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THE DISSEMINATION OF GRAVIMETRIC GAS FLOW MEASUREMENTS THROUGH AN LFE CALIBRATION CHAIN

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ABSTRACT

With the use of gravimetric methods to measure low gas mass flow a laboratory can be directly traceable to the fundamental units of mass and time with very low uncertainties. In order for the benefits of this primary technique to affect day to day calibrations, the measurements must be readily available through transfer standards using relatively simple methods. A calibration chain composed of Laminar Flow Elements (LFEs) has been implemented with the purpose of disseminating gravimetric measurements. This paper describes the calibration chain technology, structure, characterization and maintenance.

KEY WORDS: mass flow, gas flow, primary standard, transfer standard, gravimetric determination, uncertainty analysis, laminar flow elements, traceability

1. INTRODUCTION

One method of establishing traceability for mass flow measurements is by gravimetric determination. This method is now commonly used not only for non-compressible fluids but for gases at relatively low flows. Gravimetric mass flow calibrations are beneficial to a flow laboratory because measurements are made directly in the fundamental units of mass and time^[1] without requiring knowledge of the thermodynamic properties of the gas that is being measured^[2]. The uncertainty in the measurement is dominated by the uncertainty in mass and time measurements. With the use of high resolution, high performance mass balances that are available today and time measurements traceable to the WWVB time frequency broadcast service, these uncertainties can be very low.

Though gravimetric calibration is metrologically beneficial, it is very time consuming and quite technique dependent. Because of this, the gravimetric calibration is not generally suited for day to day calibrations on process instruments or large numbers of transfer standards. This is all the more true when traceability is required for a number of gases.

A calibration chain of laminar flow elements was created to be used as a direct transfer from gravimetric determinations to other transfer standards used to calibrate process instruments. This group of LFEs is constantly referenced to gravimetric determinations and individual LFEs are also linked to each other in a manner designed to minimize uncertainties. The group of LFEs covers a flow range from 2×10^{-8} to 7×10^{-4} kg/s (approximately 1 to 30 000 sccm in N₂).

2. PERFORMING GRAVIMETRIC CALIBRATIONS

Performing a gravimetric determination or comparison is conceptually very simple. Two methods are used today: dynamic mass determinations; and static mass determinations.

The dynamic method is performed by depleting gas from a bottle that is placed on a mass balance while the measurements are being made. Time measurements and mass measurements are taken simultaneously. If the mass balance can be interfaced with a computer, the mass and time measurements may be taken quickly enough to synchronize to within the mass balance's resolution. Since the mass of the bottle assembly is much greater than the depleted mass of the gas, mass balances of relatively high ranges must be used. The resolution of the balance becomes a significant uncertainty component. This method also has the advantage that more than one gravimetric comparison can be taken without removing the bottle from the system. If there is enough gas in the bottle to perform

many gravimetric comparisons, then a complete gravimetric calibration on an LFE at several flow rates may be performed without removing the bottle to perform mass measurements and reconnect and stabilize flow between measurements. Figure 1 shows a simplified diagram of the dynamic mass setup.

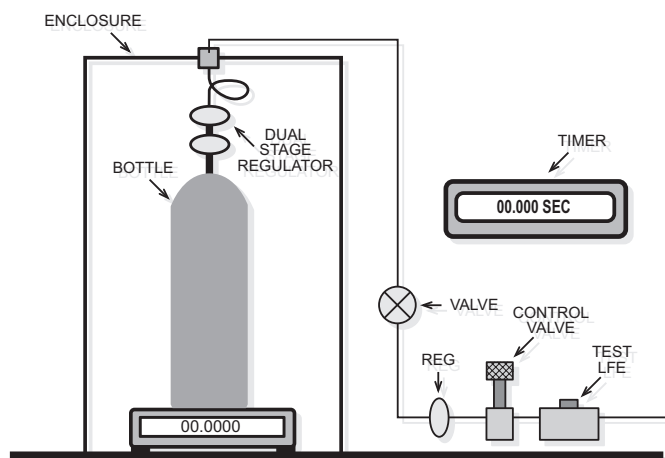


Fig. 1 Dynamic Mass Gravimetric Setup

The dynamic method generally saves time relative to the static method but has one design obstacle. The flow connection from the bottle to the test instrument prevents the bottle from being completely free from parasitic force influences as it rests on the balance. Flexible tubing can be used to limit the effect from the connection, but the pressure must be very stable to reduce the influence of an unstable "bourdon" effect contributed by the tubing.

The dynamic mass method is currently not being used to calibrate any part of the LFE calibration chain, however plans are underway to include this method by the end of 1999. This method is expected to improve uncertainties in very low flow ranges.

The method that has been used exclusively to characterize the LFE calibration chain is the static mass method. In this case, the bottle is not placed on the balance as shown in Fig. 1. The procedure involves before and after mass weighings of a bottle with gas being depleted between the mass weighings. Fig. 2 shows a similar setup to Fig. 1 without the bottle resting on the balance.

A basic procedure for performing a static mass gravimetric determination may include the following:

- Fill a bottle with the gas to be tested by first evacuating the bottle then connecting it to the supply bottle to be filled.
- Test the bottle for leaks by connecting it to the test flow circuit, capping off the end of the circuit, and identifying any flow or change in pressure.
- Purge the test flow circuit with the bottle gas and set the flow point.

- Remove the bottle from test circuit for weighing in state one. This includes measuring ambient conditions at the time of the weighing.
- Connect the bottle to the test circuit and perform the depletion test.
- Remove the bottle from the test circuit for weighing in state two. Again, this includes measuring ambient conditions at the time of the weighing.

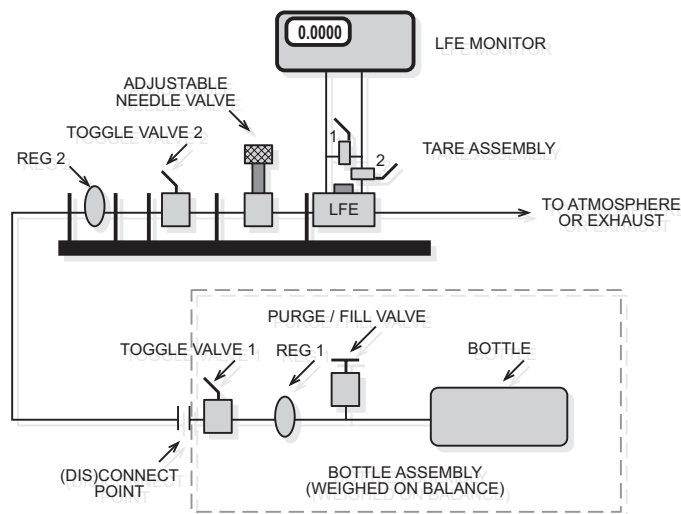


Fig. 2 Static Mass Gravimetric Setup

The test depletion times, Step e., can take anywhere from 30 to 100 000 minutes (three days), with the average being 180 minutes, depending upon the gas and set point of the determination. Time spent on the rest of the steps is approximately 30 minutes. The reason the determinations require so much time is for enough gas to be depleted so the difference between the first and second state measurements is large enough for the balance to measure it accurately. The mass difference needed is highly dependent upon the resolution and the repeatability of the mass balance. The minimum depletion time of 30 minutes is set to ensure that discrepancies from starting the test and the finishing "ramp" are insignificant.

The static mass method has a very low uncertainty. Table 1 illustrates the uncertainties^[3] typically associated with a gravimetric determination. The uncertainties that would normally be attributed to the test instrument being gravimetrically characterized are intentionally left out of this table to show the contributions that are specific to the gravimetric reference.

Table 1 is presented in this manner to illustrate how low the uncertainties can be in a gravimetric test. As long as uncertainties contributed from the test instrument are kept to a minimum, the benefits of gravimetric methods may be exploited.

| INFLUENCE QUANTITY | Unc/Mass @ K = 2 | 1 Standard Uncertainty |
|----------------------------|---------------------|---------------------------|
| Balance Resolution | 0.0024 % | 0.00140 % |
| Balance Repeatability | 0.0024 % | 0.00140 % |
| Balance Linearity | 0.0024 % | 0.00140 % |
| Reference Mass | 0.0005 % | 0.00030 % |
| Time | 0.0100 % | 0.00580 % |
| Buoyancy Correction | 0.0001 % | 0.00005 % |
| Purity of Gas | 0.0100 % | 0.00500 % |
| Type A | 0.0047 % | 0.00240 % |
| | Combined: | 0.00800 % |
| | Expanded for k = 2 | ± 0.01700 % |
| BALANCE CHARACTERISTICS | | |
| Total Mass Depletion: | 816.700 grams | |
| Balance Resolution: | 0.020 grams | |
| Balance Linearity: | 0.020 grams | |
| Balance Repeatability: | 0.020 grams | |
| Total Buoyancy Correction: | 0.008 grams | |

Table 1 Uncertainties Specific to Gravimetric Measurements

Considering the length of time currently required to perform successful gravimetric calibrations, the LFE is ideal as a transfer standard to obtain traceability from gravimetric determinations. LFEs measure flow continuously so the gravimetric test does not have to be interrupted for resetting or recycling. Also, LFEs are very stable^[2,4,5]. However, if a laboratory is required to calibrate many LFEs, it may be excessively burdensome to calibrate them all directly by gravimetric methods. Calibration turnaround times and costs can become unreasonable. For this reason, an effort was made to develop a standard that is comprised of LFEs of all necessary ranges that can be used to calibrate other LFEs without an excessive loss of uncertainty.

3. THE LFE TRANSFER STANDARDS

In order for the LFE transfer standard to be successful in calibrating other LFEs, the characteristics of the LFEs, i.e. stability and repeatability, have to be good enough for transfer uncertainties to be kept to a minimum.

The LFEs in this case are instruments that consist of two parts. The first part is the LFE itself which defines the geometry of the flow path and measures the changes in the isotherm created by the LFE with platinum resistance thermometers (PRTs). The second part of the instrument is separate from the first and houses two absolute pressure transducers to measure the absolute pressure of the gas and the differential pressure drop across the LFE. It also converts resistance measurements read from the LFE's PRTs for temperature of the gas as well as the temperature of the LFE. This "LFE monitor" is used to calculate and display the flow based on the flow path characteristics, the temperature and pressure of the gas, and the dynamic viscosity and density of the gas under flowing conditions. The LFE monitor can be

used interchangeably with other LFE monitors. Flow measurements can be made and the display updated in less than two seconds.

The LFEs themselves have very good repeatability and stability characteristics. Typically they are inside of ± 0.03 % for 12 months. The flow path is defined by stainless steel and is a solid geometrical shape, and the PRTs have stability within ± 0.01 % for 12 months.

The larger uncertainties in characterizing an LFE system are associated with the pressure and temperature measurements made on the LFE using the LFE monitor. Table 2 is the same uncertainty budget for a gravimetric calibration as is shown in Table 1, but includes the uncertainties from the LFE and the LFE display (in bold).

| INFLUENCE QUANTITY | Unc/Mass @ K = 2 | 1 Standard Uncertainty |
|-----------------------------|---------------------|---------------------------|
| Balance Resolution | 0.0024 % | 0.0014 % |
| Balance Repeatability | 0.0024 % | 0.0014 % |
| Balance Linearity | 0.0024 % | 0.0014 % |
| Reference Mass | 0.0005 % | 0.0003 % |
| Time | 0.0100 % | 0.0058 % |
| Buoyancy Correction | 0.0001 % | 0.0000 % |
| Pressure (Absolute) | 0.0200 % | 0.0100 % |
| Pressure (Diff Up) | 0.0300 % | 0.0150 % |
| Pressure (Diff Down) | 0.0300 % | 0.0150 % |
| Resistance | 0.0150 % | 0.0075 % |
| Gas Temperature | 0.0200 % | 0.0100 % |
| Type A | 0.0119 % | 0.0059 % |
| Stability | 0.0500 % | 0.0250 % |
| Purity of Gas | 0.0100 % | 0.0050 % |
| | Combined: | 0.0380 % |
| | Expanded for k = 2 | ± 0.0760 % |
| BALANCE CHARACTERISTICS | | |
| Total Mass Depletion: | 816.700 grams | |
| Balance Resolution: | 0.020 grams | |
| Balance Linearity: | 0.020 grams | |
| Balance Repeatability: | 0.020 grams | |
| Total Buoyancy Correction: | 0.008 grams | |

Table 2 Uncertainty Analysis of a Gravimetric Calibration of an LFE

The subject LFEs may be used over a downstream exhaust pressure range of atmospheric to approximately 500 kPa. They are normally either characterized over a pressure range or at a specific pressure depending on the needs of the customer. The nominal full scale differential pressure is 100 kPa when the downstream pressure is at atmosphere or 50 kPa when used above atmosphere. This makes it possible to place two LFEs in series and measure the same flow, hence the ability to directly compare one LFE to another. To designate between the two in such a comparison they are referred to as upstream or downstream LFEs.

Most applications for the LFEs to be used in the field are at pressures above atmosphere. Normally when the LFEs are used in the field, the LFE is placed upstream of the test device (process instrument) that is being calibrated requiring the LFE to be calibrated for a discharge pressure greater than atmosphere. This makes it practical to use downstream LFE transfer standards to calibrate the LFE field transfer standards at higher pressures (which can be called upstream LFEs). So it follows that the downstream LFEs (venting to atmosphere) are the ones that are calibrated gravimetrically. The uncertainty analysis shown above is for a downstream LFE characterization by the gravimetric method.

The expanded uncertainty of $\pm 0.076\%$ of reading that is indicated in the previous uncertainty table is adequate to calibrate an upstream LFE with an uncertainty of $\pm 0.2\%$ of reading with better than 2:1 accuracy ratio. This ratio, combined with the good repeatability and long term stability of the LFEs, has resulted in a very low number of out of tolerance LFEs on a one year calibration cycle.

4. THE DOWNSTREAM CROSSFLOW

In practice, for a laboratory that must calibrate a large base of test instruments for a number of gases, it is considered NOT desirable to use the downstream LFE standards for day to day calibration. This is because a great deal of gravimetric work has been invested to characterize these LFEs for each gas and range. LFEs used in day to day work are more susceptible to damage or contamination from handling which can jeopardize large amounts of high end metrology work. To reduce this risk, a method was needed to calibrate additional downstream LFE standards with the gravimetrically calibrated LFEs. The method that was created to accomplish this task is called the downstream crossflow.

A downstream crossflow is a method of calibrating a downstream LFE with another downstream LFE standard. It is performed by substitution using an

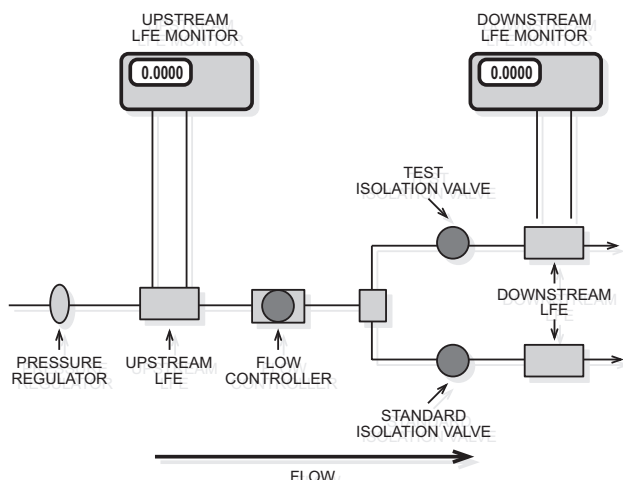


Fig. 3 Downstream Crossflow Setup

upstream LFE. The procedure is to exchange downstreams during a test to determine the difference of the two downstreams relative to the upstream. Fig. 3 shows a simple diagram of the setup for this comparison.

The test is performed by setting a flow with the downstream LFE standard being compared to the upstream while the test downstream isolation valve is closed. Leaving everything the same, including using the same downstream LFE display, the test isolation valve is opened and the standard isolation valve is closed. The difference between the standard and the test is measured using the short term repeatability of the upstream LFE and display under the exact same conditions. The repeatability of this type of test is easily determined by switching back to the standard.

Considering all conditions are kept constant while switching between the downstreams and the excellent very short term repeatability of the system, the added uncertainties to the test downstream LFE are very small. Table 3 shows the uncertainty analysis for this test which includes the expanded uncertainty from the gravimetric calibration of the downstream LFE standard shown in the previous section.

| INFLUENCE QUANTITY | Unc/Mass @ K = 2 | 1 Standard Uncertainty |
|-----------------------|--------------------|------------------------|
| Type A | 0.0300 % | 0.0150 % |
| Standard (from Above) | 0.0790 % | 0.0394 % |
| Type B2 (Temperature) | 0.0300 % | 0.0015 % |
| | Combined: | 0.0450 % |
| | Expanded for k = 2 | $\pm 0.0900\%$ |

Table 3 Uncertainty Analysis of LFE Calibrated by Downstream Crossflow

The Type A uncertainty component is a combination of the repeatability determined by switching and the residuals found in the characterization of the test downstream LFE. The Type B uncertainty for temperature is due to a possible drift in the temperature of the gas between switching of the downstreams. This is not a correlated uncertainty since the test is not dependent upon the absolute value of the temperature, only on deviations between the measurements made by the downstream standard and the downstream test.

5. THE LFE CALIBRATION CHAIN

With the benefit of a procedure that allows the comparison of two downstream LFEs with exceptional repeatability, a concept was derived that brought about a different type of downstream comparison. The procedure described in the previous section was originally created to calibrate a downstream LFE with another downstream LFE of the same range. However this procedure would also work for downstream LFEs of bordering, and overlapping ranges. This allowed for the creation of the LFE calibration chain.

The objective of the calibration chain is to verify the coherence of a large number of independent gravimetric calibrations on different range LFEs and different gases. For each range, of which there are nine, at least ten gases need to be characterized to support the calibration workload. To help ensure all gravimetric comparisons were performed correctly, each downstream LFE standard had to be compared to the next higher and lower ranges for that gas and had to agree within a global specification of $\pm 0.1\%$. This specification being the worst case uncertainty a downstream LFE standard could retain and be able to calibrate other downstream LFEs used in the day to day calibration processes.

The LFEs are gravimetrically calibrated from 10 to 100 % of their range. Comparisons in the calibration chain had to be within that range for each "link". Table 4 is an example from results obtained from the calibration chain for nitrogen gas. The comparison results are shown in the following format:

| | | Nominal Flows [sccm] | |
|-------------------|-------------------------------|----------------------|----------------------|
| Range of LFE 1 | Downstream 1 - Upstream [%] | AVG | |
| | Down 1 - Up Repeatability [%] | STDEV | |
| DOWNSTREAM LINK # | | | |
| Range of LFE 2 | Downstream 2 - Upstream [%] | AVG | Nominal Flows [sccm] |
| | Down 2 - Up Repeatability [%] | STDEV | Down 2 - Down 1 [%] |

Table 4 LFE Calibration Chain Results Format

The upstream's range is always the same range as the downstream 1 in that link. The downstream 2 in one link becomes the downstream 1 in the next link. Each downstream - upstream comparison is performed three times, switching between downstream 1 and 2, to determine the repeatability. The "down 2 - down 1" values are the result of that link.

There is a calibration chain for each gas that requires traceability. These calibration chains are redone on an annual basis to reveal changes or shifts in the any of the LFEs. Recalibration and maintenance of the calibration chain consists of regularly performing gravimetric calibrations at various flows and verifying the consistency. If an LFE appears to be inconsistent, which is very easy to see using links up and down from its range, then a complete gravimetric recalibration is performed for that LFE and the links for that range are reaffirmed.

The LFEs that comprise the calibration chain are used to calibrate the downstream working standard LFEs to perform day to day calibration work. The process that is used to calibrate the working standard is the downstream crossflow. The calibration chain is available for referencing to gravimetric determinations throughout the year.

| | | | | | |
|-------------------|--------|--------|--------|-------|-------------------|
| 10 sccm Range | 5 | 10 | AVG | 5 | 10 |
| | 0.049 | 0.013 | STDEV | -0.01 | 0.05 |
| DOWNSTREAM LINK 1 | | | | | |
| 50 sccm Range | 0.034 | 0.058 | AVG | | |
| | 0.064 | 0.012 | STDEV | | |
| DOWNSTREAM LINK 2 | | | | | |
| 50 sccm Range | 10 | 25 | 50 | AVG | |
| | 0.014 | -0.033 | -0.004 | STDEV | |
| 100 sccm Range | 0.017 | -0.013 | -0.003 | AVG | 10 25 50 |
| | 0.023 | 0.009 | 0.004 | STDEV | 0.00 0.02 0.00 |
| DOWNSTREAM LINK 3 | | | | | |
| 100 sccm Range | 20 | 50 | 100 | AVG | |
| | -0.030 | -0.059 | -0.129 | STDEV | |
| 200 sccm Range | -0.048 | -0.049 | -0.120 | AVG | 20 50 100 |
| | 0.031 | 0.009 | 0.008 | STDEV | -0.02 0.01 0.01 |
| DOWNSTREAM LINK 4 | | | | | |
| 200 sccm Range | 50 | 100 | 200 | AVG | |
| | 0.058 | 0.082 | 0.003 | STDEV | |
| 500 sccm Range | 0.025 | 0.032 | 0.041 | AVG | 50 100 200 |
| | 0.087 | 0.099 | 0.039 | STDEV | 0.03 0.02 0.04 |
| DOWNSTREAM LINK 5 | | | | | |
| 500 sccm Range | 100 | 250 | 500 | AVG | |
| | -0.134 | -0.112 | -0.253 | STDEV | |
| 1000 sccm Range | -0.135 | -0.132 | -0.278 | AVG | 100 250 500 |
| | 0.008 | 0.008 | 0.005 | STDEV | 0.00 -0.02 -0.02 |
| DOWNSTREAM LINK 6 | | | | | |
| 1000 sccm Range | 500 | 750 | 1000 | AVG | |
| | 0.097 | 0.121 | 0.143 | STDEV | |
| 5000 sccm Range | 0.077 | 0.085 | 0.115 | AVG | 500 750 1000 |
| | 0.003 | 0.002 | 0.002 | STDEV | -0.02 -0.04 -0.03 |
| DOWNSTREAM LINK 7 | | | | | |
| 5000 sccm Range | 1000 | 3000 | 5000 | AVG | |
| | 0.043 | 0.160 | -0.086 | STDEV | |
| 10000 sccm Range | 0.002 | 0.004 | 0.008 | AVG | 1000 3000 5000 |
| | 0.157 | 0.123 | -0.066 | STDEV | 0.11 -0.04 0.02 |
| DOWNSTREAM LINK 8 | | | | | |
| 10000 sccm Range | 3000 | 6000 | 10000 | AVG | |
| | 0.072 | 0.084 | -0.058 | STDEV | |
| 30000 sccm Range | 0.004 | 0.003 | 0.004 | AVG | 3000 6000 10000 |
| | 0.167 | 0.099 | 0.009 | STDEV | 0.09 0.02 0.07 |

Table 4 Calibration Chain Consistency

In addition to the internal gravimetric work and calibration chain intercomparisons, calibration chain LFEs are often compared with external flow standards of various types. These comparisons have verified the uncertainties claimed. Comparisons with national metrology laboratories have consistently shown agreement within ± 0.1 % of reading.

6. CONCLUSION

The partnership between gravimetric methods and LFEs as transfer standards has provided an opportunity to obtain traceability in mass flow to fundamental units of measurement. This is made possible primarily by two factors: the performance that is available with high precision mass balances; and most important, the performance of well designed LFE mass flow measurement systems.

The LFE calibration chain is the accumulation of a great deal of high end metrology work that is maintained on a regular basis. It is a unique resource that establishes and maintains direct traceability to mass and time with a high degree of confidence and using techniques that exploit the repeatability and stability LFEs have to offer and provides a reference that may be tapped on a routine basis by a mass flow laboratory. This has proven invaluable for a laboratory that performs many flow measurement calibrations a year.

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NOTES

The author wishes to acknowledge Pierre Delajoud for the development of the LFEs and the systems of gravimetric determination used for calibration chain traceability and also for the vision and inspiration which helped create the downstream crossflow and the calibration chain.

The use of the unit scm and slm are painfully included to provide a quick reference for readers to quickly understand the flow regimes covered in this paper. All measurements in the gravimetric determinations are performed in kg/s.