

## Dissemination of Primary Gas Flow Standards

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Driving a Higher Standard  
in Flow Measurement<sup>SM</sup>

## Abstract

Increasing the world's wealth requires dissemination of increasingly accurate measurement standards, both for field use and for comparison of geographically dispersed laboratories and NMIs. For some units of measure, such as gas flow, this has been difficult to achieve. The primary devices used at NMIs, such as mercury-sealed piston provers and bell provers, are virtually impossible to ship in a calibrated state, while secondary standards such as laminar flow elements and sonic nozzles lack the stability or dynamic range of primary standards.

To answer this need, a series of primary piston flow provers have been developed that utilize viscous seals. They are small, stable and readily shippable. The instruments presently cover the flow range of 0.1 sccm to 500 slm and are conservatively represented as having combined standard uncertainties of 0.15% to 0.2%.

These provers have been used to informally compare laboratories on multiple continents, in one instance obtaining a correlation of 0.02% between the NMIs on two continents. Using this equipment, our laboratory has been accredited by NVLAP to ISO 17025 at a combined standard uncertainty of less than 0.07% at flows from 5 sccm to 50 slm.

This paper presents a design and development overview, summary uncertainty analysis, inter-laboratory comparisons and validation data for the viscous-sealed provers. In addition, it will discuss automation of flow calibration for mass flow controllers and validation of primary provers, themselves, using these instruments.

## Introduction

Dissemination of calibration over distances is straightforward for many primary units of measure. Accurate gauge blocks or voltage references, for example, are relatively straightforward to produce and to ship over distances with great confidence. However, complex derived units such as gas flow present significant difficulties.

Nonetheless, international harmonization of gas flow is essential to allow multinational production of precision products such as semiconductors and pharmaceuticals. Interestingly, dissemination of standards is not limited to the BIPM or NMIs alone. Supranational organizations such as corporations and military organizations require disseminable calibrations as well.

## **Risk Budgets for Dissemination**

It is interesting to consider risk budgets for reproducibility of each parameter with shipment. The effects of time, temperature, vibration, shock and other factors should be assessed. The risk for each parameter can then be included in an uncertainty analysis of the entire calibration process.

As an example, pressure transducers are often affected by shipment to a degree far greater than the accuracy to which their parameter can be known locally. Yet, pressure is a major factor in gas flow measurements. In this case, there is less risk in shipping a pressure-independent artifact (standard) and applying its pressure corrections locally, or at least recalibrating its pressure transducer locally. The obvious implication is to minimize uncertainty by shipping the least amount of parameters necessary, with particular emphasis on local validation of more accurately known parameters that do not “ship well”.

## **Minimal Primary Flow Dissemination**

Since time is a very well characterized parameter, a minimal shippable parameter for flow comparison is a well-known volume, which can be used for PVTt or for constant-pressure flow determination. However, such systems are not capable of shipment by practical means.

Our approach depends upon shipment of a constant-pressure piston prover with very stable swept volume, but of very small size. This is achieved by use of extremely small tolerances. In fact, the piston and cylinder are built with a separation of only several microns between them. This allows sealing to be performed by the viscosity of the gas under test. This is a key design feature, because there is no uncertainty resulting from deformation of piston seals (elastic or mercury).

## Piston Provers

Constant-displacement flow provers are, perhaps, the simplest and most intuitive flow measurement devices. They are characterized by the most basic of quantities: Length and time. As flow is necessarily a derived unit, provers are as close as possible to direct traceability from SI units.

An idealized piston prover would consist of a massless, frictionless, leakproof, 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 (ideally) be as accurate as its physical dimensions and its clock, with insignificant drift mechanisms.

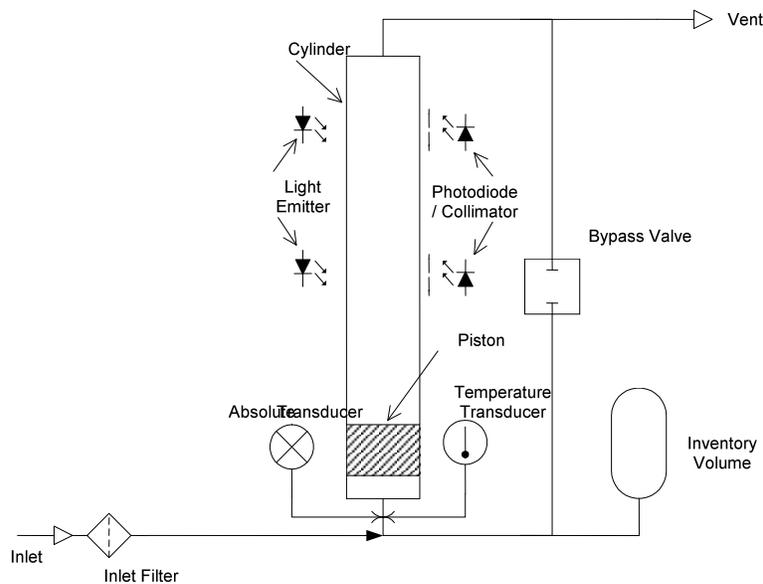


Figure 1: Piston prover principle

## Viscous-Sealed Prover Design

A typical measuring element is shown in Figure 2. It is 2.4 cm in diameter and 20 cm in length. The piston is constructed of graphite and the cylinder of borosilicate glass. Both materials have near-identical temperature coefficients of expansion of about  $3 \times 10^{-6}/^{\circ}\text{C}$ . This allows the close fit of approximately 8 microns required of the piston and cylinder, while making volumetric expansion due to temperature minimal.

Having a very well-known diameter, we must now have a very accurately-known swept path length to obtain a well-known volume. This is achieved by attaching very fine collimating slits to the cylinder, such that the distance between slits is determined by the stability of the borosilicate cylinder (Figure 3). The effective slit width is further reduced by adaptive means: The light and dark levels of each emitter-detector pair are measured on each cycle and the center point is detected as the average of the two levels. This adaptive technique eliminates the effects of ambient lighting and sensitivity of the electro-optical devices (Figure 4).



Figure 2 - Viscous-sealed piston/cylinder assembly

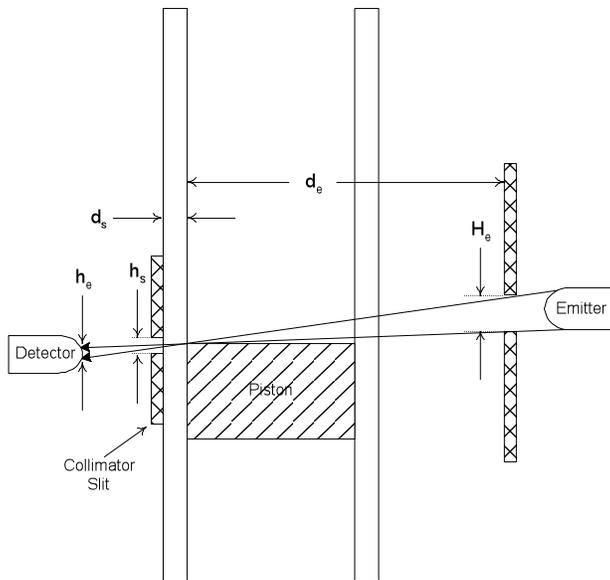


Figure 3: Optical piston detection

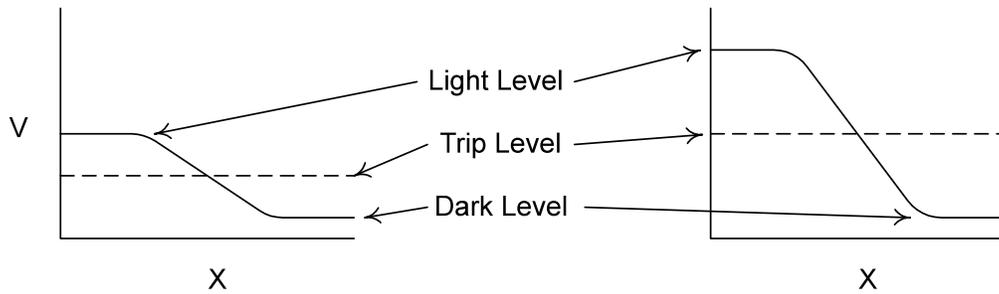


Figure 4: Adaptive slit-center detection

## Dynamic Pressure Change

As a piston prover is potentially subject to accelerative, oscillatory and piston-jamming effects, internal dynamic pressures must be measured to minimize uncertainty. To a first order, pressure only needs to be measured at the beginning and end of the ti-med measurement period. From the Ideal Gas Law, flow will be given by:

$$\dot{V} = \dot{V}_O \left[ \frac{P_2}{P_A} + \left( \frac{P_2 - P_1}{P_A} \right) \frac{V_I}{V_M} \right]$$

Where:

$\dot{V}$  = Flow

$\dot{V}_O$  = Uncorrected Flow

$P_A$  = Ambient Pressure

$P_1$  = Pressure at start of timed period

$P_2$  = Pressure at end of timed period

$V_I$  = Inventory Volume

$V_M$  = Measured Volume

Uncorrected, the measured volume contains an error equal to the difference in internal pressure at the start and the end of the measuring period, amplified by the ratio of dead volume to measurement volume, as well as that of the pressure within the cylinder at the end of the timed period.

For this reason, true dynamic pressure measurement has been incorporated in these provers. Once the dynamic pressure correction is determined, it is used to correct for the potential uncertainty, thereby enhancing the instrument's accuracy. With knowledge of the inventory volume, which will be constant for a given instrument design using a specified amount of external volume, the uncertainty resulting from the dynamic pressure differences can be minimized. This approach's effectiveness is limited by the pressure measurement's total accuracy (including secondary uncertainties such as synchronicity and quantization) and the inventory volume uncertainty.

## Calibration Methodology

As a transfer standard, no calibration of the prover would be necessary. However, our goal is not merely inter-laboratory comparison, but also dissemination of actual calibrations. Thus, we must accurately calibrate our volume.

Analysis had yielded a very low uncertainty contribution from the piston-cylinder gap. Our diameter is thus determined by measuring the piston diameter alone. This is performed by using a laser micrometer as a comparator between a precision plug gauge and the piston being calibrated. The gauge is measured before and after the piston, which is measured in six places. The average piston readings are then proportioned to the average plug gauge readings. Note that a positioning fixture is used through the entire process to eliminate sine-theta effects from the measurement.

Swept path length is measured with a digital micrometer acting on the assembled cylinder-optoelectronic assembly. This process eliminates any possible handling effects after the calibration.

Pressure and temperature are precisely calibrated by routine means.

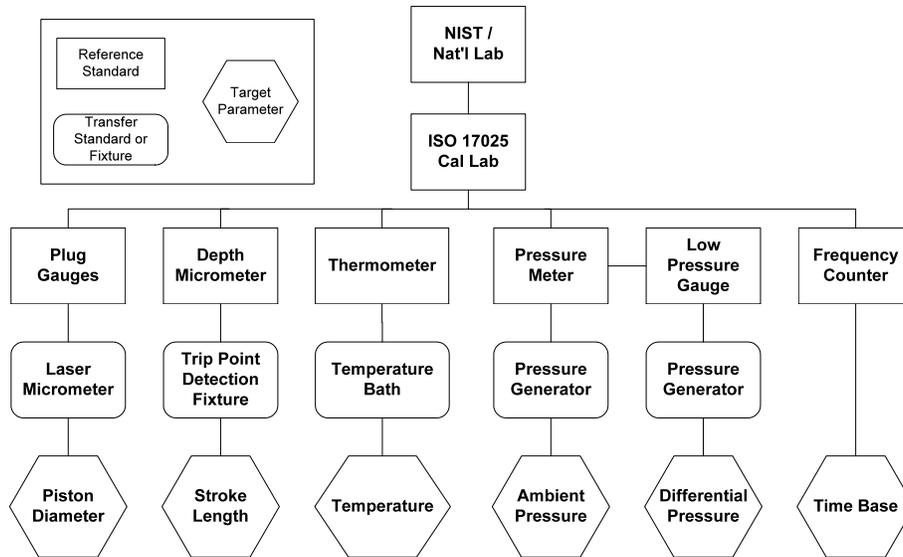


Figure 5: Calibration methodology

## Uncertainty Analysis

Finally, the prover is characterized by a comprehensive uncertainty analysis [1]. As an absolute (non-transfer) standard, the uncertainty is slightly greater than 0.06%, at which level our laboratory is accredited to ISO 17025. As a transfer standard, the uncertainty would be somewhat lower. The great majority of the world's NMIs are accredited to a lesser accuracy, so the prover is suitable as a comparison and dissemination standard.

## Informal Inter-Laboratory Comparisons

During its initial development, the prover design was tested for linearity by comparison with NIST's critical flow venturis (Figure 6). Three CFVs were tested against a single prover with remarkable agreement among the three CFVs.

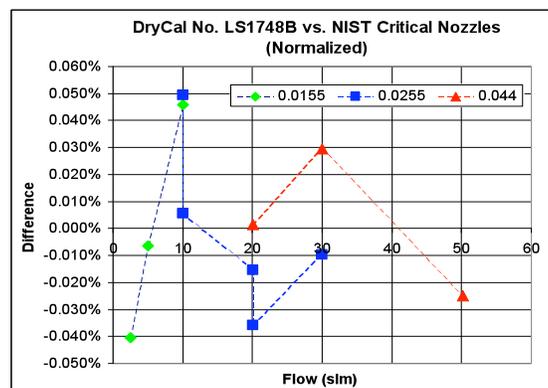


Figure 6: NIST linearity test

Two early provers were then tested for total accuracy at NMIJ in Japan (Figure 7). Agreement was typically within 0.1%. This encouraged us to proceed with a production design.

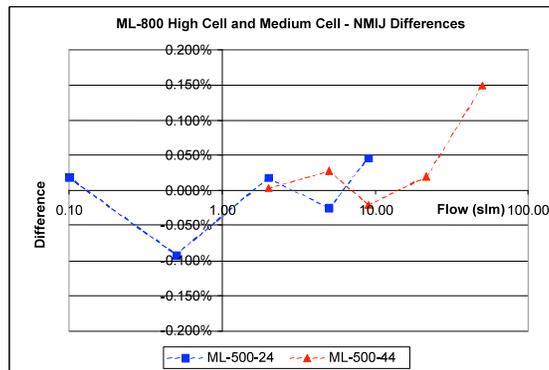


Figure 7: NIST linearity test



Figure 8: DryCal commercial calibrator (with MFC)

We then proceeded to compare the two provers to a total of six laboratories on three continents. These same two provers were shipped by air but otherwise not adjusted between comparisons. Based on the Youden plot, the provers exhibited approximately 0.05% reproducibility (as evidenced by the distance of the points from the 45° line), while the laboratories (with the exception of NIST and NMIJ, who were within 0.02% of each other) agreed to a lesser degree. Two national laboratories disagreed approximately 0.1% and 0.2%, while two commercial laboratories disagreed by approximately 0.1% and 0.4%.

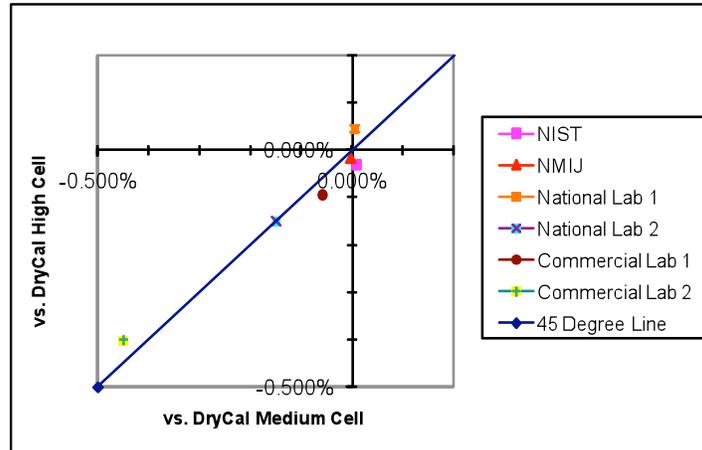


Figure 9: Youden plot, two provers vs. six laboratories

## Conclusions

As the design of instruments progresses, it is possible to compare laboratories and to disseminate complex units of measure over large distances using primary devices. This can potentially improve harmonization of laboratories, as well as allowing commercial processes to be controlled within global organizations.

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