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VERIFICATION OF GAS FLOW TRACEABILITY FROM 0.1 SCCM TO 1 SCCM USING A PISTON GAUGE

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Abstract – Fluke Calibration is accredited for gas flow measurements in the range of 0.1 sccm to 6000 slm in nitrogen and air. Traceability is maintained directly through a gravimetric flow standard but only recently from 1 to 10 sccm. The traceability of flow in the range of 0.1 to 1 sccm is based on extrapolation of the use of laminar flow elements below 1 sccm. This part of the range has never been completely verified through interlaboratory comparisons, proficiency testing or other means of measurement assurance. In an internal document from DH Instruments in the early 1990s it was suggested that a piston gauge could be used to gain traceability for very lows gas flows. In order to prove out traceability in this range an attempt was made to use a piston gauge using a piston-cylinder size of 35 mm diameter as a reference.

One of the reasons for choosing a piston gauge as a reference is its ability to control pressure. This is crucial when measuring gas flow through a laminar flow element (LFE) in this design and range. In addition, the effective area is known to within 0.001% leaving the vertical displacement of the piston to dominate the uncertainty of the dimensional part of the flow test. This was a challenge because the measurements needed to be made in absolute mode and the internal piston position sensor supplied with the piston gauge did not have sufficient precision. This paper describes the theory and design of the gas flow measurement system, the current results, and improvements needed or suggested. Two different designs are discussed, one with a single piston gauge as a reference and the possibility of two piston gauges measuring flow on either side of the laminar flow element.

Note: sccm (standard cubic centimeters per minute) is an industry accepted alternative to kg/s [1]. It is used out of convenience to normalize flow rates of gases with significant differences in density.

Keywords: piston gauge, laminar flow element, gas flow

1. INTRODUCTION

Fluke Calibration, Phoenix – Pressure and Flow laboratory is an accredited laboratory to ISO/IEC 17025 through a United States accreditation body. The accredited scope includes fluid quantities that cover gas flow for a number of gases, primarily nitrogen and air. Included in this part of the scope is a range from 0.1 to 1 sccm, with an expanded uncertainty of ±0.5% of reading. From 1 sccm to 6000 slm traceability is well established through gravimetric and successive addition techniques [2]. Recently a rate of rise reference was implemented to help validate gravimetric based traceability as low as 1 sccm [3] but was not attempted any lower due to the constraints of the system. In addition, there are not any proficiency testing or interlaboratory comparisons available in this range.

The accreditation is based on the use of a laminar flow element with a circular segment as described in [4] but nominally ten times smaller than the example described in that reference. Figure 1 is a drawing of the subject LFE with an inset of the type of gap from [4].

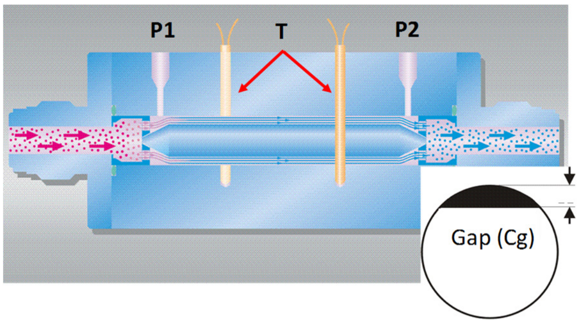

Figure 1. Drawing of LFE with inset showing circular segment gap that defines the geometrical constant (Cg)

The calculation for mass flow for this LFE is derived from the Hagen-Poiseuille equation:

$$qm = \frac{P\,(P_1 - P_2)\,\rho_N\,T_N\,Z_N}{T\,Z_{(P,T)}\,\eta_{(P,T)}\,P_N}\,C_G \tag{1}$$

where:

qm = Mass flow [kg s^{-1}]
P_1 = Upstream absolute pressure [Pa]
P_2 = Downstream absolute pressure [Pa]
$P = \frac{(P_1 + P_2)}{2}$ [Pa]
T = Absolute temperature of gas [K]
T_N = Standard temperature, 273.15 K [K]
ρ_N = Standard gas density [kg m^{-3}]
$\eta_{(P,T)}$ = Dynamic gas viscosity under P,T conditions [Pa s]
P_N = Standard pressure, 101325 Pa [Pa]
Z_N = Gas compressibility factor under standard conditions [-]
$Z_{(P,T)}$ = Gas compressibility factor under P,T conditions [-]
C_G = Experimentally determined geometrical constant [m^3]

The engineering design document for these LFEs published in 1990 internally to DH Instruments, Inc., gave a possible solution for very low gas flows where gravimetric gas flow referencing was not practicable. The suggestion was to use a piston gauge as a volume flow reference and calculate mass flow based on known gas temperature and pressure. The suggestion at the time was to use a piston gauge that operated in gauge mode and to add barometric pressure for the thermodynamic calculations. From the document:

$$q_m = A_{eff}\, v\, \rho_N \frac{(P_g + P_{ATM})\, T_N\, Z_N}{P_N\, T_g\, Z_g} \qquad (2)$$

where:

A_{eff}	=	piston-cylinder effective area	[m^2]
v	=	the fall rate of the piston	[m s^{-1}]
T_g	=	gas temperature	[kg m^{-3}]
Z_g	=	gas compressibility	[-]
P_g	=	gauge pressure	[Pa]
P_{ATM}	=	Atmospheric pressure	[Pa]

The flow rate is corrected by the rate of gas leaking through the piston-cylinder annulus using the same calculation in equation 2, however a second order fit is needed for changes in atmospheric pressure. In this case the mass flow correction is subtracted since the intent is to put the piston gauge upstream of the LFE being tested.

An attempt was made in the early 2000's with very limited success yielding expanded uncertainties at ±1% or worse. Large contributions to the uncertainty included the inadequacy of accounting for changing atmospheric pressure and to precisely measure the piston rate of change. The internal piston position sensor on the piston gauge is only intended as course reference, has limited resolution and cannot be adjusted with sufficient precision. In 2016, to comply with proficiency testing requirements it was estimated that success could be attained by operating in absolute mode and using an alternative means for monitoring the piston position.

2. SYSTEM DESIGN

There were two configurations investigated: one where the piston gauge was upstream of the LFE, and one where the piston gauge was downstream of the LFE that yielded greater success. This paper discusses the latter. The design for the system is as shown in Figure 2.

The piston gauge is operated in absolute mode by application of vacuum. The piston-cylinder used is a 35 mm diameter size with a nominal mass to pressure relationship of 10 kPa/kg and a nominal area of 980.5 mm^2. It is set with a constant nominal mass load of 9.7 kg providing approximately 97 kPa. Once the pressure is set there is no need to change masses or break vacuum unless the piston-cylinder becomes contaminated. The pressure and corresponding mass load was chosen because it is very close to average atmospheric pressure at the laboratory's location, which supports the calibrated operating condition of the LFE. The stroke of the piston-cylinder used was ±4 mm, with ±5 mm allowable before hitting upper and lower stops but was not needed.

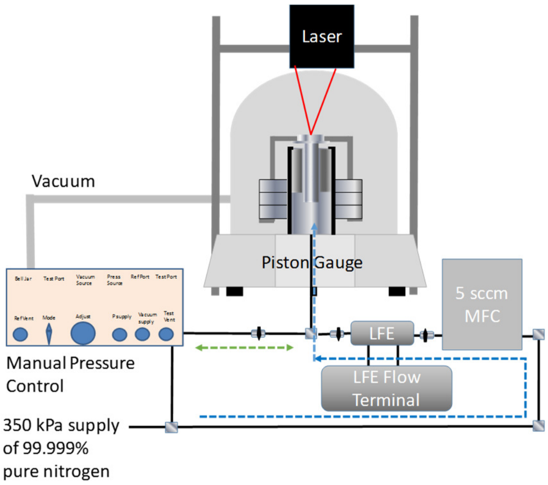

Figure 2. Design of the piston gauge flow test.

The LFE is one of the Fluke Calibration downstream #2 working reference molblocs^{TM} [5]. This means that the downstream pressure, P_2, is kept constant venting to atmosphere and upstream pressure, P_1, is varied as shown in equation 1 and Figure 1. This LFE was calibrated with an average downstream pressure of approximately 97 kPa, hence the reason for setting the piston gauge to that pressure. The flow is calculated by the flow monitor based on the average of two absolute pressure transducers, a differential pressure transducer to resolve the differential pressure given in equation 1, and two platinum resistance thermometers mounted in the LFE to measure the temperature of the gas. The range of differential pressure that corresponds to 0.1 to 1 sccm is approximately 1 to 10 kPa. In this range differential pressure is read by the differential pressure transducer. Above this flow the differential pressure is read by the difference of the two absolute transducers. The combination of the LFE and this particular flow monitor is what provides the accredited scope uncertainty of ±0.5% of reading expanded to 95% confidence.

The 5 sccm mass flow controller (MFC) has a range that is as low as what is available to our laboratory at this time. It is able to set all flows with reasonable stability. The lowest flow attempted, 0.1 sccm, could not be set very well to the nominal flow, but did stabilize very well at 0.06 sccm.

The manual pressure controller is used to keep the piston floating when not taking measurements and to reset the piston position to its lower portion of the stroke before a test is started. Though temperature control in the laboratory is as good as ±0.3 °C, the connecting hardware between the mass flow controller and the piston gauge is insulated as best as possible to eliminate influences from changes in laboratory ambient temperature.

The triangulating laser used for displacement has a range of 10 mm with a standoff of 50 mm. In its 2x exposure mode has a linearity specification of ±3 um. The calibration certificate supplied with the laser supports this specification.

The data rate is variable. For the piston gauge flow test 500 samples per second is selected and is well within the laser's capability. Data acquisition was set to 100 samples per second to keep data files manageable under 40 000 readings per test, with each test lasting no more than five minutes.

Though the laser came with its span calibrated, this could not be depended upon due to the fact it is being used to measure displacement on the other side of the glass bell jar. There are two ways of accounting for this error, both in-situ methods. The more straight forward method is to measure the full displacement of the piston from top to bottom stops in gauge mode without the bell jar, then adjust the laser accordingly in absolute mode through the bell jar. The other method is to place a known step, in this case a gauge block, in line with the laser measurements as the piston-cylinder rotates. Using these two methods a standard uncertainty in rate of change of piston position could be verified within 0.07%. Figure 3 is a picture of the working piston gauge flow test with an inset showing the laser target on the top of the masses, and also a closer look at the insulation of the LFE and connecting tubing.

Data acquisition is through the manufacturer's software for laser displacement and a Fluke Calibration software package for reading all other data including the pressure and temperature of the gas, and the flow rate as calculated by the flow monitor. Though not completely necessary because of the flow stability obtained with the mass flow controller, the data acquisition is synchronized within one second. Time is monitored through the computers clock used for data acquisition. A simple verification with a laboratory frequency counter showed this to be within 0.01%.

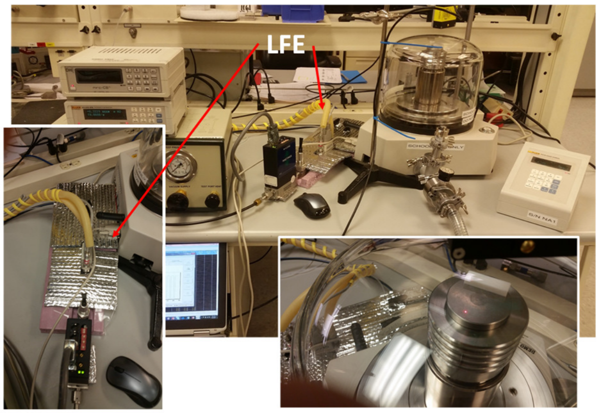

Figure 3. Picture of piston gauge flow system with insets showing laser target with dimensional step, and LFE and hardware insulation.

3. PROCEDURE AND RESULTS

The process for taking a flow point is reasonably straightforward, but somewhat manually intensive. Most of the points are taken in less than five minutes, and the entire process for specific flow rate would take approximately an hour, depending on how many points are desired for the flow rate. At the beginning of the process the laser is adjusted for its span as described in the previous section. The piston gauge is then put into float and thermally stabilized for one hour. At this time the residual pressure underneath the bell jar is allowed to reduce below 1 Pa. The following is a step by step procedure of the rest of the process for each point.

1. The piston is rotated to approximately 10-15 rpm.
2. The LFE is opened to the piston gauge without flow applied. At this time the 'tare' function on the flow monitor is activated to correct line pressure errors in the differential pressure transducer.
3. The flow rate is then set by applying the appropriate voltage to the MFC.
4. Once the flow rate is set, the manual pressure controller is used to put the piston position at the lower end of its stroke. The position is chosen based on the flow rate with the intent to cover a piston position that is symmetrical to the mid float position.
5. The manual pressure controller is isolated and the data acquisition is implemented.
6. The piston position is observed and when it reaches a value that is the negative value of where the piston position began, i.e. equal across the midfloat position, the data acquisition is stopped.
7. To get multiple points at the same flow, steps 4 through 6 are repeated. Rotation rates are kept between 5 and 15 rpm so it is possible the piston needs to be increased if slowed below 5 rpm.

A procedure similar to this was observed to measure the piston fall rate correction. The difference was that the LFE was isolated from the test and the test was performed at +2, 0 and -2 mm piston positions to detect an influence from changing piston-cylinder gap size as the piston moves. The change in piston position for each of these measurements were approximately 0.4 mm, or 2.2 to 1.8, 0.2 to -0.2 and -1.8 to -2.2 sections of the piston stroke. Figure 4 shows the results of the characterization of the drop rate at those positions.

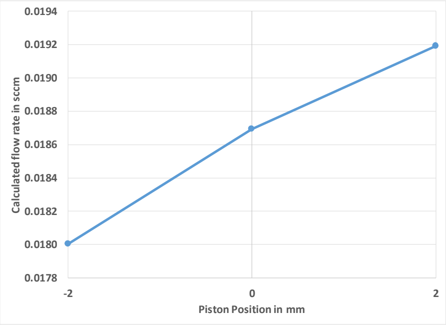

Figure 4. Results of drop rate tests with no flow at -2, 0 and 2 mm respectively to define the flow leak correction.

It was sufficient to use the average value of the drop rate correction since the flow comparison measurements with the LFE were taken symmetrically across zero. The standard error of the fit is used as an uncertainty.

At least five flow points were taken for each flow of 0.9, 0.7, 0.5, 0.3 and 0.06 sccm using the procedure. As stated earlier in this paper the laser was forced to track a disc on top of the masses that included a 5 mm gage block to use as a span correction for the laser. In order to use this data it had to be filtered for the transition points between the surface of the disc and the top of the gage block. Figure 5 shows an example of the data used to determine the rates. For each test a rate defined by measuring the surface of the disc and another rate using +5 mm from the gage block surface were combined to get the final result.

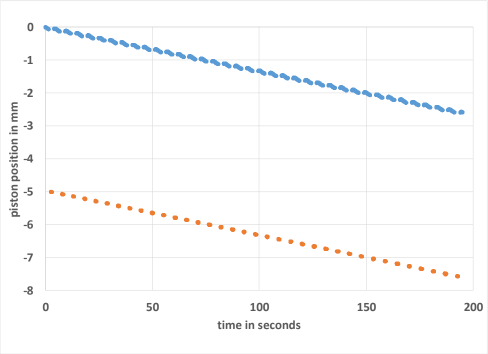

Figure 5. Filtered data for piston position and time for an example of 0.7 sccm flow rate.

The comparison between the LFE and the piston gauge flow reference is in sccm, not kg/s which changes equation 2. In this case the volume flow rate is calculated then converted to standardized volume flow rate for mass flow by correcting the volume flow rate by the ratio of the gas density, ρ_g, to the standard density of nitrogen. k is the span correction for the laser and $q_{m(sccm)dr}$ is the correction for the drop rate determined using the same procedure without the LFE.

$$q_{m(sccm)} = A_{eff}\ v\ k\ \frac{\rho_g}{\rho_N} - q_{m(sccm)dr} \qquad (3)$$

The densities used are calculated using NIST REFPROP7 [6] database both for the piston gauge flow reference and what the flow monitor uses to calculate flow from the LFE. Figure 6 is an error chart for all points taken.

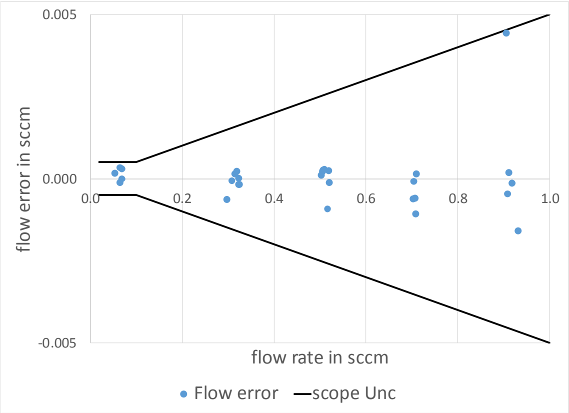

Figure 6. Error chart for all data taken.

The black lines in figure 6 represent the Fluke Calibration accredited scope uncertainties as a reference. There was an outlier at one of the 0.9 sccm measurements. The cause of this is yet unknown so is not excluded from the data set and is included as part of the Type A uncertainty for that point.

A list of uncertainties includes the Type A from the repeated tests as standard deviation of the mean [7]. An explanation of the other contributions to measurement uncertainty follows.

The correction for the drop rate of the piston-cylinder is calculated the same as the flow rate measurements and is considered fully correlated. It also includes the standard error of the fit with respect to the piston position.

The uncertainty in the gas density calculation and the ability to measure the temperature and the pressure of the gas in the piston gauge must be considered. The pressure is well known to within ±0.002% expanded to 95% confidence. The temperature is measured by the piston gauge mounting post, which has an uncertainty of ±0.1°C expanded to 95% confidence on its own, but the assumption that the gas has enough time to equalize to mounting post temperature is estimated to be an additional ±0.2°C. This is based on the measurement of the temperature exiting the LFE. This uncertainty should also include the model itself based on NIST REFPROP7 and is estimated at less than ±0.01% expanded to 95%.

$$u_{\rho gas} = \sqrt{{u_{TPG}}^2 + {u_T}^2 + {u_P}^2 + {u_{model}}^2} \qquad (4)$$

The uncertainty in fall rate includes the Type A uncertainty of the in-situ gage block corrections, including the uncertainty of the gauge block itself. The Type A was calculated as the standard deviation of the mean at 0.13% expanded to 95% confidence. The gauge block used has a verified specification of less than ±1 um. A conservative estimate of ±3 um is used. Finally, the linearity of the triangulating laser is included. The data from the manufacturer supports the claim of ±3 um and is applied accordingly to the length of the stroke for each flow measurement.

$$u_{rate} = \sqrt{{u_{Type\ A}}^2 + {u_{GB}}^2 + {u_{linearity}}^2 + {u_{time}}^2} \qquad (5)$$

Note: Though Fluke Calibration is not accredited in dimensional measurements, it is viable to use an uncertainty in a test such as this as long as the length measurements are not offered to the public and the uncertainties are considered properly.

The uncertainty in effective area of the piston-cylinder is ±0.001% expanded to 95% confidence.

The final combined uncertainty of the piston gauge flow reference test can be calculated as:

$$u_{qm} = \sqrt{{u_{Type\ A}}^2 + {u_{\rho gas}}^2 + {u_{rate}}^2 + {u_{Aeff}}^2} + u_{drate} \qquad (6)$$

Table 1 shows an uncertainty budget for the influences described above in sccm. The expansion is using a coverage of 2 since the final effective degrees of freedom supported this for an approximate 95% confidence.

Table 1. Uncertainty budget for piston gauge flow test.

	Nominal Flow Points and Standard Uncertainties (sccm)				
Flow Rate	0.06	0.3	0.5	0.7	0.9
Type A	8.9E-05	1.1E-04	1.7E-04	2.4E-04	1.3E-03
fall rate Type A	3.5E-05	1.8E-04	2.9E-04	4.1E-04	5.3E-04
gage block	1.8E-05	9.0E-05	1.5E-04	2.1E-04	2.7E-04
laser linearity	1.8E-04	2.3E-04	2.5E-04	2.6E-04	3.4E-04
gas density	2.2E-05	1.1E-04	1.9E-04	2.6E-04	3.3E-04
effective area	3.0E-07	1.5E-06	2.5E-06	3.5E-06	4.5E-06
drop rate correction	9.5E-05	9.5E-05	9.5E-05	9.5E-05	9.5E-05
Combined	3.0E-04	4.3E-04	5.8E-04	7.3E-04	1.6E-03
Expanded	6.0E-04	8.7E-04	1.2E-03	1.5E-03	3.2E-03
Expanded (%)	1.0%	0.29%	0.23%	0.21%	0.36%

In Table 1 the uncertainty in time is not shown since it is an insignificant component to the rate uncertainty. Also not shown is the uncertainty budget for the piston-cylinder drop rate on its own, however the expanded result is included in the last row as a correlated uncertainty. Figure 7 shows the average flows with the expanded uncertainty represented by the error bars.

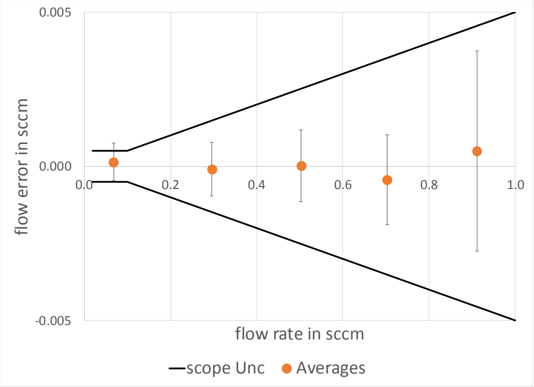

Figure 7. Final results shown with error bars representing measurement uncertainty expanded to 95% confidence.

4. CONCLUSION

In order to support this range in gas flow for Fluke Calibrations scope of accreditation a method needed to be designed to gain confidence as is required for accreditation. It is possible that without any resources a laboratory might choose rate of pressure rise or decay to fulfil the requirement. With the availability of high performance piston gauges there was a strong desire to implement them as described in this paper.

Though the triangulating laser had many desirable traits including excellent precision with small changes in displacement, it is clear that an integrated laser in the piston gauge under vacuum would make for a much better standard for flow. However, the results were good enough to validate the traceability claimed in the laboratory's scope of accreditation.

Not discussed here is the fact that other piston-cylinder sizes could be used for better uncertainty in lower or higher ranges. A 50 mm diameter piston-cylinder with a controlled clearance pressure to adjust the gap smaller could go a long way to reduce the uncertainty of the drop rate. Higher flow rates could be taken due to the larger diameter. Also not presented in this paper was preliminary work with a 16 mm diameter piston-cylinder which was marginally more precise at the lower flows. Considering these aspects, it may be possible to design a piston gauge flow standard to cover a broader range.

And finally, in the original concept of this project, it would be possible to use a second piston gauge upstream of the LFE. The process for setting a flow would be much quicker and simpler by just setting the differential pressure using the upstream piston gauge relative to the downstream piston gauge, gaining ultimate flow stability through the LFE due to the piston gauges ability to control pressure.

5. ACKNOWLEDGMENTS

The author would like to acknowledge Pierre Delajoud, the designer of both the piston gauge and the LFE used and for the suggestion of the method 26 years ago. Also acknowledged here is Casey Rombouts, Fluke Calibration Flow Metrologist, for the use of his LFE flow reference and guidance on system connections and general flow metrology.

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