

Calibration

The Fundamentals of Gas Flow Calibration

Application Note



Introduction

Understanding the fundamentals of gas flow calibration is essential for evaluating calibration systems, estimating the magnitude of error sources, developing calibration methods and resolving measurement problems. This application note develops the ideal gas laws into tools that can be applied to real gases in a flow calibration system. In addition it discusses the two ways that flow can be measured and explains why one is preferred over the other. It also covers flow standards and provides guidelines for making reliable measurements.

Ideal gas laws

Gases are compressible. When a gas compresses its density increases, and this affects the volume flow rate. As a result, an accurate, direct comparison of volumetric gas flow requires accounting for gas density. To understand what factors affect gas density we turn to the ideal gas laws. See side bar on page 2.

Gas flow

There are two basic flow quantities that can be measured: mass flow, and volumetric flow. The volumetric flow rate is the volume of fluid that passes through an imaginary surface per unit time. Similarly, the mass flow rate is the mass of a substance that passes through a surface per unit time. These ideas can be expressed using a variety of units.

Unit	Description	Type of flow
ccm or cc/min	Cubic centimeters per minute	Volumetric
accm	Actual cubic centimeters per minute	Volumetric
sccm	Standard cubic centimeters	Volumetric
	per minute	(proportional to mass flow)
slm or slpm	Standard liters per minute	Volumetric
		(proportional to mass flow)
cfm or cf/min	Cubic feet per minute	Volumetric
scfm	Standard cubic feet	Volumetric
	per minute	(proportional to mass flow)
kg/sec	Kilogram per second	Mass
lb/hr	Pounds per hour	Mass

The volumetric flow rate is affected by pressure and temperature. A restriction in the plumbing can cause a pressure drop that leads to a change in flow rate. If a volume of gas is flowing across a restriction we can calculate the flow rate on the other side of the restriction using the gas laws.



Ideal gas laws

Boyles' law

Sir Robert Boyle, a seventeenth century scientist, stated that if temperature remains constant, the change in volume (V) of a given mass of gas is inversely proportional to the change in absolute pressure (P). This is known as Boyle's law.

Charles' law

French scientist Jacques Charles, building on the work done by Boyle, concerned himself mainly with the effects of temperature on a gas. He stated the change in volume of a fixed amount of gas is proportional to the change in absolute gas temperature. This is known as Charles' law.

Ideal gas law

The ideal gas law is a combination of these two laws. The ideal gas law is a good approximation for most gases under moderate temperature and pressure. However, the equation above neglects intermolecular effects leading to the compressibility observed in real gases.

n = Number of moles of gas in the volume V

- **mole** = the amount of substance of a system which contains as many elementary entities as there are atoms in 0.012 kg of carbon 12. Its experimentally determined value is approximately 6.022142×10²³.
 - **R**= Gas constant (different for different gases)
 - \mathbf{Ru} = Universal gas constant (same for every gas) =8.314472 J·K⁻¹·mol⁻¹
- **Joule** = Unit of energy $(J) = n \cdot m = Pa \cdot m^3$
 - **M**= Molecular weight of the gas in question
 - T = absolute temperature (Kelvin = Celsius +273.15)
 - **P**= absolute pressure



Figure 1a. Density (n/(V)) changes in a fixed volume are a result of changes in temperature and pressure

As a consequence of this law we know as temperature increases the number of molecules decreases if volume remains constant. Likewise, we know when volume is constant and pressure increases the number of molecules also increases.



Assuming there are no leaks then the number of particles, expressed by n, will stay constant. The relationship of the gas volume under the two sets of conditions is given by the ideal gas law as:

Solve for V₂

$$T_1 = T_2$$

 $V_2 = V_1 = \frac{P_1}{P_2} = \frac{T_2}{T_1}$

 $\frac{P_1V_1}{P_2V_2} = pP_2 = \frac{P_2V_2}{P_2V_2}$

Differentiate the volume with respect to time to get the equation for the volumetric flow rate.

$$q_{v2} = \frac{dV_2}{dt} = \frac{dV_1}{dt} \frac{P_1}{P_2} \frac{T_2}{T_1}$$
$$= q_{v1} \frac{P_1}{P_2} \frac{T_2}{T_1}$$

Calculate the new volumetric flow rate $q_{\rm v2}$. Note that absolute pressure and temperature values must be used in the calculations.

$$q_{v2}$$
 = 200 x $\frac{850}{425}$ x $\frac{293.15}{293.15}$ = 400 cc/min

The same equations can be used to reference a volumetric flow rate to standard atmospheric conditions. This is what is done in order to express flow rates in frequently used standardized flow units, like sccm. For example, if a the flow rate is 200 cc/min, the pressure is 850 kPa, and the temperature is 30 °C, we can determine what the equivalent flow rate would be under standard atmospheric conditions (i.e. T=20 °C, P=101.325 kPa).

$$q_{v2}(T_N, P_N) = q_v \frac{P}{P_N} \frac{T_N}{T}$$

 $q_v(T_{N'}, P_N)$ = volume flow rate referenced to standard atmospheric conditions

 $P_{\rm N}$ = standard pressure (absolute)

 T_{M}^{N} = standard temperature (absolute)

$$q_{v2}(T_{_N}, P_{_N}) = 200 \times \frac{850}{101.325} \times \frac{293.15}{303.15} = 1622 \text{ cc/min}$$

Actual volumetric flow rate = 200 cc/minStandard volumetric flow rate = 1622 sccm(at 1 atm and $20 ^{\circ}\text{C}$)









A common approach used in gas flow measurement is to use volumetric units referenced to standard atmospheric conditions. Examples of units include standard cubic centimeters per minute (sccm), standard liters per minute (slm) and standard cubic feet per minute (scfm). These measures can be used as a proxy for mass flow units, such as kilograms per second because the two will always be proportional to each other.

Be careful with standardized units. Although the standard reference temperature of 0 °C is common and internationally used, many devices and users reference 70 °F, especially in North America. If you assumed the reference temperature was 0 °C and the device was referenced to 70 °F, then your error would be 7.7 percent. Obviously this is a big problem. Other less common reference temperatures are 60 °F, 20 °C and 25 °C. For this reason, it is important to always state the reference temperature when standardized units are used. Luckily, standard pressure is always 1 ATM (101.325 kPa or 14.696 psi absolute).

Volumetric gas flow: real gases

So far, the equations above treat all gases as if they behave exactly the same. As stated before, the ideal gas law is a good approximation for most gases under moderate temperature and pressure. However, the ideal gas law neglects compressibility which is observed in real gases. Real gases as opposed to ideal gases differ from each other in terms of compressibility, and the compressibility of each real gas depends on pressure and temperature conditions. For example, the compressibility of natural gas is different from the compressibility of Argon and the compressibility of each gas depends on both pressure and temperature. As a result, to reference the flow rate of real gases to standard conditions, the compressibility needs to be taken into account.

$$q_v(T_N, P_N, Z_N) = q_v \frac{P}{P_N} \frac{T_N}{T} \frac{Z}{Z_N}$$

Z= compressibility of specified gas at actual conditions

 ${\rm Z}_{\rm N}{=}$ compressibility of specified gas at standard conditions

Mass flow

Mass flow (q_m) is related to volume flow (q_v) by the gas density (ρ) , which depends on temperature (T) and pressure (P).

$$q_m = q_v \rho$$

 $\rho = \frac{n}{V} \frac{P}{RT}$

For example, if the gas is oxygen with a molecular weight $M=32 \text{ g·mol}^{-1}$ and atmospheric conditions are P=850 kPa, and t=20 °C (T=293.15 K) then the density of the gas is:

$$\rho = \frac{n}{V} = \frac{P}{RT} = \frac{PM}{R_{\rm u}T} = \frac{850,000 \times 32}{8.314 \times 293.15} = 11.16 \,\rm kg \cdot m^{-3}$$

If the volumetric flow $q_v = 2 \ m^3 \cdot min^{-1}$ then the mass flow rate would be

$$q_m = q_v \rho = 2 \times 11.16 = 22.32 \text{ kg} \cdot \text{min}^{-1}$$

Nearly all gas flow processes depend on mass flow rather than volume flow. For example, mass flow measurement is critical in natural gas custody transfer, because buyers and sellers want to be sure that the quantity of gas metered at one point along a pipe is equal the quantity of gas delivered at another point along the pipe. Gas density changes with temperature and pressure which will vary throughout a plumbing system. An accurate direct comparison of volume flow at one location to volume flow at another location without accounting for changes in density would result in errors.

Since mass flow rate is constant throughout a system, if you measure mass flow rate at a reference location you will know what the mass flow rate is at the test location. Even when calibrating volumetric devices, a mass flow standard can be used. The mass flow at the device under test (DUT) will be equal to the mass flow at the reference and the volumetric flow rate can be computed under standard conditions using the equations presented above.

Mass flow standards

For reasons explained earlier it is best to calibrate flow devices using a mass flow standard. Mass flow calibration standards can be divided into two main categories: primary standards and transfer standards.

- 1. Primary mass flow standards derive flow directly from the fundamental units of mass (kg) and time (s). An example of a primary standard would be a gravimetric gas flow standard. These devices measure the decrease in mass of a container from which gas is flowing over a measured time interval. High accuracy primary standard measurements would take into account such things as buoyancy effects caused by expansion of the container, and corrections required for local gravity. The complexity and expense involved in making primary standard measurement is usually higher than measurements involving transfer standards.
- 2. Transfer standards always take their value by direct comparison to a primary standard, or to another standard in a chain of comparisons that lead back to a primary standard. Because the accuracy of the transfer standard is based on a comparison to other standards, it is critical that the primary standard used in the chain is very accurate. Transfer standards are generally easier to use and less expensive that primary standards. Most calibration laboratories prefer to us transfer standards because they are easier to use, sufficiently accurate, less expensive, less prone to error, and more rugged.

There are two technologies that have proven to be very successful transfer standards in gas flow measurements.



1. Laminar Flow Elements (molbloc -L)

A laminar flow element calculates flow based on the properties of fluid dynamics when laminar flow is observed. The flow of a known gas in the laminar flow regime can be calculated from the following three things:

- 1. Flow path geometry
- 2. Gas pressure (absolute and differential)
- 3. Temperature

The conditions necessary for laminar flow can be summarized in a factor called the Reynolds Number.

$$\mathsf{Re} = \frac{\rho \mathsf{VD}}{\eta}$$

 $\begin{array}{l} \rho = \mbox{ density} \\ V = \mbox{velocity} \\ D = \mbox{tube diameter} \\ \eta = \mbox{gas dynamic viscosity} \end{array}$

Laminar flow: Re<2300

An example of a laminar flow element is the molbloc-L. For Re < 1200 flow elements are available for flow rates from 1 sccm up to 100 slm. The molbloc-L can be used upstream or downstream from the device under test. Calibration options for the molbloc-L include upstream low pressure (250-325 kPa), upstream high pressure (325-525 kPa), and downstream atmospheric pressure (100 kPa), depending on the application. The laminar flow element is originally calibrated in N2 and up to six other available calibration gases: He, Ar, SF6, H2, CO2, and air.

The patented design of molbloc–L relies on two unique characteristics to achieve better precision than was possible using traditional laminar flow elements. The very narrow flowpath of molbloc–L elements creates a relatively large differential pressure, up to 50 kPa (7 psi), which can be measured with high precision and combines with the large LFE body to promote heat transfer between the gas and the molbloc. The gas is forced to take on the temperature of the molbloc body rather than cool as it expands and the system directly measures the temperature of the stainless steel body. The result is very high precision and repeatability. However, this design is only practical for relatively low flows.

2. Critical flow nozzles (molbloc-S)

When Reynolds numbers in the molbloc increase, the flow is eventually no longer laminar and laminar flow elements of the molbloc-L design cannot be used. Beyond that range a different technology, the critical flow nozzle (CFN) also called a sonic flow nozzle produces better results. Its operation is based on the critical flow venturi principle. The mechanical design of the element that contains the nozzle maintains a uniform temperature and a unidirectional flow stream upstream of the nozzle. Once the inlet pressure is set above a certain threshold pressure (relative to the downstream pressure) the nozzle

goes into critical flow and the mass flow rate can be calculated from the following three things:

- 1. Flow path geometry
- 2. Gas pressure
- 3. Temperature

An example of the sonic nozzle is the molbloc-S.

- Flow range: < 1 slm to over 5000 slm
- Calibration gases: N2, He, Ar, SF6, H2, CO2, and air
- Calibration pressure options:
 - 1. 50 kPa SP to 500 kPa LP
 - 2. 20 kPa SP to 200 kPa SP
 - 3. 200 kPa HP to 2000 kPa HP

With the molbloc-S the flow control needs to be upstream from the inlet. The mass flow range of the nozzle is defined by the pressure range. The sonic nozzle technology requires that the ratio of the outlet to inlet absolute pressure (known as the back pressure ratio, or BPR) be less than a critical value. Once this condition is met, the velocity of the flowing gas in the nozzle throat reaches its maximum-the speed of sound. As long as the BPR is maintained, the volumetric flow rate cannot be affected by increasing the inlet pressure or changing downstream conditions. With a known volumetric flow rate, all we need is the gas density to determine the mass flow rate. Temperature and pressure sensors inserted into or connected to the molbloc-S provide the temperature and pressure information needed to calculate gas density and from that the mass flow rate. Mass flow rate is then controlled by varying the absolute upstream pressure (density) of the gas delivered to the sonic nozzle.



A laminar flow element (LFE) is a very high precision mass flow transfer standard providing uninterrupted gas flow measurement in the laminar flow regime.



A critical flow nozzle (CFN) or sonic flow nozzle is a very high precision mass flow transfer standard providing flow measurement with uninterrupted steady-state flow in the critical flow regime.



Making reliable measurements

There is a basic assumption made in flow calibration when the reference and device under test are connected together and compared in series: The flow rate through each device must be equal. The truth of this assumption depends on at least three factors:

- 1. Is the measured quantity the same at the reference and device under test?
- 2. Are there any leaks present?
- 3. Have transient effects been removed?

The quantity measured

The best way to ensure that the quantity measured is the same at the reference as the device under test is to use mass flow. Even when calibrating volumetric devices, a mass flow standard can be used. The mass flow at the DUT will be equal to the mass flow at the reference. Mass flow is easily converted to volumetric flow referenced to any desired temperature and pressure using the equations in the gas laws section above.

Leaks

If there are any leaks between the reference and the DUT, then the two will not be measuring the same flow. So much depends on leak free plumbing in flow calibration, that the expert metrologist often becomes an expert plumber. When expert plumbing fails, reliable leak detection can pick up some of the slack.

Common methods of detecting leaks:

- 1. Leak detection fluid
- 2. Isolate the system and measure pressure decay
- 3. Isolate the system and observe flow indication

The first method is to use leak detection fluid. Leak detection fluid is applied to the exterior of the plumbing. If there is a leak, then bubbles will form. This can be a very useful way of locating a leak that you already know is there, but it may not be the most reliable method for proving that no leaks exist

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in the system. Specialized leak detection fluids that dry clean without leaving residue are best. Soap and water mixtures are generally undesirable because it may stain or leave a residue on the instruments.

The best way to verify your system is leak free is to isolate the system and measure the pressure decay. That means pressurizing the system but preventing flow. When there is a leak we would expect to see the pressure to decline. If the system is leak free, then the pressure would be expected to remain constant; unless the leak is very small. When the leak is small the flow indication will likely be more sensitive to leakage than the pressure indication.

Flow indication can also be very helpful in isolating the location of the leak. For example, if the readout indicates negative flow, then we know the leak is in the direction of the reference inlet, however if the direction of the flow is positive we can focus our efforts on finding the leak on the outlet side.

On-board leak detection is a very useful feature when available in commercial readout devices. For



The molbox1 + flow terminal displays the results of flow measurements from either the molbloc-L laminar flow element or the molbloc-S sonic flow nozzle, and provides on-board leak detection.



example, the molbox 1 + flow terminal is the part of the molbloc/molbox gas flow calibration system that indicates the flow measured by a molbloc reference flow element. The molbox on-board leak check function will check both for pressure decay and measurable flow indication to assist in detecting and isolating the location of a leak in the flow system. If no leaks are found then the system is ready for calibration.

Transient effects

Achieving stable steady-state flow is critical to calibration. The mass flow controller plays an important role in establishing stability in the measurement system. If the DUT is a mass flow controller, then it may be used in control mode. However, mass flow controllers should be installed up stream when sonic nozzles are used as a reference. If the outlet of a DUT exhausts to atmospheric pressure or vacuum then any pressure regulators or mass flow controllers should be installed upstream too.

Over a sufficiently long period of time the average flow rate through the reference will be equal to the average flow rate through the DUT. However, even with a mass flow controller, over short time periods the flow rates may vary. Here are a few things you can do to improve results:

- 1. Wait for stability after adjusting flow
- 2. Optimize pressure and flow control hardware for stability
- 3. Average several readings over time to reduce noise
- 4. Average each device over the same time period

Positive displacement collection devices (provers) alternate between two modes where gas flows into a container to move a piston for measurement or where gas bypasses the piston. These devices experience fluctuation in pressure and flow when flow switches from bypass to measurement. This affects the ability of the prover to establish steadystate flow. With traditional low speed bell provers or mercury sealed piston provers, special care must be taken to wait for stability after piston motion begins. High speed dry provers cause continuous cyclical pressure and flow fluctuations, affecting the indication of devices being compared. With those devices, averaging multiple cycles is recommended. However, even with averaging, all transient affects may not be removed, because the prover measures only during its measurement cycle and the compared device measures during both the measurement cycle and the bypass cycle. Sampling only during one mode of operation and not the other with the reference may introduce a bias into the measurements.

Alternatively, laminar flow elements and sonic nozzles allow the system to establish a steady-state of continuous flow. This allows the system time to become stable and is never interrupted or perturbed in the course of a measurement. Laminar flow



Unlike laminar flow elements and sonic flow nozzles, a piston prover's ability to establish steady-state flow is affected by its continuous need to alternate between flow measurement and gas bypass modes.

elements and sonic nozzles also provide the ability to sample readings over the same time intervals that the DUT is being measured, eliminating a potential source of bias.

Conclusion

This paper provided a definition for flow and discussed the relationship between volumetric flow and mass flow. Unlike volumetric flow mass flow was shown to be equal at different location in the plumbing of the flow calibration system, making mass flow standards preferred over volumetric flow standards. The impact of density and compressibility were discussed and both were shown to depend on atmospheric conditions of temperature and pressure. Finally, some guidelines for making effective measurements were provided around choice of standards, leak detection, and maintaining steady-steady state flow.

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