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A HIGH ACCURACY, PORTABLE CALIBRATION STANDARD FOR LOW MASS FLOW

by

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ABSTRACT

The devices used to control and measure low mass flows of gases in many critical industrial processes must be calibrated regularly. The systems traditionally used for calibration have low response time, limited performance at very low flow and are not readily portable. A mass flow calibration system has been developed to provide an easy to use, compact and rugged, high accuracy calibration source. It is founded on well established laminar flow theory and the known thermodynamic properties of gases. A new design of laminar flow element combined with today's modern materials, sensor, mathematical modeling and data processing technologies results in new levels of performance for laminar flow element based measurements. The system is calibrated using a gravimetric technique and a complete uncertainty analysis has been performed. Over the past several years, numerous intercomparisons with other standards have been made.

KEY WORDS: Mass flow calibration

1. MASS FLOW CALIBRATION NEEDS

1.1 Mass Flow Measurement In Industry

The measurement and control of the low mass flow of gases (<1 sccm to 30 slm) is critical in many industrial processes, particularly semiconductor manufacturing where yield and product quality are dependent on the accurate introduction of small quantities of highly reactive gases at certain points in the production process. For the instruments used to measure and control mass flow to meet their performance potential and to conform with formal quality assurance requirements, they must be verified and/or calibrated regularly. A typical thermal mass flow controller (MFC) has an accuracy specification of ± 1 % of full scale so, following the conventional 4:1 guideline, a standard of at least ± 0.25 % is needed to calibrate it.

1.2 Traditional Mass Flow Calibration Practices

Traditionally, the only calibration methods able to provide the necessary accuracy to calibrate MFCs in a reasonable amount of time have been based on the measurement of displaced volume or of pressure change in a fixed volume. These methods do not provide real time measurement, limited performance at very low flow rates and relatively high capital and maintenance costs. In addition, they tend to be voluminous and delicate making it very difficult to transport them to establish the coherence of measurements made at different locations. This situation has led to the widespread use of "golden" MFCs to check other MFCs, a one to one comparison that violates the most basic principles of good metrology.

Clearly, there is a need for a low mass flow calibration standard that is highly stable over time, has fast response, good performance at very low flows and is small in size with the ruggedness to be truly portable.

2. SELECTING THE LAMINAR FLOW ELEMENT AS THE BASIS FOR A CALIBRATOR

Studying the requirements for a mass flow calibration system, it becomes apparent that the application of traditional laminar flow theory offers interesting possibilities. The flow elements can be purely mechanical and very stable, they can be of a relatively small size, response time should be limited only by pressure measurement speed and, theoretically, very low flow ranges can be covered. Laminar flow measuring techniques are known to be very linear and the rangeability is excellent since flow is directly proportional to differential pressure. However, the accuracy of around ± 1 % typically associated with laminar flow elements is far from the ± 0.2 % needed to meet today's requirements for a calibration standard.

Investigating the traditional application of laminar flow techniques shows that accuracy has been limited primarily by the poor knowledge of pressure, poor knowledge of gas temperature, failure to account for temperature and pressure effects on the laminar element dimensions, lack of basic stability of the element itself and failure or inability to apply the full flow equation real time taking into consideration the actual conditions of the flowing gas, in particular the effects of pressure and temperature on compressibility and viscosity. Studying the limitations just mentioned and finding solutions for them has allowed the development of a mass flow calibration system that meets today's requirements for accuracy, speed and low flow performance in a compact, rugged and easy to use package.

3. THE CRITICAL MEASUREMENTS: PRESSURE AND TEMPERATURE

3.1 Pressure Measurement

The accuracy with which the mass flow of a gas in laminar flow can be calculated depends upon knowledge of the pressure drop across the laminar element as well as the absolute pressure of the flowing gas to determine its actual compressibility and density. To achieve accuracy on flow on the order of ± 0.2 %, the accuracy of the pressure measurements needs to be about ten times better or ± 0.02 %.

The static pressure range to be covered is set by the application since the devices to be tested are usually operated between 100 and 300 kPa absolute. In order to get a low enough uncertainty on differential pressure and to have the desired rangeability of 10:1, it was determined that a differential pressure range of at least 50 kPa would need to be used. Such large differentials will cause relatively large adiabatic temperature changes in the gas but a solution to that problem would have to be found to avoid limiting the accuracy due to pressure measurement uncertainties.

3.1.1 The Pressure Sensors

The sensors selected for pressure measurement are based on the measurement of the change in the natural oscillating frequency of a quartz crystal resonator with changes in the stress applied by pressure through a miniature, precision bellows. A second quartz element not subjected to pressure stresses is used to measure temperature independently of pressure. Both quartz elements are sealed in an evacuated cavity. Calibration determines the relationship between oscillating frequency, pressure and temperature. In operation, a microprocessor applies the coefficients determined by calibration to derive pressure from the two frequency readings. Very extensive experience with these pressure transducers in pressure calibration products has established that, typically, they provide useable resolution on the order of 1 ppm of full scale, zero stability better than 100 ppm/year and span stability better than 50 ppm/year.

Two pressure sensors of a 0 to 400 kPa absolute range are used. There are several advantages to using two absolute sensors rather than one absolute and one differential as is frequently done. The basic sensor technology is inherently absolute since the quartz crystal is in a vacuum; using two sensors of the same range allows them to be regularly compared against each other to check for drift; using the average of two completely independent upstream and downstream measurements reduces the uncertainty on the measurement of the gas's absolute pressure. From a more practical standpoint, not using a low range differential sensor avoids the risk of overpressuring it when pressurizing the system and it is easier to calibrate two identical 300 kPa sensors simultaneously than to calibrate one 300 kPa absolute and one 50 kPa differential at various static pressures. Whenever static pressure is changed, the two sensors are compared at zero flow (thus zero differential pressure) and the difference observed in their readings is used to tare the subsequent readings so that the differential measurements are not subject to the full scale zero and span errors of the two sensors.

3.1.2 Where Pressure is Measured

The upstream and downstream pressures are picked off from pressure equalization chambers (Fig. 1) at either end of the active laminar flow path. Each chamber is connected by small diameter tubing to the two pressure transducers which are located in a separate support unit.

Laminar Flow Element

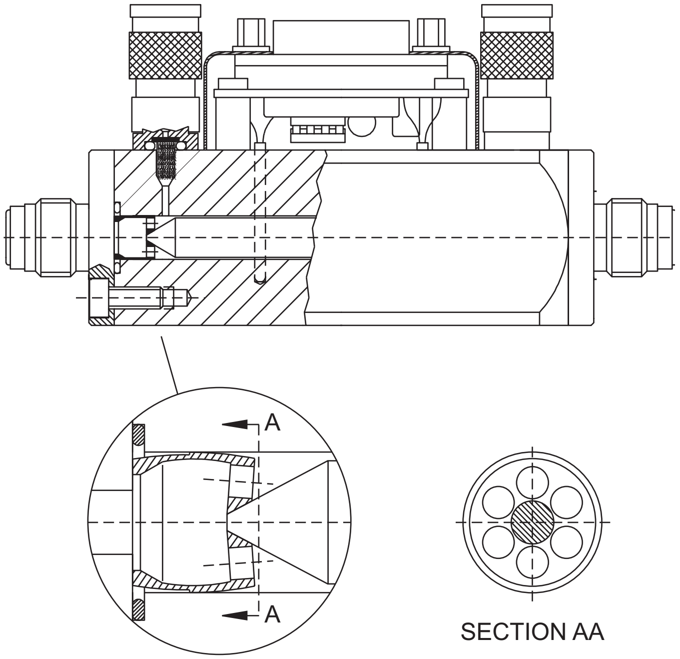

Fig. 1 *Cutaway of laminar flow element with an annular flow path defined by a piston in a cylinder. Piston holders act as secondary laminar elements and define pressure stabilization chambers. Two PRT's are mounted symmetrically in the cylinder body along the flow path.*

3.2 Temperature Measurement

Temperature affects gas density, compressibility and dynamic viscosity, all of which must be known to calculate the mass flow of a gas through a laminar element. The relative change in density with temperature is the temperature change divided by the absolute temperature which around ambient temperature (293 K) gives 0.35 %/K. The effect on dynamic viscosity, for typical calibration gases is 0.3 %/K. Both effects are of the same sign giving a total effect of temperature on density and viscosity of 0.65 %/K. Thus, in order to make ± 0.2% mass flow measurements, the temperature of the gas as it goes through the element must be known with accuracy better than ± 0.1 K.

The traditional method of determining the gas temperature is by installing temperature probes in the gas flow, usually just upstream of the element. This technique is not adequate to meet the ± 0.1 K requirement due to the slow response time of a temperature probe and considering the relatively large gas temperature changes that are likely to occur in the element itself as the gas expands with the pressure drop of up to 50 kPa. A different approach is needed.

3.2.1 Controlling Gas Temperature by Thermal Mass Ratio

In order to know the gas temperature accurately it is necessary to create conditions for an isothermal gas expansion and to find a way to know the temperature value of that isotherm. One solution is to force the temperature of the gas to that of a larger, more stable thermal mass whose temperature can be readily measured. An ideal flow path to apply this principle is a longitudinal annular space of relatively great length. Indeed, an annular flow path provides a very large surface of exchange and a low Reynolds number for the flowing gas relative to an equivalent tube shape. Increasing the length provides more time, at a given gas velocity, for heat exchange to occur and increases differential pressure. By calculation, to achieve a nominal flow range of 1 slm of nitrogen at a static pressure of 200 kPa and a differential pressure of 50 kPa, for a bore of 8 mm diameter, an annular space of 43 microns for a length of 60 mm is needed.

3.2.2 Practical Implementation of a Longitudinal, Annular Flow Path

A convenient way of defining a longitudinal annular passage is by centering a piston in a cylinder. Techniques for producing and characterizing piston-cylinders with excellent geometry and small clearances are well known from high accuracy deadweight testers. The design of the laminar flow element (Fig. 1) uses a piston in a cylindrical bore. The piston is held in place and centered by two holders that also act as secondary laminar flow elements. The bore is in a block whose thermal mass is very large relative to the thin film of gas in the space between the piston and the cylinder. Two platinum resistance thermometers are mounted symmetrically to measure the average temperature of the block along the working part of the passage and to average a gradient in the body should one be present due to the influence of an unevenly distributed outside temperature source. The two PRT's are measured in series and read using electronics located in a separate support unit. The electronics are self calibrating using two reference resistors.

4. ELEMENT STABILITY

The stability over time of the laminar flow element depends upon the mechanical stability of the elements that define the longitudinal annular flow path. All the mechanical elements are made of low carbon stainless steel. The active surfaces are lapped and electropolish finished to an Ra of better than 0.15 microns.

The greatest risk of instability comes from possible movement of the piston in the cylinder. Since flow is a cubic function of the space, very small changes in piston position would have very large effects on the flow coefficients of the element. For this reason great attention was paid to devising a means of simultaneously centering the piston in the cylinder and minimizing the possibility of movement after assembly. This was accomplished using a specially designed holder (Fig. 1). The holder is cylindrical and of a diameter slightly smaller than the cylinder bore. At the axis of the holder is a concentric centering hole. The coned end of the piston fits into the centering hole. The overall length of the piston and two holders is slightly greater than the cylinder bore length. Mounting the end flanges and torquing the screws that hold them to the cylinder block until the flanges meet the block generates controlled longitudinal forces that cause the holders to elastically expand which symmetrically engages the walls of the bore and precisely positions and holds the piston concentrically in the bore. So long as the longitudinal forces are maintained, i.e. the flanges are installed, the piston cannot move.

5. REAL TIME CALCULATION OF THE COMPLETE FLOW EQUATION TAKING INTO ACCOUNT ALL VARIABLES

5.1 The Mass Flow Equation

Following Poiseuille's law, the mass flow of a compressible fluid in laminar flow through an annular pathway can be expressed as:

$$qm = \frac{(P_1 - P_2) \bullet \rho_{(P,T)} \bullet \pi \bullet R \bullet h^3}{\eta_{(P,T)} \bullet 6 \bullet L} \qquad (1)$$

where:

qm = mass flow $[kg \cdot s^{-1}]$
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 $[K]$
$\rho_{(P,T)}$ = gas density under P,T conditions $[kg \cdot m^{-3}]$
R = flow passage radius $[m]$
h = gap between piston and cylinder $[m]$
η = dynamic gas viscosity $[Pa \cdot s]$
L = length of laminar flow passage $[m]$

Knowing that:

$$\rho_{(P,T)} = \rho_N \cdot \frac{P \cdot T_N \cdot Z_N}{P_N \cdot T \cdot Z_{(P,T)}} \qquad (2)$$

where:

ρ_N = standard gas density $[kg \cdot m^{-3}]$
P_N = standard pressure $[101.325\ Pa]$
Z_N = gas compressibility factor under standard conditions $[-]$
$Z_{(P,T)}$ = gas compressibility factor under P,T conditions $[-]$

by defining the dimensional constant that determines the range of the laminar flow element:

$$C_D = \frac{\pi \cdot R \cdot h^3}{6L} \qquad (3)$$

which becomes the geometrical constant, C_G, by calibration; we have the equation used to calculate mass flow through the laminar element:

$$qm = \frac{P \cdot (P_1 - P_2) \cdot \rho_N \cdot T_N \cdot Z_N}{T \cdot Z_{(P,T)} \cdot \eta_{(P,T)} \cdot \rho_N} \cdot C_G \qquad (4)$$

5.2 Handling the Mass Flow Equation

The laminar flow element is associated with a support unit that contains the two absolute pressure sensors and the electronics to read the element's PRTs. The support unit is driven by a powerful microprocessor that has the capability to rapidly solve equation (4) using imbedded algorithms, the calibration constants of the laminar element, measured pressure and temperature data and stored information on the characteristics of various gases.

Referring to equation (4) and identifying the variables: P_1, P_2 and T are available roughly once per second by direct measurement using the pressure transducers and the element's PRTs; P is the average of P_1 and P_2; ρ_N, Z_N, $Z_{(P,T)}$, $\eta_{(P,T)}$ are known properties of gases [1] stored in non-volatile memory; T_N and P_N are defined standard conditions; C_G and its Reynolds number dependence are determined by calibration as described immediately below. Also taken into consideration in C_G are the change in the element geometry and dimensions with pressure and temperature relative to reference conditions using P and T. How to compensate for these influences by calculation is well known from experience with the piston-cylinders of high accuracy piston gauges.

6. CALIBRATION

Referring to equations (1), (3) and (4) above, calibration of the laminar flow element consists of determining C_G as compared to C_D. Of course, the pressure transducers and the element's PRTs also need to be calibrated but for the accuracy levels required these are routine and thus not further described here.

One reason for the difference between C_D and C_G is that laminar flow theory is not precise enough to assure the desired level of accuracy. This makes it necessary to refine the value of C_D by direct measurements and to modelize the flow element as a function of the Reynolds number of the mass flow. Reynolds number is used in the modelization rather than mass flow because Reynolds number contains the effect of gas viscosity making Reynolds number based compensation valid for different gases. Reynolds number also has the advantage of taking into account gas velocity in the space. The difference between C_D and the C_G determined by calibration also reflects the differences between the ideal dimensions and geometry from which C_D was calculated and the actual dimensions and geometry of the manufactured parts.

6.1 Selection of a Calibration Method

To determine C_G, a fundamental method of measuring mass flow with very high accuracy is needed. Three techniques are commonly used by flow metrologists: the measurement of displaced volume, the measurement of pressure change in a fixed volume and the direct measurement of collected or lost mass. The mass measurement method, which is frequently called gravimetric, was selected. This method, the only one which is truly primary, has the great advantage of working directly with the fundamental units of mass and time from which mass flow is derived. It is not dependent, as the other methods are, on difficult gas pressure and average temperature measurements and imperfect knowledge of the gas's thermodynamic properties.

6.2 The Calibration Procedure

In the gravimetric procedure used, the flow element is connected to a volume with two-stage regulation and an isolation valve upstream and a metering valve downstream (Fig. 2). The flow is set to the rate to be tested using the metering valve. The volume assembly is then removed, filled with the necessary mass amount of the calibration gas, and weighed, taking into account all air buoyancy, pressure deformation and temperature effects.The volume assembly is then reconnected. The isolation valve is opened allowing flow through the element for a specified amount of time and then closed. The volume assembly is reweighed to determine the change in mass, ΔM.

During the time that gas was flowing, every two seconds the computer calculated a current mass flow value following equation (4) using C_D and current pressure and temperature measurements. The total mass that was flowed can be derived by integrating those individual mass flow measurements over time. A comparison of the calculated total mass, m, flowed through the molbloc with the measured mass change, ΔM, of the volume is performed. The process is repeated several times from 10% and up to 100% of the maximum flow rating of the element. C_G is the constant term of the linear fit of the data calculated using equation (5) as a function of Reynolds number for the different calibrated flow rates and the first order term represents the Reynolds number dependency. The relationship between C_D and C_G is:

$$C_G = C_D \left(\frac{\Delta M}{m} \right) \tag{5}$$

Where:

C_G = measured element-specific geometrical constant $[m^3]$

C_D = theoretical dimensional constant [101.325 Pa]

ΔM = measured loss of mass in the volume [-]

m = calculated mass of gas through under P,T conditions [kg]

The gas normally used for the calibration is nitrogen. Theoretically, the value of C_G determined with nitrogen should be valid for any other gas whose characteristics are well known. In practice, due to limitations in laminar flow theory, varying uncertainties on the knowledge of gas characteristics and laminar element specific effects, for best accuracy with other gases, particularly poorly known or far from ideal gases, it is necessary to calibrate with that specific gas. The value, or values, of C_G and the Reynolds number dependencies of several gases can be and are recorded on the laminar element's EEPROM from which they are retrieved by the support unit when needed. The uncertainty on the term C_G determined by calibration is dependent on the errors introduced in the direct mass measurement and the measurement of elapsed time. Typically these are estimated, 2σ, at ± 0.035 % on ΔM and ± 0.007 % on elapsed time. A detailed uncertainty analysis is available [2].

7. PRACTICAL IMPLEMENTATION

7.1 The Laminar Elements and Support Unit

The laminar flow element and its support unit are available commercially as a low mass flow calibration system. There are eight laminar flow element ranges between 10 sccm full scale and 30 slm full scale, based on nitrogen flow at 200 kPa static pressure and 50 kPa differential pressure. The different ranges are achieved by varying the piston size in a common bore to change the space between the piston and the cylinder. The two highest ranges use a larger cylinder block and bore to maintain a Reynolds number of the flowing gas under 1 200. The support unit is compact (12 x 32 x 30 cm). It is accessed through a front panel keypad and display or over standard digital interfaces. It contains the pressure sensors, the electronics needed to measure temperature from the PRTs, the microprocessor to drive the system and extensive non-volatile memory for

Gravimetric Calibration Setup

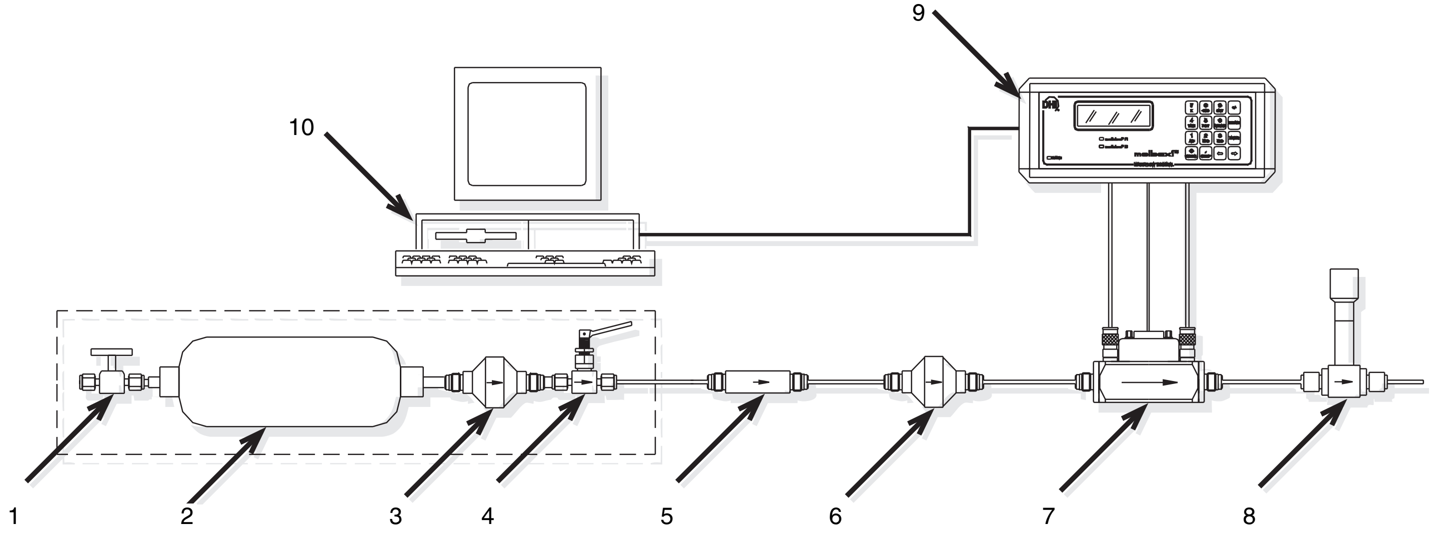

Fig. 5 Gravimetric Calibration Set-Up: 1. inlet valve, 2. volume, 3. first stage regulator, 4. shut-off valve, 5. high purity filter, 6. second stage regulator, 7. flow element being calibrated, 8. metering valve, 9. support unit for reading temperature and pressures, 10. computer

storage of gas characteristics. Currently, 16 different gases are included. The support unit also has analog input and output capability for reading back from a device under test and sending set point commands if applicable. Off the shelf software is available to interface with the system and allow fully automated calibration using a personal computer.

7.2 Specifications

The commercially available system provides the performance necessary to characterize today's modern analog and digital mass flow measuring and controlling devices and to act as a means of comparing high precision fundamental measurements made at different locations. The two sigma accuracy claim including the uncertainty on the absolute calibration is ± 0.2 % of reading from 10 to 100 % of full scale and ± 0.02 % F.S. below 10 % of the range. One year reproducibility is ± 0.1 % of reading or ± 0.01 % F.S., whichever is greater. Response time is less than two seconds allowing very rapid measurements relative to other calibration techniques.

8. INTERCOMPARISONS

Over the past several years, many intercomparisons using the commercially available unit have been performed with different types of standards including volumetric, rate of rise and gravimetrically based systems operated by different organizations. In general, these have shown very good agreement even using a variety of gases. On a number of occasions, the high speed and repeatability of the laminar element system has revealed inconsistencies between systems which their operators had not previously been able to detect. Three examples of intercomparisons made include:

In early 1992, comparisons with two commercially available volumetric systems were performed at Intel Corporation's mass flow laboratory in Rio Rancho, New Mexico. Helium, argon and nitrogen gases were flowed between 500 sccm and 15 slm. The data provided independent confirmation that the modelization using Reynolds number dependency described above was valid.

In the fall of 1993, Oak Ridge National Laboratory's Instrumentation and Controls Division, to qualify laminar element systems as part of the establishment of an MFC Development and Test Facility, performed a comparison with their volumetric standard in the 1 slm range using nitrogen. The agreement was inside the combined accuracy claims of the two systems and the laminar element systems was subsequently used in MFC accuracy testing.

Most recently, an intercomparison with France's Laboratoire National d'Essais (LNE) was completed. This laboratory has implemented gravimetric standards with real time weighing techniques. A laminar element system was calibrated in Arizona using the gravimetric system described above and then shipped to France. Several months later, it was compared with the LNE standards using nitrogen at flow rates between 500 sccm and 5 slm. All the points taken agreed well within the combined accuracy tolerances of the two systems of ± 0.3%.

A future publication will provide further information on intercomparisons and their results.

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