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A PRIMARY CALIBRATION SYSTEM FOR THE SUPPORT OF HIGH PERFORMANCE GAS FLOW TRANSFER STANDARDS

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Abstract: The practical application of high performance gas flow transfer standards requires means to efficiently and reliably calibrate them with very low measurement uncertainty. A unique primary gas flow calibration system has been developed to support gas flow transfer standards based on laminar flow and sonic nozzle based elements in the range of 2 x 10^{-5} g·s^{-1} (1 Ncc min^{-1}) to 50 g·s^{-1} (2500 Nl·min^{-1}). The primary calibration system is made up a gravimetric standard up to 0.2 g·s^{-1} (10 Nl min^{-1}) and a group of sonic nozzle based flow elements to extend the gravimetric reference flow measurements to higher values. Any non-corrosive gas can be studied. The gravimetric system measures depleted gas mass real time using a force balanced load cell and an automated taring system to eliminate force measurement drift over time. The group of nozzles uses the extensive nature of flow and a successive addition technique to build up the gravimetric reference measurements to higher flows. The primary calibration system supports a calibration chain made up of a group of laminar flow and sonic nozzle based elements characterized with a variety of gases. The structure of the group allows verification of the coherence of flow measurements up and down the chain and the precision of the chain to be quantified and maintained over time. The calibration chain is used for day to day calibration of gas flow transfer standards. A complete uncertainty analysis for the primary gravimetric system and the calibration chain has been performed.

1. INTRODUCTION

DHI is the manufacturer of very precise transfer standards for measuring flow[1,4]. Though the flow through these transfer standards can be determined following fundamental laws of physics, achieving traceable measurements with the desired low level of uncertainty requires a primary flow calibration system.

Originally, a static gravimetric flow system with a laminar flow element calibration chain[2] was implemented to achieve traceability in flow. Though adequate at the time, the calibration chain was difficult to maintain due to the excessive amount of time needed to perform gravimetric tests, in particular, tests at flows lower than 2 x 10^{-3} g·s^{-1} (100 Ncc min^{-1}). Because of the time consuming nature of these gravimetric tests they were not available commercially and were only used to characterize the molbloc-L™ calibration chain. Finally, because the static gravimetric system must start and end in a zero flow condition, it does not permit comparisons with the sonic nozzles (molbloc-S™) that DHI introduced in 2003 as a product to extend the molbloc range to much higher flows.

To overcome these shortcomings, a project to develop a dynamic gravimetric system was started in 1999. This decision was inspired by the success achieved with a similar dynamic gravimetric system developed and implemented at the French national metrology institute (LNE) in the mid to late 1990's[3]. The goals of the new system designed at DHI were:

- Require less mass depletion to reduce the amount of time necessary to take a point.
- Be able to take gravimetric points "on the fly" without having to remove bottles for weighing.
- Reduce the total uncertainty to a level of ± 5•10^{-4} of reading or better.

This system, now called GFS (gravimetric flow standard), was developed primarily in this millennium defines traceability for all flow measurements performed at DHI. It is also a commercially available product.

The implementation of the GFS by itself was not sufficient to support traceability in flows above 0.2 g·s^{-1} (10 Nl min^{-1}). This is primarily due to influences of excessive temperature changes induced by the higher flow rates. With the introduction of sonic nozzles (molbloc-S™) offering ranges up to 100 g·s^{-1}

(5 000 Nl min^{-1}) a method to extend traceability to higher ranges was necessary. Taking advantage of the excellent repeatability of the nozzles, an extensive measurement technique called successive addition was implemented in a calibration chain to meet the traceability needs at higher flows.

This paper examines the uncertainty and the results of the combination of the GFS and the successive addition technique used to build the molbloc-S calibration chain.

2. GFS DESIGN AND UNCERTAINTY

The primary design of the GFS is described in a previous publication[4]. Figure 1 and the table that follows identify the primary components of the GFS.

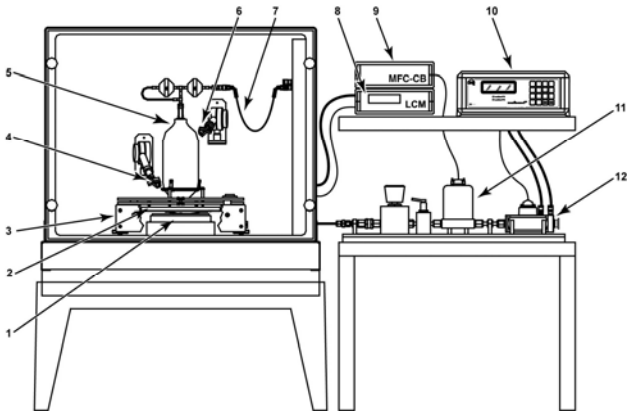

Fig 1. *GFS schematic*

1. Precision mass balance
2. Reference mass
3. Automated mass handler
4. Ambient conditions probe (for the cylinder)
5. Reference gas cylinder
6. Ambient conditions probe
7. Catenary gas conveyance loop
8. Laboratory conditions monitor (LCM)
9. MFC control box
10. Flow terminal (for device under test)
11. Mass flow controller
12. Device under test
13. IR temperature probe (not shown)
14. GFS Tools software (not shown)

Table 1. GFS main components

The following sub-sections provide a brief description of the components listed in Table 1.

2.1 Precision Mass Balance and Reference Mass

The mass balance used with the GFS was chosen for characteristics of speed, reproduceability and resolution. The balance used is a Mettler XP2004S with a resolution of 0.1 mg and a repeatability of ±0.1 mg.

The reference mass serves two purposes. The first is to calibrate the span of the balance in the region that is normally depended upon for the mass depletions. The second is to define a baseline value for taring the balance while a test is run.

2.2 Automated Mass Handler

Even with the lower depletion times required by the GFS compared to the static gravimetric system, lower flow points can take a significant amount of time. Because the mass balance is being used continuously there is not an opportunity to stop and manually recalibrate the balance to account for drift the mass balance experiences.

To correct for this an automated mass handler is used to lift the gas cylinder and place a tare mass on the balance to remove drift of the balance experienced during the test. The mass of the tare mass is close to the mass of a filled bottle. The taring process can be performed without interruption to the gravimetric determination and thus can be carried out as many times as necessary during the test.

2.3 Reference Gas Cylinder and Catenary Loop

The cylinders used for GFS are of a composite material made with an aluminum internal lining, wrapped in carbon fiber in epoxy resin for strength and covered with a fiberglass lining and gel coat finish for resistance to abrasion, impact and UV degradation. The fiber composite design allows the cylinders to be lighter than most high pressure cylinders to minimize the contribution of the cylinder to the total filled cylinder assembly mass.

Attached to the reference gas cylinder is a dual stage regulator that ensures pressure stability in a catenary gas conveyance loop. The pressure must be stable to maintain a constant density in the tube defining the catenary and avoid pressure changes which could cause changes in the forces within the tube due to a bourdon tube effect. This avoids a change in the force contributed by the tube that is tared out at the beginning of the test.

2.4 Laboratory Conditions Monitor (LCM)

To provide traceability in time and also to automate ambient pressure and several temperature measurements the GFS uses the LCM. Measurements the LCM supports are:

- Time
- Cylinder temperature (IR probe)
- Ambient temperature of the air surrounding the tare mass(platinum resistance thermometer)
- Ambient temperature and humidity of air surrounding the cylinder
- Ambient temperature and humidity of air surrounding the regulator

The LCM and the mass balance are interfaced with a computer to automate data acquisition using GFS Tools™ software.

2.5 MFC Control Box and Mass Flow Controller

In order to fully automate the GFS a mass flow controller and an MFC control box interfaced with the system computer are used to set and stabilize flows. Though not a component of uncertainty these elements are necessary to maintain a constant flow at the flow point and indispensable to automate multiple gravimetric flow points without interruption.

2.6 GFS Tools software

The software supplied to control the GFS is critical in the automation and the precision of the system. GFS tools holds all measured values for the metrological elements and test parameters, controls all the data acquisition from the various sensors and mass balance, and performs all calculations for the GFS including mass totalizing for the device under test in order to compare to the GFS depletions.

2.7 Uncertainty In Flow For GFS Mass Depletions

The main uncertainties identified for a mass depletion performed by the GFS are:

- Mass
- Time
- Air buoyancy (cylinder)
- Air buoyancy (regulator)
- Type A – Contributed by the balance

When determining the uncertainty in flow as measured by the GFS it is important to understand that many of the uncertainties are only a function of the change in conditions since the most recent tare, not in the absolute value of the measurement itself. This eliminates much of the uncertainty that would otherwise be contributed by the temperature and pressure measurements.

In tables 1 and 2 lists of uncertainties are given for a 20 gram and 5 gram depletion. Note that there is very little difference in the uncertainties calculated.

GFS - 20 gram depletion in N2

Influence	value K=2 (g)	value K=1 (g)
Time	0.00020	0.00010
Resolution	0.00010	0.00003
*Repeatability	0.00048	0.00024
*Linearity	0.00037	0.00019
Tare mass	0.00028	0.00014
Buoyancy	0.00241	0.00120
	combined	**0.00125**
	expanded	**0.0025**
	expanded in %	**0.013%**

GFS – 5 gram depletion in N2

Influence	value K=2 (g)	value K=1 (g)
Time	0.00005	0.00003
Resolution	0.00010	0.00003
Repeatability*	0.00048	0.00024
Linearity*	0.00037	0.00019
Tare mass	0.00028	0.00014
Buoyancy	0.00241	0.00120
	combined	**0.00125**
	expanded	**0.0025**
	expanded in %	**0.050%**

*Includes Type A components determined experimentally

Table 2. *Uncertainty in flow for a GFS – 5 and 20 gram depletions*

In the uncertainty examples given above the change in ambient conditions is predicted to be ±5°C, ±2 kPa and any humidity from 5 to 95%RH. This is considered to be a worse case scenario since GFS is normally used in a very well controlled reference laboratory environment. The use of the taring feature resets the compensation for changing ambient conditions every time it is used.

It's apparent from the uncertainty tables that the relative uncertainty reduces as the depleted mass increases. This means at low flows it is worth the wait to deplete as much gas as possible. A 2×10^{-4} $g \cdot s^{-1}$ (10 Ncc min^{-1}) gravimetric test requires approximately 7 hours. The automated mass

handler is crucial for keeping the uncertainties low in tests this long.

2.8 Traceability To Higher Flows

Since the GFS can currently only practically define flows up to 0.2 g/s (10 Nl min^{-1}) a method was developed to extend the traceability to higher flows. This is accomplished using DHI molbloc-S sonic nozzles configured in a unique calibration chain that originates from two low gravimetric points.

The method that is used to propogate the traceability to higher flows is called successive addition. Successive addition may work with any flow device that has multiple ranges available. However, low uncertainties are only realized if the device has sufficient repeatablility. Molbloc-S sonic nozzles have repeatability better than 1 x 10^{-4} which allows for many comparisons to be performed before the repeatibility begins to significantly effect the uncertainty of the successive addition. Also, a unique characteristic of sonic nozzles is that they are not sensitive to changes in downstream pressures as long as the nozzle is sufficiently choked. This helps when downsteam pressure changes such as when the upstream sonic nozzles are used either in parallel or by themselves, or when changed from a downstream to an upstream position in the comparisons.

The method is theoretically simple. To help describe the method Figure 2 shows a link in the structure of the calibration chain that is maintained by successive addition comparisons starting with a primary, traceable GFS point at 0.2 x 10^{-4} $g{\cdot}s^{-1}$ (10 Nl min^{-1}) and linked together using the successive addition technique.

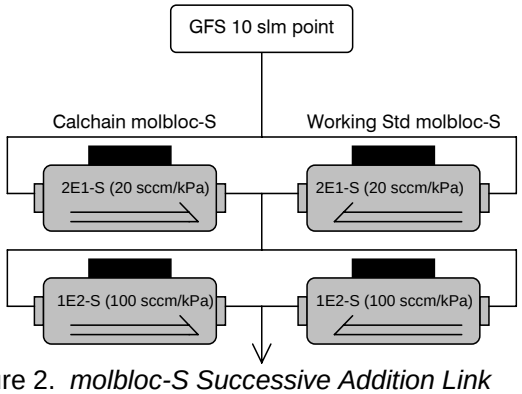

Figure 2. *molbloc-S Successive Addition Link*

In the successive addition link shown in Figure 2 there is a 5:1 ratio between ranges. This is necessary to keep the upstream pressure of the downstream nozzle low enough to keep a sufficient back pressure ratio on the upstream nozzle to ensure it is choked. As a rule for successive addition the ratio must be at least 4:1 between the ranges.

Two 2E1 (20 sccm/kPa) nozzles with a nominal range of 0.02 x 10^{-4} to 0.2 x 10^{-4} $g{\cdot}s^{-1}$ (1 to 10 Nl min^{-1}) are each compared to the GFS at 10 Nl min^{-1}. They are then put in parallel in an automated valving hardware set-up, controlled by PC software, and compared individually and also in parallel to a 1E2 (100 sccm/kPa) nozzle at various flows from 0.1 to 0.4 $g{\cdot}s^{-1}$ (5 to 20 Nl min^{-1}) including a 0.4 $g{\cdot}s^{-1}$(20 Nl min^{-1}) and 0.2 $g{\cdot}s^{-1}$ (10 Nl min^{-1}) point that connects it to the two 0.2 $g{\cdot}s^{-1}$ (10 Nl min^{-1}) gravimetric points performed on the 2E1 nozzles. This point is referred to as a transfer point. Table 4 shows the points taken for this test, the comparison performed, the nominal stagnation pressure for the upstream nozzle(s) and downstream nozzle. At least three comparisons are performed at each point as a measure of repeatability. Ranges to the left of the equal sign are upstream.

Flow Pnt	Comparison (Value is range in sccm/kPa)	Nom Prs Upstream (kPa)	Nom Prs Downstream (kPa)	BPR Upstream
5	20(1) + 20(2) = 100(1)	125	50	0.4
5	20(1) = 100(1)	250	50	0.2
5	20(2) = 100(1)	250	50	0.2
10	20(1) + 20(2) = 100(1)	250	100	0.4
10*	20(1) = 100(1)	500	100	0.2
10*	20(2) = 100(1)	500	100	0.2
20	20(1) + 20(2) = 100(1)	500	200	0.4
5	20(1) + 20(2) = 100(2)	125	50	0.4
5	20(1) = 100(2)	250	50	0.2
5	20(2) = 100(2)	250	50	0.2
10	20(1) + 20(2) = 100(2)	250	100	0.4
10*	20(1) = 100(2)	500	100	0.2
10*	20(2) = 100(2)	500	100	0.2
20	20(1) + 20(2) = 100(2)	500	200	0.4
40**	100(1) + 100(2)	200	For next SA transfer…	

* Transfer point from GFS
** Transfer point to next SA transfer

Table 3. *Successive addition test*

Starting from the transfer point, the 1E2 nozzle is characterized by the disagreement between the 1E2 and both 2E1 molblocs in the test by themselves and in parallel. The key in this calculation is that all errors are derived from the original transfer point either at higher or lower flows than the transfer point. The same test is performed for another 1E2 and it is then put in parallel with the other 1E2 to perform the next successive addition link to two 5E2s (500

sccm/kPa). This continues until the highest range nozzle is characterized.

2.9 Uncertainty in Flow for a Successive Addition Transfer

The uncertainty analysis performed for the molbloc-S calibration chain is for the discharge coefficients for each nozzle in the chain in the range they are tested. To determine these coefficients the successive addition flow points are used. The uncertainties identified for a single successive addition test are:

- Original reference flow from the GFS
- Transfer point pressure
- Transfer point temperature
- Repeatability of the test (Type A)

A unique characteristic of the successive addition transfer of flow traceability is there is no contribution of uncertainty in linearity of the molbloc-S. As long as the transfer points are maintained at stagnation pressures that are reasonably high in the range of the pressure transducers used, and the temperatures are reasonably stable, the uncertainties from temperature and pressure are nearly insignificant.

Normally because of the low uncertainties contributed by repeatability of the molbloc-S, the dominant uncertainty is the reference uncertainty at the beginning of the successive addition. Table 5 shows the uncertainties from a typical successive addition for the previous example given, i.e. the 2E1 to 1E2 successive addition link.

1E2-S (1)	(% rdg)	1E2-S (2)	(% rdg)
Ref Uncert	0.0389	Ref Uncert	0.0389
Press	0.0030	Press	0.0030
Temp	0.0075	Temp	0.0075
StdDev	0.0009	StdDev	0.0015
Final Uncertainty		Final Uncertainty	
	0.0397		0.0397

Table 5. *Successive addition uncertainty example*

The uncertainties from Table 5 are not expanded and are shown for k=1. Note that the final uncertainty in the transfer is only slightly inceased from the original reference uncertainty from the GFS.

With the data obtained from the successive additions performed throughout the calibration chain the discharge coefficients are calculated in the range the successive addition was performed. When these coefficients are determined they are done so with a fit to the inverse square root of Reynolds number and forced to fit through the transfer points for that specific molbloc. This ensures there is no propogation of uncertainty throughout the calibration chain from any non-linearities that may be present.

Because ranges are skipped to obtain a ratio of 4:1 or 5:1 there are two paths of successive addition that currently end at 52 $g \cdot s^{-1}$ and 105 $g \cdot s^{-1}$ (2500 and 5000 Nl min^{-1}). Once both paths are characterized, the overlapping ranges from all the nozzles on one side can be comapred to the other side, i.e. the 1E1 compared to the 2E1, 5E1 compared to the 2E1 and the 1E2. Agreement between the two links are used as tolerance criteria for the succesive addition tests.

3. Verification of Traceability and Uncertainty

DHI takes every opportunity to verify traceability and estimated uncertainties in the flow measurements reported. Though many opportunities arise, an example for the GFS and the molbloc-S calibration chain is presented here.

To verify the uncertainty of the GFS, comparisons were performed with the existing static gravimetric system that has been in place at DHI for over 10 years. Though many points have been compared, Table 6 shows the agreement through gravimetric points taken on a 1E2 (100 Ncc min^{-1} full scale) laminar flow element, SN 495, that is a reference in the DHI laboratory. Shown are the disagreements between the 1E2 (laminar) and the static gravimetric for two comparisons performed at 100 Ncc min^{-1}, and two GFS comparisons performed on the same molbloc at the same nominal flow.

Date Tested	GFS (% of rdg)	Static (% of rdg)
28-Oct-05	0.095	---------
5-Jan-06	---------	0.07
31-Oct-05	0.077	---------
18-Jan-06	---------	0.07

Table 6. *GFS/Static gravimetric comparison*

The agreement is inside of 0.025% of reading and well within the combined uncertainties of the

systems. For the static gravimetric tests the amount of mass depleted was 161 grams of nitrogen. This is a duration of 20 hours for the depletion to which is added time for the before and after bottle weighing. The two GFS points for 100 Ncc min^{-1} were accomplished in approximately 80 minutes with only a 5 gram depletion.

A recent opportunity for the verification of traceability was presented by a calibration that was performed at DHI at the highest flows achievable and was promptly re-tested at CEESI against their primary B reference[6]. Table 7 shows the results of the calibration, the reference uncertainties and agreement through the test.

	CEESI	DHI		
Nominal Q (Nl min^{-1})	Actual Q (kg/s)	Actual Q (kg/s)	Difference (mg/s)	Difference (% or rdg)
475	0.010566	0.010582	0.016	0.15%
475	0.010532	0.010542	0.010	0.09%
2066	0.038821	0.038802	-0.019	-0.05%
2066	0.038437	0.038460	0.023	0.06%
2066	0.038810	0.038806	-0.004	-0.01%
3658	0.069618	0.069541	-0.077	-0.11%
3658	0.072973	0.072910	-0.063	-0.09%
5250	0.12396	0.124142	0.182	0.15%
5250	0.11433	0.114363	0.033	0.03%

DHI Uncertainty: +/- 0.2% reading @ K=2

CEESI Primary B Uncertainty: +/- 0.1% reading @ K=2

Table 7. *High flow comparison with CEESI*

4. Conclusion

With the development and implementation of the GFS there is much less dependency on the molbloc-L calibration chain[2] . This is due to the realization of lower uncertainties in gravimetric flow measurements, the speed at which those measurements are made and the automation capabilities of the GFS. The GFS is also used as the starting point for the molbloc-S successive addition calibration chain which could not be accomplished with the older static gravimetric system because sonic nozzles cannot totalize mass starting from zero flow.

With the implementation of the molbloc-S (sonic nozzle) calibration chain the lower uncertainties realized from the GFS are available up to much higher flows.

The successive addition technique is unique because it is very successful in extending traceability to entire ranges and much higher flows starting from one traceable point from the GFS. The extent of the range of flow capable with successive addition is only limited by the ancillary hardware required to supply and remove the gas from the DHI metrology laboratory.

References

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