Presented 1996 August 27
at the
1996 NCSL Workshop and Symposium
Monterey CA

THE NEED FOR EVOLUTION IN STANDARDS AND CALIBRATION TO IMPROVE PROCESS MEASUREMENT AND CONTROL OF LOW MASS FLOW

by

Pierre Delajoud and Martin Girard

1996 August

DH Instruments, Inc.
1905 W. Third St.
Tempe, AZ 85281-2490
USA
Tel 602/967-1555
Fax 602/968-3574
dhi@dhctx.com
www.dhinstruments.com

DHI
Calibration Solutions for Pressure and Flow™

In Europe:
CalTechnix S.A.
120, av. Charles de Gaulle
92200 Neuilly sur Seine
FRANCE
Tel 1.46.40.37.70
Fax 1.46.40.37.71
caltechnix@compuserve.com
www.caltechnix.com

THE NEED FOR EVOLUTION IN STANDARDS AND CALIBRATION TO IMPROVE PROCESS MEASUREMENT AND CONTROL OF LOW MASS FLOW

AUTHORS

Pierre Delajoud
CalTechnix S.A.
120, av. Charles de Gaulle
92200 Neuilly sur Seine
FRANCE

Martin Girard
DH Instruments, Inc.
1905 W. Third St.
Tempe, AZ 85281
USA

ABSTRACT

Improvements in mass flow measurement and control are critical to progress in a number of industrial processes. For these improvements to occur, the metrological system that supports process instruments must evolve to meet new requirements. The current mass flow metrological support system differs considerably from the conventional structure found in other widely used measurement disciplines. The main differences have developed in reaction to the lack of transfer standards and have created a situation in which it is difficult for significant improvements to occur. The introduction of new laminar flow based transfer standards offers the possibility of the mass flow metrological support system moving towards a more conventional structure and the benefits it offers.

KEY WORDS: Mass flow, gas flow, primary standard, transfer standard, secondary standard, calibration

1. INTRODUCTION

The precise measurement and control of the low mass flow of gases is critical to a number of industrial processes, particularly semiconductor manufacturing. In this field, improving mass flow measurement and control is identified as key to greater yield and indispensable to next generation production processes. The requirements include very low flows (< 1 sccm) and the measurement of a wide variety of gases including heavy, non-ideal gases.

In seeking to meet the requirements for improved process flow measurement and control, the obvious first place to look is the process instruments themselves. Perhaps less obvious, but equally important, is the metrological support system for the measurements made by the process instruments. These calibration and verification tools and methods ultimately underlie the quality of all process measurements. No matter how good the basic performance of the process instruments, their potential benefits cannot be realized if adequate means to assure the accuracy of the original calibration, field verification and periodic recalibration, are not available.

Therefore, any meaningful effort to improve process mass flow measurement and control must include careful consideration of the calibration and standards issue.

2. CALIBRATION, ACCURACY AND PRIMARY STANDARDS

The typical calibration or verification process consists of determining the correlation of an instrument under test with a standard. In a verification, the correlative information will usually be compared to limits to determine whether the test instrument is "good" or "bad". In a calibration, the information will be used as the basis for adjusting the instrument under test to improve the correlation and thus improve test instrument accuracy. In both cases, the role of the standard is to provide accurate values of the quantity being measured.

The simplest definition of *accuracy* is maximum deviation from the true value; the degree to which an instrument's indication of the numerical value of the quantity it is measuring can be expected to deviate from the true numerical value of that quantity. The true value is an unknown ideal never realized in the real world. It is, however, very explicitly defined. In the 100+ countries that are signatories to the Treaty of the Meter, the true value is defined in terms of the International Bureau of Weights and Measures' (BIPM) International System of Units (SI) seven base units. In SI, mass flow is a quantity of mass per quantity of time ($M \bullet T^{-1}$) and is derived from the base unit of mass, the kilogram, and the base unit of time, the second ($kg \bullet s^{-1}$) [1]. Thus, any and all *accurate* mass flow measurements must ultimately have been derived from the base units of mass and time. This connection between SI true values and day to day measurement is usually called *traceability*. Note: The unfortunate tradition of using volumetrically based units,

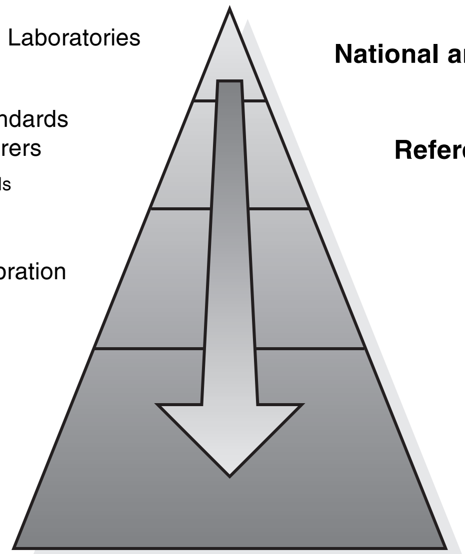

Fig. 1 *"Pressure Rate of Rise" Mass Flow Standard*

such as sccm, to define mass flow leads to confusion between volume flow and mass flow. The "s" in sccm refers to standard conditions so a "standard" volume of gas really defines a mass of gas, that which occupies the volume *under standard conditions*. Mass flow, mass over time, does not need defined reference conditions.

Primary standards are the methods and associated instrumentation that allow the physical embodiment of the SI unit definitions into tangible quantities. They are the starting point for accurate measurements in the real world. Primary standards for the seven base units apply the unit's fundamental definition. Primary standards for derived units derive their measurement directly from the defining base units. A primary standard for mass flow ($kg \cdot s^{-1}$), must provide a method for deriving mass flow directly from its base units: mass (kg) and time (s).

3. CONVENTIONAL METROLOGICAL SUPPORT OF A UNIT OF MEASURE

The conventional support system for the practical use of a unit of measure in the worlds of science and industry generally follows a common structure designed to assure that the benefits of the standardization provided by the BIPM and SI are maximized. These benefits are: a) the maintenance of a single, coherent system of measurement for a wide variety of units and b) global consistency of measurements.

The support system starts at the basic definition of the unit using primary standards. As this process tends to be difficult, time consuming and of use to a narrow market, it is normally performed by a limited number of highly qualified facilities where the time and expertise necessary can be made available. Recognizing this situation and the importance of a sound measurement foundation to the national interest, most countries use public resources to fund a national measurement laboratory, such as NIST in the United States, whose duties include this function.

The primary values, painstakingly derived by specialists, are transferred to the measurement community at large by a transfer system made up of a variety of facilities, procedures and instruments. These assure the link between a relatively small number of very high accuracy primary measurements and the nearly infinite number of lower accuracy day to day measurements made in science and industry. The instruments used to make the transfers are frequently called *transfer or secondary standards*. Unlike primary standards, whose most important characteristics are their primary nature and the minimization of absolute uncertainties with secondary regard for usability or cost issues, the most important characteristics for transfer standards are precision, stability over time, compatibility with the application and cost.

The conventional metrological support structure tends to be well developed for units of measure that are very widely used and/or whose measurement is particularly critical.

An instructive example is the base unit of length, the meter. The SI definition of the meter is the length of the path traveled by light in vacuum during a very small fraction of a second. Researchers at NIST and other national laboratories conduct research using primary standards to physically embody this definition. Then a very extensive multi-level system of transfers assures the conveyance of these values to support everything from sub-micron parts inspection to school children's rulers.

An example for a derived unit is pressure. Pressure, a force over an area, is derived from the base units of mass, length and time. Two primary techniques are available and include the deadweight tester or piston gauge and the liquid manometer. Again, a relatively small number of highly specialized facilities use these in a primary manner to arrive at absolute definitions of pressure values. Again, a very extensive multi-level system of transfers exists to support a wide range of practical measurement requirements.

Length and pressure are both very widely measured quantities whose accuracy is critical in a multitude of applications. For both of them today, an advanced metrological support structure exists to meet the most demanding real world needs. This structure is founded on two complementary and indispensable elements: on one-hand, the continually improving elaboration of very high accuracy reference values by specialists using primary standards; on the other, the availability of an extensive, highly efficient system for transferring those values.

4. CONVENTIONAL METROLOGICAL SUPPORT FOR THE LOW MASS FLOW OF GASES

In the past few of years, the need for increased accuracy in low mass flow measurement and control at the process level has drawn a good deal of attention to mass flow process instruments and to their metrological support.

Taking a look at the metrological support system for the low mass flow of gases which has been prevalent, it is immediately apparent that it differs from the conventional structure just described for length and pressure.

The major difference is that transfer standards in the traditional sense of the term are almost completely missing. A transfer standard normally means a fast and easy to use instrument of significantly greater precision and stability over time than the device to be calibrated. In the world of low mass flow, no such instrument has been available. Specially calibrated or "golden" process instruments have been used to meet the practical need for fast and easy to use calibration sources but these generally do not provide an adequate ratio of precision and stability over time with the test instrument to meet the customary metrological requirements of a *transfer standard*. In this context, the *transfer standard* is no longer recognized for the beneficial role that it plays in the conventional metrological support system but rather, it has become synonymous with the practice of using a process instrument to calibrate or verify a like process instrument. Lack of confidence in this practice has led a number of end users to insist that their process instruments be calibrated only with "primary" standards, a requirement that runs counter to the principles of the conventional metrological support structure and would make no sense in most other measurement disciplines.

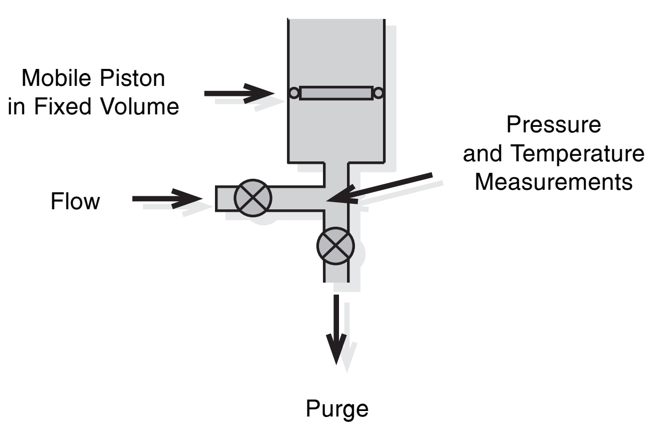

Fig. 2 *"Volumetric" Mass Flow Standard*

As the role of primary standards and transfer standards are intimately linked in the conventional metrological support structure, it is not surprising that the lack of transfer standards for low mass flow has had a profound impact on the development and use of primary standards.

Only one method is available for directly deriving mass flow from the base units of mass and time. That is to directly measure the mass of gas flowed over a period of time. This *gravimetric* method, since it measures mass directly, has the great advantage of independence from knowledge of a gas's thermodynamic properties and the actual conditions of the flowing gas. Ultimately, in exact agreement with the definition of a primary standard, its uncertainty is dependent only on the direct measurement of the base units.

Other more indirect, though fundamental, methods of determining mass flow exist. These techniques, though generally designated "primary", do not derive mass flow directly from the base units of mass and time and, unlike the gravimetric technique, are dependent on gas pressure and temperature measurements and imperfect knowledge of gas thermodynamic properties. The most common are the *volumetric* and *rate of rise* methods.

In the *volumetric* method, gas is flowed into a known, variable volume, usually defined by a piston in a cylinder. With gas flowing into the volume under the piston, the time required to move the piston a certain distance is measured yielding a volume over time. The mass of gas in the volume is determined by deriving the gas density under actual conditions from the measured gas pressure and temperature and the gas's thermodynamic properties. Then, again depending on knowledge of the gas properties, gas density is brought back to standard conditions. Total mass over total time yields average mass flow. [2,3]

In the *rate of rise* method, gas is flowed into a known, fixed volume. The time required for the pressure of the gas to change a given amount is measured. Then using the volume, gas pressure and temperature and gas thermodynamic properties, the total mass of gas flowed over the period of time can be calculated.

A third fundamental or calculable method of determining mass flow is using *critical flow Venturi nozzles*. In this technique, the gas is passed through a contoured restriction in which the gas flow accelerates to the critical velocity at the throat (this being equal to the local sonic velocity). At the critical velocity the mass flow rate of the gas flowing through the Venturi nozzle is the maximum possible for the existing upstream conditions.[5] This technique, though very widespread for high flow rates, is not generally used for low flows due to the difficulty of manufacturing very small diameter nozzles and to their limited rangeability.

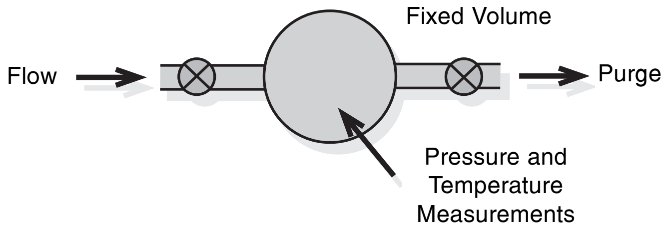

Fig. 3 *"Pressure Rate of Rise" Mass Flow Standard*

For reaching the objectives normally associated with the use of a primary standard --- derivation of reference values directly from the base units with the lowest uncertainties possible --- the gravimetric method is the obvious first choice. However, until recently, this method has been little used and the most common sources of reference values in low mass flow have been the first two indirect techniques (volumetric and rate of rise). This apparent inconsistency is the result of the lack of adequate transfer standards. In the absence of transfer standards, fundamental methods have had to be used to provide the day to day support of process instruments. In that context, the selection of the technique is very largely dependent on speed and ease of operation, features which are normally of secondary importance for primary standards. The gravimetric technique has clear advantages from the purely metrological standpoint. However, its operation is too intricate and time consuming to allow its use in the day to day calibration of process instruments and there have been no tools available to transfer the primary reference values it produces. Therefore, there has been little reason to use it at all.

The metrological support system for low mass flow, in response to the lack of adequate transfer standards, has evolved away from the conventional structure that is used so effectively to support practical measurements of other quantities such as length and pressure. Rather than primary work being performed by a limited number of specialists who select their methods and equipment with the overriding objective of minimizing uncertainty to generate centralized reference values that are passed on through efficient transfer methods, various fundamental techniques are used directly on a very widespread basis for day to day calibration. The selection of the fundamental technique is based primarily on speed and ease of use and the systems are characterized and used independently with little or no opportunity for coherence checks between them.

The traditional metrological support structure for low mass flow brings disadvantages today and severely limits prospects for improvement in the future. The indirect fundamental techniques currently in widespread use to do the job normally handled by transfer standards have major shortcomings in that role including size, tedious and slow operation and excessive uncertainties at low flows. Looking towards the future, there is little prospect for improvement as the existing fundamental techniques appear to offer little potential for reduced uncertainties when applied in the typical calibration environment and the inability to intercompare them makes it difficult to convincingly document coherence. In fact, in the current context, there is little motivation to make improvements in primary standards unless the standards can be used directly in day to day calibration.

5. LOOKING TOWARD THE FUTURE OF LOW MASS FLOW CALIBRATION

It is clear from studying the current metrological support structure for low mass flow that significant changes must occur to provide the support necessary for process mass flow measurement and control to improve. The key to this improvement is the development of adequate transfer standards for the flow range and gases needed.

In response to this situation, in the early 1990s, **DH Instruments** undertook the development of such a transfer standard. After studying the various alternatives, the *laminar flow* principle was selected as the technical basis of the transfer standard. Laminar flow, though not mentioned above, is a fourth fundamental method of making mass flow determinations. In this technique, the gas is flowed through a flow element under conditions that assure that the flow regime is laminar. Then, from knowledge of the element's geometry, differential pressure across the element and the flowing gas's temperature, pressure and thermodynamic characteristics, the mass flow through the element can be calculated.[6, 7]

Like other fundamental techniques, the *laminar flow* technique is dependent on knowledge of gas thermodynamic properties but, for use in the traditional role of a transfer standard, it has the great practical advantages of compact size, continuous reading, high rangeability and very high stability. Thanks to the application of modern technology and a number of design innovations that improve the knowledge of gas pressure and temperature [7], the new laminar flow based system provides one year reproducibility of better than ± 0.1 % of reading. This level of performance, combined with the system's practical advantages, give it the characteristics needed to fulfill the transfer standard role which has been missing.

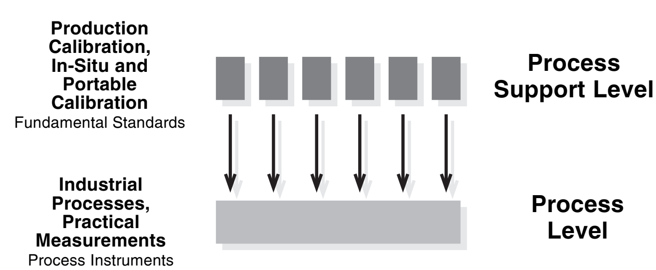

Fig. 4 *Conventional Metrological Support Structure for the Low Mass Flow of Gases*

Another advantage of the laminar flow system in the role of transfer standard is that its continuous reading characteristic allows it to be calibrated using the gravimetric technique. This technique provides greater accuracy in large part by eliminating uncertainties associated with lack of knowledge of specific gas characteristics that affect other systems. To fully exploit these advantages, a unique gravimetric calibration facility has been developed and dedicated to the calibration of the laminar flow elements. Significantly, in the same time period, a number of national measurement laboratories and other state of the art facilities have undertaken development of gravimetric standards.

6. CONCLUSION

The availability of a new laminar flow based transfer standard makes it possible for the mass flow metrological support system to move ahead towards a more conventional structure with the advantages that structure offers: improved primary reference values, greater efficiency in day to day calibrations and greater coherence of measurements at lower levels in the measurement system.

Primary standards can be used to fill their normal role. That is not to serve as day to day calibration tools but rather to provide the best possible reference values for widespread, coherent dissemination by transfer standards. This can release the potential of advanced, though impractical, primary techniques, such as gravimetric, that offer real promise for improving accuracy, especially with heavy and non-ideal gases. At the process instrument calibration level, the choice is no longer limited to one to one comparisons with like process instruments on one hand or fundamental methods that are poorly suited to day to day use on the other. The laminar flow system provides a true transfer or secondary standard, an instrument designed for speed and ease of use with accuracy based on high precision and stability over time supported by centralized, high quality primary measurements.

BIBLIOGRAPHY

[1] "The International System of Units", 6th Edition (1991), BIPM, Sevres, France.

[2] Alberti, F., Wang, C., "Gas Flowrate Calibration Using a Piston Prover and a Laser Doppler Displacement Meter", Proceedings of the NCSL Conference and Workshop, Chicago, IL, July, 1994.

[3] Korpi, D.M., "Automated Positive Displacement Methods Improve Primary Standards for Gas Flow Calibration", Proceedings of the NCSL Conference and Workshop, Washington, D.C., July, 1992.

[4] McCoubrey, A.O., "Guide for the Use of the International System of Units", NIST Special Publication 811, U.S. Government Printing Office, Washington, D.C., 1991.

[5] "Measurement of Gas Flow by Means of Critical Flow Venturi Nozzles", ISO Standard 9300:1990 (E), International Organization for Standardization, Geneva, Switzerland.

[6] Tison, S., "Development of a Laminar Flow Transfer Standard", Proceedings of the 25th Measurement Science Conference, January, 1996.

[7] Delajoud, P., Girard, M., "A High Accuracy, Portable Calibration Standard for Low Mass Flow", Proceedings of the XIII IMEKO World Congress of Metrology, September, 1994, Torino, Italy.

[8] Delajoud, P., Bridge, M., "Molbloc Uncertainty Analysis", Internal DH Instruments, Inc. quality assurance document, August, 1993, Tempe, AZ.

[9] Mattingly, G.E., "Gas Flowrate Metrology", NCSL Newsletter, Vol. 29, No. 1, January, 1987.

[10] Mattingly, G.E., "Gas Flow Measurement: Calibration Facilities and Fluid Metering Traceability at the National Bureau of Standards", Procs. Inst. for Gas Technology Confr. on National Gas Energy Measurement, Chicago, IL, April, 1986.

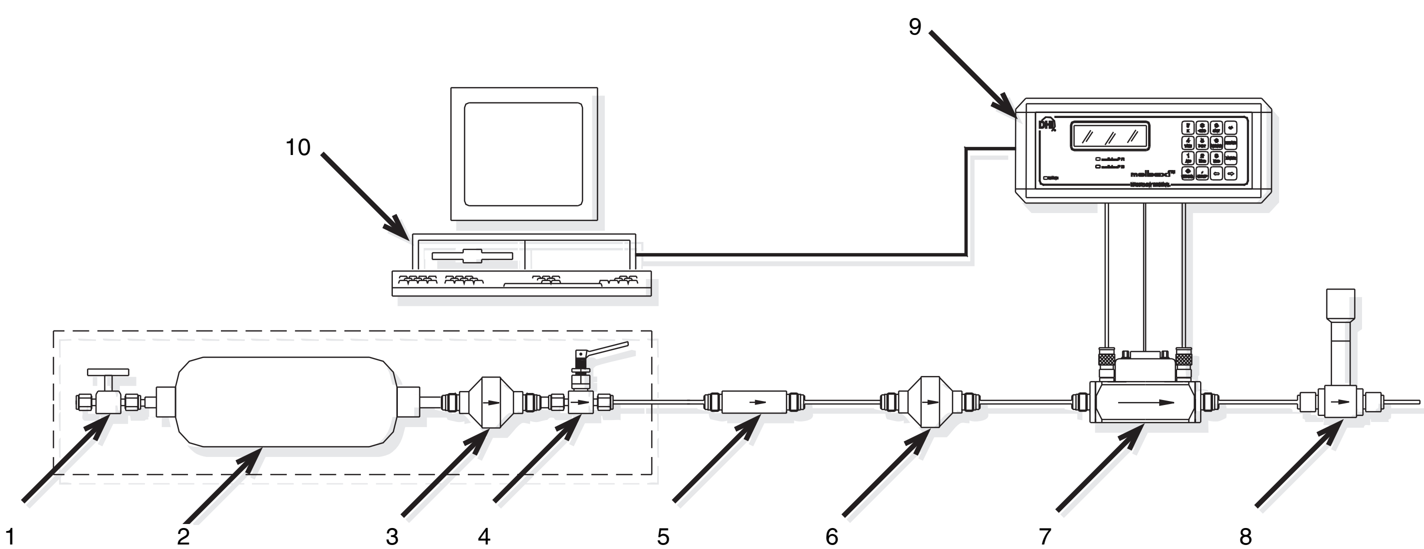

Fig. 5 *Gravimetric Calibration Set-Up: 1. inlet valve, 2. volume, 3. first stage regulator, 4. shut-off valve, 5. high purity filter, 6. second stage regulator, 7. flow element being calibrated, 8. metering valve, 9. support unit for reading temperature and pressures, 10. computer*