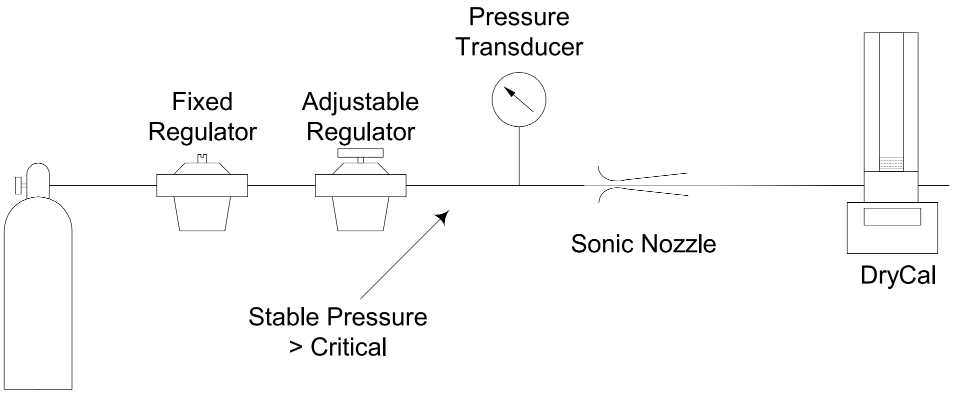

Figure 14 – Configuration For critical flow venturi Transfer Standard

5.0 Inter-Laboratory Comparisons

5.1 Initial Linearity Study (NIST)

We wished to eventually demonstrate the absolute accuracy, long-term reproducibility and linearity of the DryCal over its operating range. Because they possess extremely well defined standards over a wide range of flows, the CRITICAL FLOW VENTURI-based systems of NIST and NMIJ were used to demonstrate linearity for flows of 0.1 slm to 50 slm. It is hoped that, in the future, NIST's low-flow piston generator can be used to similarly characterize flows below 100 sccm.

Initially, we only tested linearity at NIST, as the complete, standardized instrument had not been completed. The readings were standardized to the laboratory's ambient conditions. The NIST tests consisted of 30-second groups of DryCal readings, which were compared to the average indicated flow of the NIST critical flow venturis during the test interval. Such tests were conducted several times for each flow on each cell.
An example is shown in Table 5.

Table 5 – Typical NIST flow comparison

NIST 4-9-03 Medium Cell. 2000 sccm		
NIST Flow	**Indicated Flow (Standardized)**	**Deviation Indicated/NIST Flow**
2195.53	2195.6	0.003%
2195.53	2195.4	-0.006%
2195.53	2195.3	-0.010%
2195.53	2195.0	-0.024%
2195.53	2195.4	-0.006%
2195.53	2195.2	-0.015%
2195.53	2195.6	0.003%
2195.53	2195.6	0.003%

The results were quite encouraging, exhibiting a linearity of ±0.045% with respect to NIST's critical flow venturis. The agreement between the different NIST critical flow venturis was remarkable, typically ±0.02%.

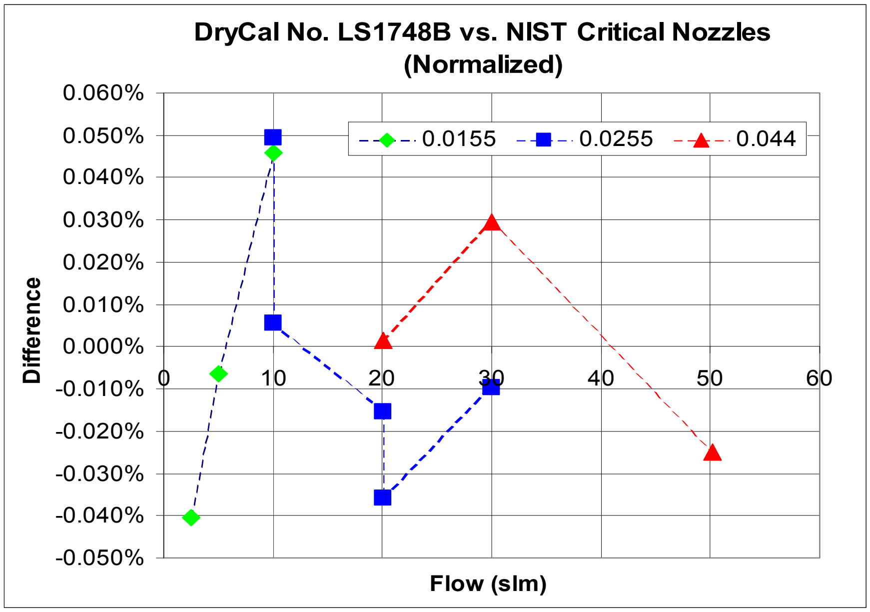

Figure 15 – Linearity test at NIST, manually standardized to ambient

5.2 Linearity and Bias

Of course, linearity is only a beginning point. The effects of internal standardization and bias required confirmation. At NMIJ, and later at NIST, we conducted tests of absolute correlation against each laboratory's calibrated flow critical flow venturis.

5.3 NMIJ

NMIJ's excellent critical flow venturis allowed us to compare flows down to 100 sccm. Flows were measured in groups of 10, with NMIJ supplying their average flow reading for comparison (Table 6)

Table 6 – Typical single test group at NMIJ (average of 10 readings)

NMIJ 5-27-03 – Medium Cell, 2000 sccm		
NMIJ Flow	**Indicated Flow**	**Difference**
2000.651	2000.6	-0.003%
2000.651	2000.4	-0.013%
2000.651	2000.5	-0.008%
2000.651	2000.7	0.002%
2000.651	2000.9	0.012%
2000.651	2000.9	0.012%
2000.651	2000.6	-0.003%
2000.651	2000.6	-0.003%
2000.651	2000.6	-0.003%
2000.651	2000.5	-0.008%

Several groups were taken for each critical flow venturi at each flow. The results are presented graphically in Figure 16 (results of flow readings outside the cell's rated ranges are omitted). As with the earlier NIST linearity tests, the two differently sized pistons exhibited close correlation (approximately ±0.03%) at similar flows.

All results were within the expected range based upon the combined uncertainties of the two systems. Nonetheless, two points were of concern. At 500 sccm, there was a discrepancy of almost 0.1%. Since this point is in the middle of the cell's dynamic range, the discrepancy was not expected. At 50000 sccm, the DryCal indicated 0.15% higher flow than NMIJ. As this point is at the extreme high end of its cell's range, non-linearity due to inadequate acceleration distance or insufficient valve holding pressure may be possible causes.

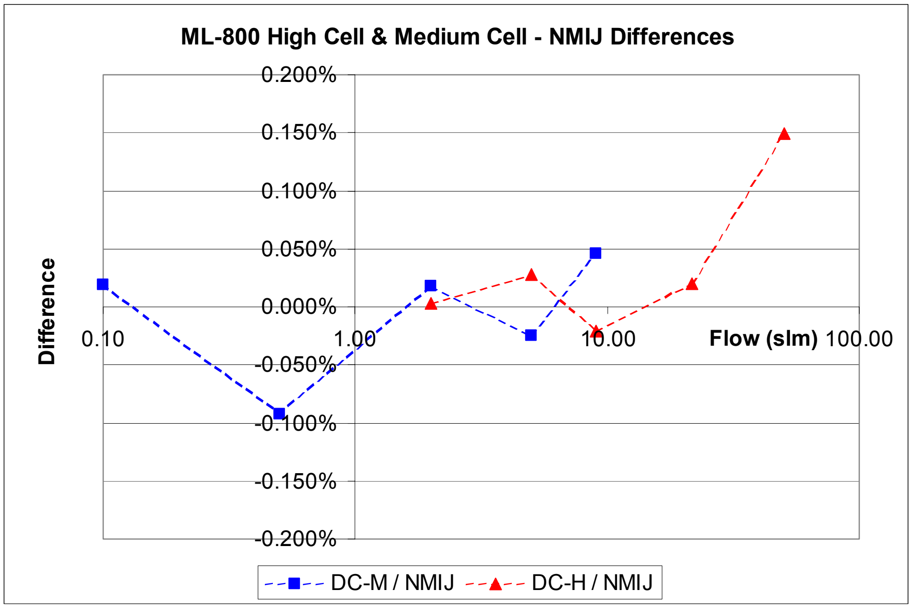

Figure 16 – NMIJ comparison, May 2003, medium and high flow cells (No. 100, 101)

5.4 NIST

To assess long-term stability, absolute flow comparisons were performed at NIST in October 2003 using the two cells tested five months earlier at NMIJ. In addition, two newly manufactured cells were tested, one of each size. All four cells compared within acceptable limits, with the exception of the 50000 sccm point of Cell No. 101 (Figure 17), which had been 0.15% higher than NMIJ's readings in the earlier study.

In order to determine whether more piston acceleration distance was required because of the high piston velocity at 50000 sccm, Cell No. 10's acceleration path was lengthened. Nonetheless, its readings at 50000 sccm were 0.20% higher than NIST's.

A comparable newly manufactured cell, No. 447, displayed only 0.035% discrepancy at NIST. It appears that Cell No. 101's 0.15% to 0.2% disagreement was anomalous, and that further investigation will be required.

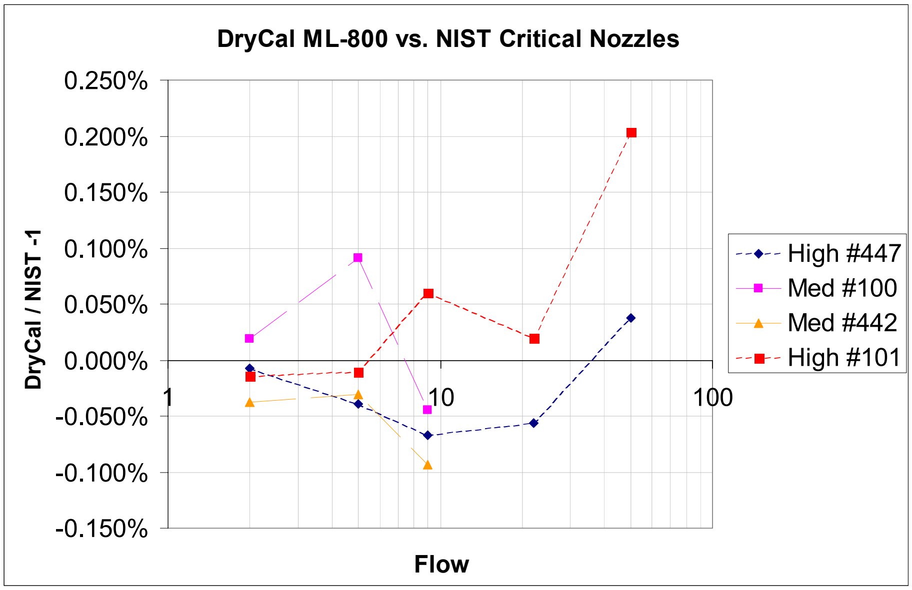

Figure 17 NIST comparison, October 2003, cells (No. 100, 101) and two new cells

5.5 Single Flow Multi-Laboratory Study and Youden Analysis

A flow of 2 slm can be measured by a majority of the world's labs. Therefore, comparisons were made with a number of national and private labs, over a period, at that flow. One medium-flow (0.1 to 5 slm) cell and one high-flow (1 to 50 slm) cell were tested at each laboratory. The percentage deviation of each lab's readings with respect to the two cell's readings was used as the X and Y-axes for a single point to form a Youden plot (Figure 18). So far, six points have been plotted: NIST and NMIJ (using critical flow venturis), two other national labs and two commercial labs (all using piston provers).

As can be seen, the two DryCal cells' readings grouped within ±0.05% of the 45-degree line, implying that the cells and their application error were reproducible to ±0.05%.

NMIJ and NIST were within 0.04% of the origin, exhibiting the excellent agreement between all three measurement systems. "National Lab 1" and "Commercial Lab 1" use piston provers with MFC flow generators and they remained within 0.1% of the origin. "National Lab 2" used critical flow venturis. However, they were due for calibration and exhibited a bias of 0.15%. "Commercial Lab 2" used bell provers with an MFC flow source, but the regular operator was not available and we suspect misapplication caused the apparent error of 0.4%.

We believe that this plot demonstrates both the suitability of the Youden method for inter-laboratory comparison and the ability of the viscous sealed prover to serve as reliable, transportable transfer standard for such use. It is interesting to speculate on Youden plots constructed with two independent methodologies: A critical flow venture for one axis and a viscous-sealed prover for the other. The diversity of methodologies could well improve the validity of the results.

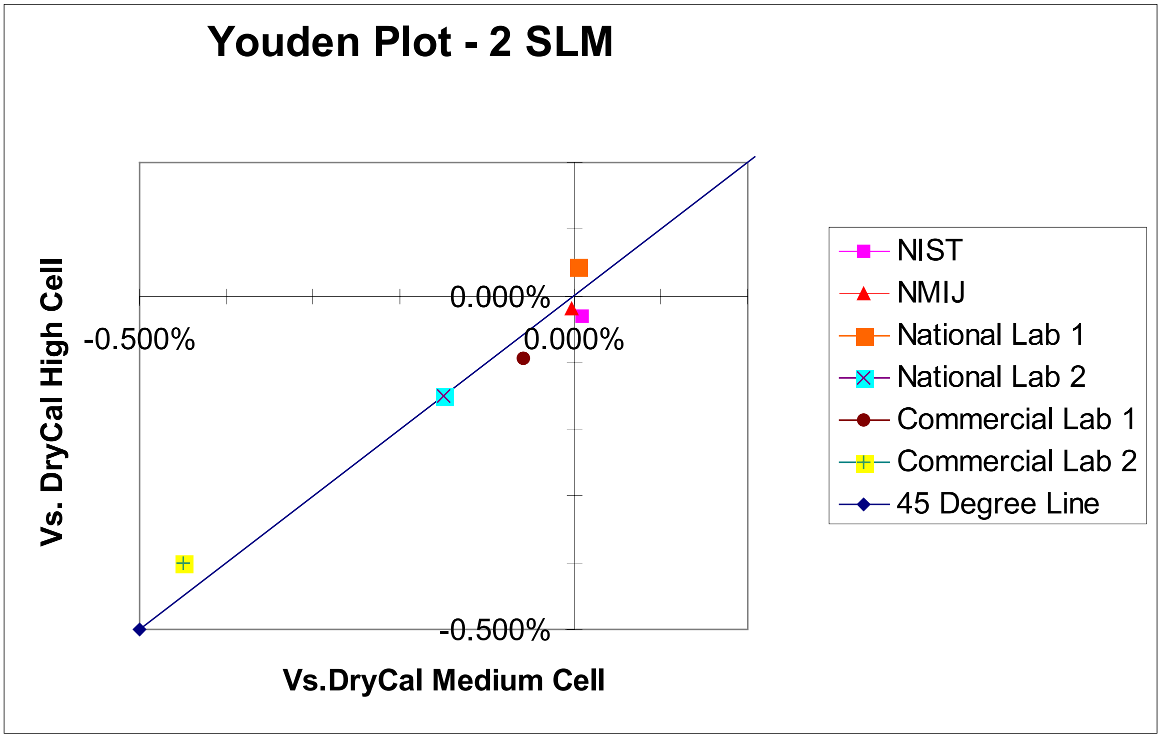

Figure 18 – Youden plot, medium and high cells No 100 and 101 vs. six laboratories

6.0 Conclusions

The results have been very encouraging. Typically, the dry piston provers exhibited discrepancies of about 0.05% in comparison with critical flow venturis at NIST and NMIJ, confirming their uncertainty analyses.
In addition, the provers exhibited excellent accuracy and transportability as inter-laboratory transfer standards.

On the other hand, we have concern about the 0.2% discrepancy observed on a single cell at 50000 sccm.

7.0 Future Studies

We must determine whether the 0.15% to 0.2% discrepancies exhibited by Cell 202 at 50,000 sccm were anomalous or a limitation of the design. Therefore, we are conducting additional linearity studies to determine a proper specification for flows in the 30000 to 50000 sccm range. Linearity can be assumed at low piston velocities provided it is tested at a range of higher velocities. This can be achieved for the two lower-flow range cells by comparison in the range where each one overlaps a higher-flow range cell. However, we cannot use such a method for the highest range.

We propose to characterize linearity at the highest flows by a simple summing process. We will measure two stable, independent flow sources with the same prover. Next, we will combine the two sources with a Y-fitting and measure the combined flow with the same prover. The non-linearity will be the sum of the individual flows less the combined-flow reading. Specifically, for testing the 50000 sccm point, we will combine the flows from two critical flow venturis (CFVs). The use of CFVs for the flow sources will minimize any error caused by the pressure increase which will be introduced by summing the flows in to a single fitting. Based upon the results, we can test a number of flow cells and determine an appropriate instrument specification for the highest flows.

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