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MARCH 1997

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European Technology p. 123

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Consistent ±3 Sigma Calibration For Mass Flow Controllers

Production mass flow control measurements, at near-NIST accuracy, challenge calibration standards technology.

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Key Technologies:

- **Mass flow controller**
- **Process capability**
- **Statistical metrology**

At A Glance:

Described in detail is a system which Unit Instruments developed over the past two years to measure and track the calibration accuracy and consistency of a mass flow controller manufacturing process. The ability to tighten mass flow controller (MFC) accuracy specifications and to specify calibration accuracy as a percentage of setpoint rather than a percentage of full scale is examined with consideration given to statistical process capability C_{pk}. The article also presents a historical viewpoint of MFC manufacturing specification limits to illustrate the need for a new approach to MFC production metrology.

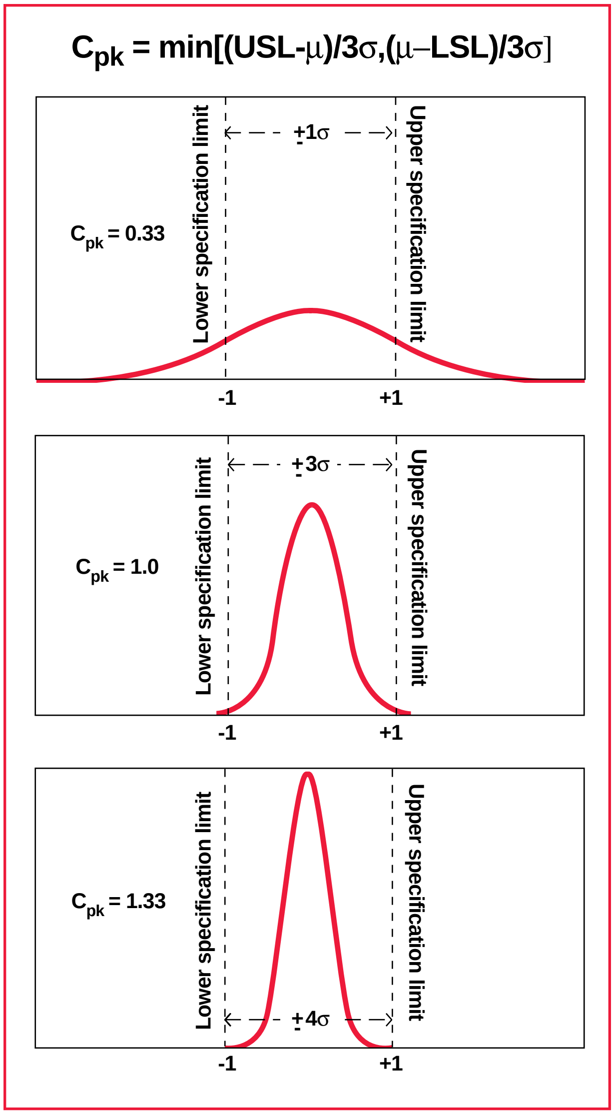
Since the 1970s, mass flow controller (MFC) manufacturers have specified the calibration accuracy of MFCs used in the semiconductor industry as ±1% of full scale. However, production MFCs have never consistently met that specification. Many in the industry realized over the years that calibration equipment was not capable of consistently producing true ±1% results with high confidence. Unfortunately, once that 1% number appeared on the first MFC data sheet, no MFC manufacturer could publish a higher specification number and expect to sell product, even if the higher number was more statistically descriptive of the MFC's performance. MFC users have been aware of the significant variability in the calibration of all MFCs, but they have had no way to determine the actual calibration tolerance of the

Unit Instruments measures and tracks the output of its MFC calibration process, which is traceable to NIST standards.

devices they receive from published accuracy specifications. The methodology explained here gives manufacturers and users a way to set realistic expectations for the accuracy of production MFCs.

The metrology squeeze

The practice of specifying MFCs to 1% and delivering MFCs that do not meet that specification has principally been a metrology issue. Until recently, calibration systems suitable for use on MFC manufacturing lines were not process capable or stable enough to produce devices that consistently meet specifications. Compounding this are customers' expectations for process capability today, where a process capability index (C_{pk}) of 1.0 (99.7%, or 6σ of production meets specification) is considered the minimal level of acceptability (Fig. 1). The problem has a simple origin. In order to meet 1% specifications, it is necessary to calibrate production MFCs with devices that are as accurate as the primary calibrators at a national standards laboratory. The National Institute of Standards and Technology (NIST) fluid flow laboratory in Gaithersburg, Md. quotes a total flow uncertainty of 0.22%. According to conventional wisdom in metrology practice embodied in MIL Standard 45662, accuracy degrades 4:1 as a measurement is transferred from a central laboratory. This is not adequate to meet semiconductor industry requirements. Assume for the moment that NIST could certify the principal calibrator in a laboratory and that calibrator was then used to certify production calibrators for MFCs. This procedure is three calibration steps from NIST to the final product. Using a 4:1 generational degradation at each setup, MFCs could be certified with high confidence to $\pm 0.22\% \times 4^3 = \pm 14\%$, a value which is unacceptable. Fortunately, the 4:1 generational degradation assumption does not hold with the best current technology. Local primary calibrators can now be maintained at NIST accuracy levels and portable standards for production can inherit this accuracy with little generational degradation.



1. The difference between standard variation and process specification is described by the process capability index, C_{pk} = min ((USL-μ)/3σ, (μ-LSL)/3σ).

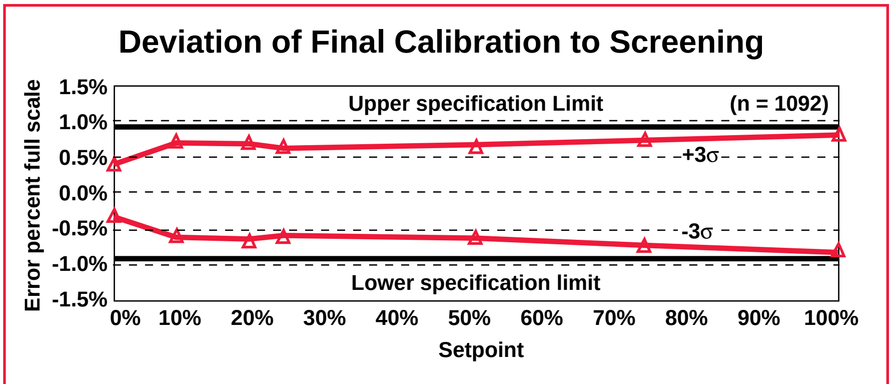
Measuring flow

The standard cubic centimeter per minute (sccm) is a derived unit; a combination in the simplest terms of mass over time. The sccm is not usually traceable as a flow rate, but through standards for length, force, time, temperature, and faith in the perfect gas law, PV=nRT. Primary calibrators are complex machines that measure flow based on some or all of these units. Primaries, other than gravimetric, fix temperature and either pressure (force) or volume (length) and measure how the other changes over time. It is possible to compare primary calibrators with other calibrators as a means of verifying their accuracy, but they cannot be compensated in good conscience with some "fudge factor." If so, they become secondary standards; devices that have no intrinsic relationship to fundamental units, but are dependent on having been calibrated by another flow device with higher authority. If a primary calibrator does not accurately measure flow, then it is necessary to correct how it incorporates the fundamental units – length, force, time, temperature, and PV=nRT (see sidebar, "Thermal Mass Flow Control").

It is common practice to specify primary calibrators at 0.25% total uncertainty. If used in production they would seem to be capable of producing 1% MFCs consistently. Some companies calibrate at least a portion of their output on primaries for that reason, but the practice is not widespread. This is because primary calibrators are usually agonizingly slow and relatively expensive. They are also complex machines that can harbor or develop systematic errors which usually do not become apparent until it is too late. Engineers in the semiconductor industry sometimes place unwarranted faith in primary calibrators, assuming they are robust and always meet their quoted accuracy specifications.

MFC manufacturers usually get around the slow speed and temperamental nature of primary calibrators by using them to calibrate transfer standards and then using the transfer standards to calibrate production MFCs. In addition, MFC manufacturers often use other MFCs as transfer standards. However, MFC transfer standards deservedly have a poor reputation. They may be fast, but calibrators using MFCs as references for other MFCs are neither sufficiently reproducible nor stable under production conditions if ±1% is the goal.

Acceptable flow standards for MFCs used in manufacturing need to be well characterized with respect to calibration and must measure flow with an accuracy of better than 0.5% of reading.



2. The capability of the manufacturing process is illustrated by a plot of the 3 sigma limits against the ±1 percent specification limits.

They should be extremely rugged and stable, and small enough so as not to physically dominate the production line. They should generate an almost instantaneous flow reading in order to allow for quick adjustments to the device being calibrated.

A transfer calibration system, tradenamed "Molblocs" and manufactured by DH Instruments (Tempe, Ariz.), using laminar flow elements (LFE) and pressure transducers, generally meets these requirements. We carefully characterized and monitored these "Molblocs" within a comprehensive metrology system. We created armored versions of these transfer standards, which consistently produced good results.

Capability of calibration processes

The variability of a mass flow controller manufacturing process, like other processes, can be described statistically. No two devices are, or can be, exactly alike. When measuring a parameter such as calibration accuracy over a number of devices, a distribution of values is observed which can be plotted and analyzed. In a well-controlled process, the distribution of values follows the familiar bell-shaped curve of a normal (Gaussian) distribution. Most of the values lie close to the ideal, diminishing in number as the degree of error increases. The relationship between the shape of the distribution and the device specification limits describes the statistical confidence that any given device will meet its specifications and the statistical capability of the process.

The production calibration process described here produces an error distribution of better than ±3 sigma at the specification limits (Fig. 2). This means that more than 99.7% of product shipped conformed to calibration specifications. Previously, limited tests using MFC transfer standards on production calibrators indicated that approximately 70% of product shipped conforms to calibration specifications. That 1 sigma calibration process was improved to a better than 3 sigma process at the worst-case point closest to the specification limits. Interestingly, the data shows that MFCs are now relatively more accurate in the mid- and lower portions of their range than a strict percentage-of-full-scale specification would indicate. If the measured error at all of the calibration points of all the MFCs is included in one calculation, the 3 sigma value is 0.713% of full scale. This data supports the potential to re-specify product in terms of percentage of setpoint rather than percentage of full scale over at least a portion of the flow range. Digital MFCs with more sophisticated calibration ability will benefit even more from this new calibration process capability.

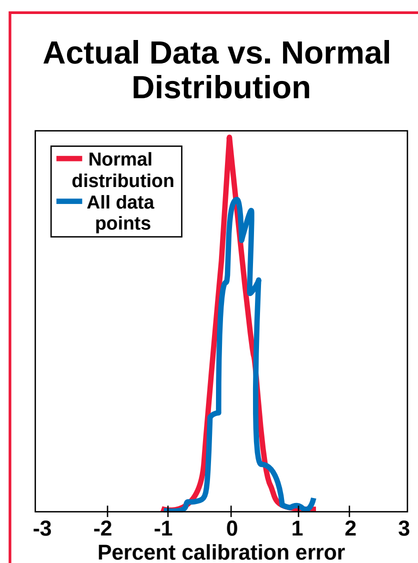
A statistical analysis of the type presented here presumes that the data follows a normal distribution. To test that assumption in a visually obvious manner, we plotted the distribution of all the points in the database against a mathematically-generated normal distribution containing the same number of points with the same standard deviation. The results, plotted in Fig. 3, show that the data is distributed very much like the theoretical bell-shaped curve.

The methodology developed to measure and track the output of the MFC calibration process operates as follows: we periodically pulled samples from the end of the production line for metal sealed MFCs and measured their calibration again. We used multiple sets of the same calibration equipment for production, and never screened an MFC on the same calibrator used to produce it. MFCs for many different flow ranges and gases were in production at the same time. We randomly selected samples from this group so that the resulting data was representative of the MFCs shipped.

The real error in the calibration of individual MFCs cannot be determined precisely because the uncertainty of the equipment used to check MFC calibration is, at the worst point, only three times better than the specification limits for the MFC. Put another way, the MFC could be dead accurate and the measuring equipment could indicate that it was in error by a third of the specification limit. In another example, the MFC could be within specification but close to one of the specification limits. The measuring device, adding its uncertainty to the measured value, could indicate erroneously that this MFC was not within specification.

Calibration data for MFCs

We addressed the problem of calibration uncertainty by measuring each MFC on two different calibrators and examining the difference between the two readings obtained over a large number of MFCs. This statistical data contains the differences between

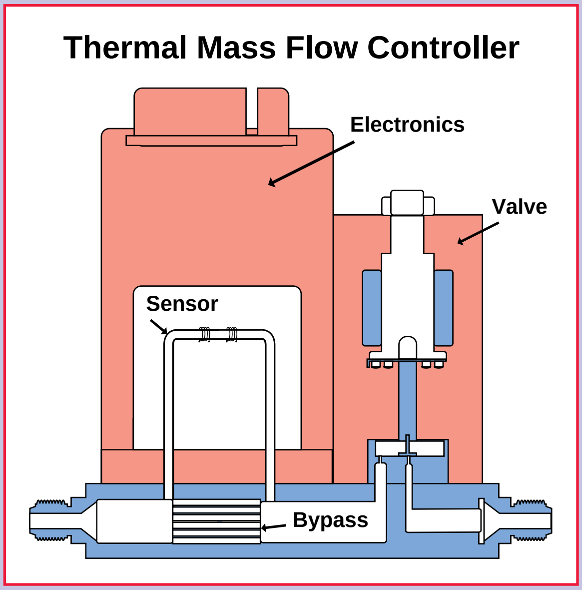


3. The distribution of all points in the database against a normal distribution shows that the data is distributed near the theoretical bell-shaped curve.

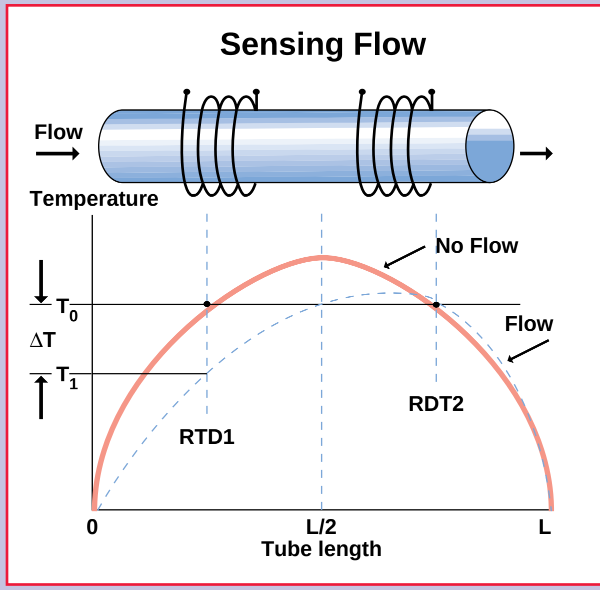
calibrators, as well as the short-term reproducibility errors of the MFCs. When all sets of calibration equipment used to manufacture MFCs are included in the testing, then the data is a measurement of the uncertainty in calibration of the entire population. This assumes that the average of the production calibrators is accurate. We examine this assumption below.

We collected data for a sample of 1092 mass controllers over 14 months, from June 1995 through July 1996. The database identifies each MFC by model, serial number, calibration gas, range, date tested and test system used. We measured MFC calibration at seven setpoints including zero: 0, 10, 20, 25, 50, 75 and 100% of full scale and tested MFC flow ranges from 100 sccm through 20 slm (= 20,000 sccm) The sets

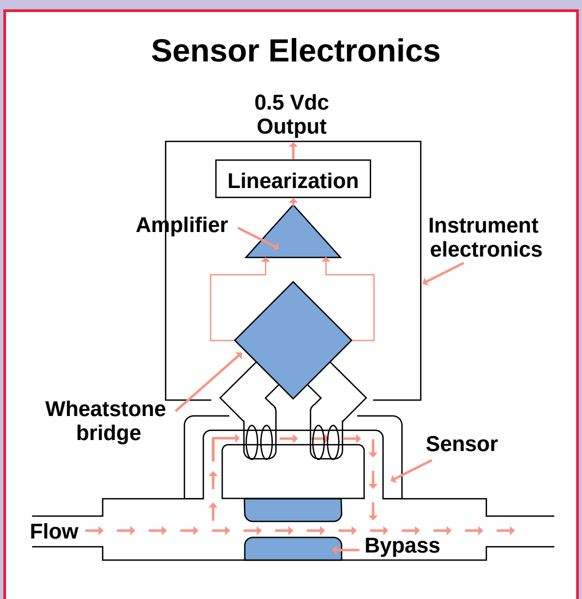
Thermal Mass Flow Control



a. A partial cutaway of a mass flow controller shows the capillary tube.



b. The difference between the two RTDs, ΔT, corresponds to mass flow rate.



c. Changes in sensor coil resistance are converted to a 0-5 VDC signal.

Mass flow meters use the thermal properties of a gas to directly measure flow rate from the equation $q = (dm/dt)c_p \Delta T$, where q is heat lost to gas flow, dm/dt is the mass flow, c_p is the specific heat for a constant pressure, and ΔT is the temperature difference. The sensor in an MFC is a long, thin stainless steel tube, often called a capillary tube because of its shape. At the midpoint of the capillary tube, two wire coils are wrapped side by side which serve two functions: first as heaters, and second as temperature sensors. Since the resistance of the coils vary with temperature, the coils function as resistance temperature detectors, or RTDs, which measure the temperature of the gas. When there is no gas flow, heat from the coils generates a uniform temperature gradient about the midpoint of the tube. As gas flows through the sensor, the gas flow shifts the temperature so that a temperature difference develops between the two sensors. This temperature difference can be converted into a flow reading. □

Table 1. Final Calibration Data - Deviation From Ideal Value in Percent of Full Scale

Number of samples	1092						
Setpoint	0%	10%	20%	25%	50%	75%	100%
Average	0.03%	0.02%	0.05%	0.01%	0.01%	0.02%	0.02%
Std. Dev.	0.07%	0.21%	0.12%	0.08%	0.09%	0.10%	0.11%
+3 Std. Dev.	0.24%	0.64%	0.42%	0.26%	0.28%	0.31%	0.33%
-3 Std Dev.	-0.18%	-0.60%	-0.33	-0.24	-0.26%	-0.27%	-0.30%

Table 2. Deviation From Final Calibration to Screening in Percent of Full Scale

Number of samples	1092						
Setpoint	0%	10%	20%	25%	50%	75%	100%
Average	-0.02%	0.01%	0.00%	0.00%	-0.01%	-0.01%	-0.01%
Std. Dev.	0.13%	0.18%	0.18%	0.18%	0.22%	0.26%	0.31%
+3 Std. Dev.	0.37%	0.54%	0.55%	0.53%	0.64%	0.78%	0.92%
-3 Std. Dev.	-0.42%	-0.53%	-0.55%	-0.54%	-0.65%	-0.80%	-0.95%

of measurements for both final calibration and for the special calibration screening test performed on this sample are included in the database.

We analyzed the data in several ways as a means of process control. We examined the readings that were recorded when the MFC was produced to confirm that operators were consistently calibrating MFCs to the specifications listed in the manufacturing procedures. We found that the deviation at the 10% setpoint was relatively larger because these MFCs do not have a separate linearity adjustment below 25% of full scale. The more precise linearization of a digital MFC is expected to bring the data at this setpoint closer to the ideal. Table 1 describes calibration data recorded at the last stage of production. Error at the 10% setpoint is relatively large because the MFCs have no linearity adjustment below 25% of full scale.

We also monitored the deviations between the measurements at final calibration and at screening to confirm that the calibration equipment, operators and procedures were producing product within specifications. By subtracting the final calibration data from the screening data for each MFC, we removed the effect of any offset which may have been calibrated into the MFC. This left only the error in the process. Table 2 presents the differences between the final calibration data and subsequent data at retest on another calibration station. As summarized in Table 2 and plotted in Fig. 2, the data shows that the process produced MFCs at the ±3 sigma limits well within the ±1% calibration specification. Ninety-five% of the product falls within two standard deviations and was within ±0.6% of full scale.

The need for absolute accuracy

The data presented here addresses the important question of the variability of the MFC manufacturing process. It describes how tightly the calibration error distribution is grouped around the average value of the process. To completely describe the calibration accuracy of the population, however, it is necessary to know the degree to which the process average differs from absolute accuracy. A process may be tightly controlled, but if the metrology on which it rests is not centered on the true value, devices can still fall out of specification. The multiple sets of production calibration equipment need to remain accurate with respect to some external reference, and not just consistent within themselves, requiring that production calibration equipment be maintained within a comprehensive metrology calibration system. As noted above, to achieve the level of accuracy required to meet a 1% gas flow specification requires that production equipment be very closely referenced to a primary metrology system that is as accurate as a national standards laboratory.

Using critical flow nozzles (CFNs) and LFEs as transfer standards, three separate primary calibration methods — gravimetric, variable volume and variable pressure — were compared to center calibration processes. Direct comparisons were made with NIST and other laboratories using CFNs and LFEs. By maintaining different primary calibrators which do not share the same error sources, it is easy to detect a problem that may develop in any one of the calibrators. The LFEs (in this case, MolBlocs) are subsequently used to calibrate production MFCs, which gives a high degree of confidence that the average of all production calibration measurements is accurate to within several tenths of a percent.

Both absolute calibration accuracy and tight calibration process control are necessary to meet the needs of the industry. These calibration processes are necessary to guarantee that MFCs produced at any given time are all the same. Accurate fundamental metrology is necessary to ensure that the calibration process center remains the same over time and that products produced in multiple plants or with multiple sets of standards are comparable. Flow can only be measured by active machines; the more primary calibrators that agree in their readings, each measuring flow by different principles, the more confidence there can be that their combined results are truly accurate. In this way, the traditional 4:1 generation allowance for each transfer step is overcome. The measurement cannot be transferred from NIST. It must be created locally and transferred to production MFCs in one step. Process engineers sometimes say, "Forget accuracy, just make them all the same," but that statement misses the point. The only metrology task that is more difficult than consistently measuring an accurate value is to consistently measure some value that is not the truth. It is impossible to ignore either accuracy in metrology or process capability, and still make all MFCs the same. □

Reprinted from **SEMICONDUCTOR INTERNATIONAL** March, 1997