Comparison Demonstrates Factor of Three Improvement in Gas Flow Measurements

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A comparison of seven gas flow calibration laboratories piloted by NIST used four laminar flow meters as the transfer standards for nine nitrogen gas flows ranging from 1 sccm to 10 slm.[†] The comparison reference value was calculated from the uncertainty weighted average of the three participants with independent flow traceability chains. The 63 comparison results were evaluated by the traditional criterion (normalized degree of equivalence, $|E_n| \leq 1$) and also by a probabilistic criterion that allows the possibility of inconclusive results. The $|E_n| \leq 1$ criterion determined that 2 of the 63 results were outside of uncertainty expectations. The probability-based criterion found the same 2 results were outside of uncertainty expectations and 3 results were inconclusive. Based on these results and other evidence, all participants were found proficient over the range of flows they tested. A comparison of the present results to those from a similar comparison between four of the same participants conducted in 2003 shows that the labs have improved their flow measurement capabilities by a factor of three.

Introduction

The US Department of Defense (DOD) Calibration Coordination Group (Physical/Mechanical) asked NIST to assess the proficiency of a set of gas flow laboratories. NIST piloted a comparison among the participating laboratories to test their uncertainty specifications and capabilities using transfer standards, protocols, and calculation methods developed for international key comparisons during the past two decades by the members of the Working Group for Fluid Flow (WGFF) and the Consultative Committee for Mass and Related Quantities (CCM). An overview of the consensus comparison methodology is given in reference [1] and guidance documents and templates can be found in the CCM and WGFF web pages [2].

In 2003, NIST also piloted a gas flow comparison involving four of the same labs [3]. The gas flow standards in all of the labs have evolved significantly over those 16 years, improving in uncertainty, maintenance costs, and ease of operation. NIST automated and reduced the

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[†] sccm = standard cubic centimeter per minute at reference conditions of 0 °C and 101.325 kPa.
slm = standard liters per minute at the same reference conditions.

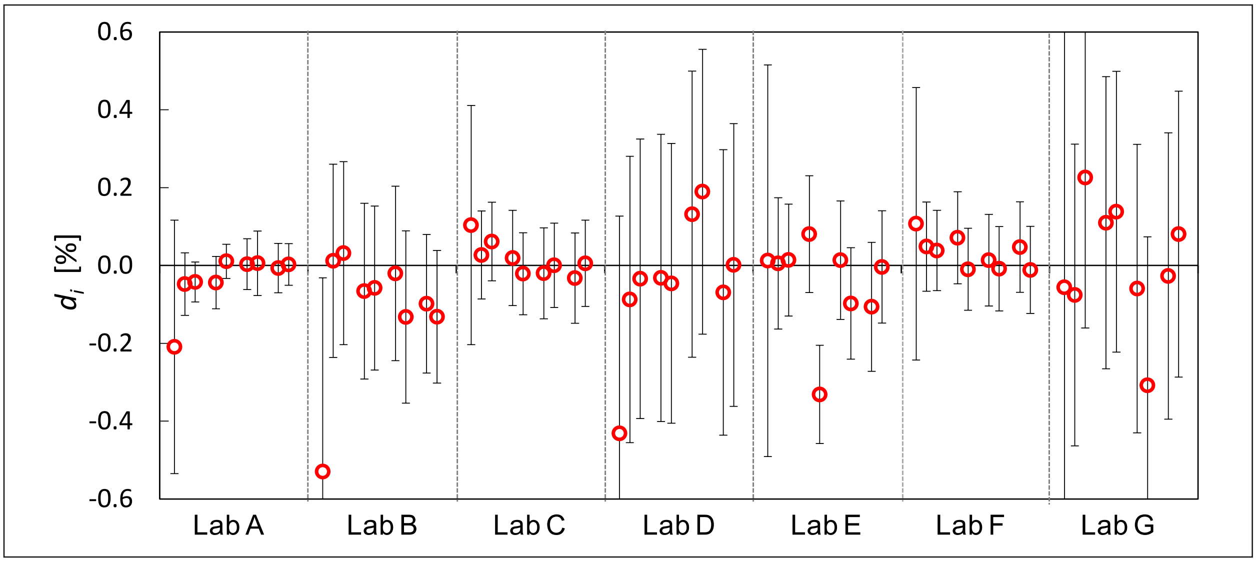

Figure 1. Degrees of equivalence for each laboratory with respect to the comparison reference value (CRV). The symbols represent the nine flow set points used in the seven participants' labs. The error bars show the expanded uncertainty of the degree of equivalence for each calibrated value. Degree of equivalence d_i is defined as the difference between each participant's result and the comparison reference value. In general, error bars crossing the d_i = 0 line indicate a comparison result within uncertainty expectations.

uncertainty of its primary standards by switching from bell and piston provers (0.19 %*) [4] to a *PVTt* standard (0.025 %) [5]. NIST's uncertainty reduction and the documented long-term calibration stability of commercially available flow meters [6] enabled the DOD Primary Standards Labs (PSLs) to send working standards (Molblocs† and critical flow venturis) to NIST for calibration and also enabled the PSLs to use the working standards to calibrate their customers' instruments. This approach is easier to maintain and operate than the piston and bell provers they used in the past but still meets DOD uncertainty goals. (Some PSLs still maintain primary standards such as piston or bell provers.) DOD secondary labs also use sets of working standard Molblocs and critical flow venturis to calibrate other flow meters.

Since 2003, the participants have also responded to their calibration customers' demands and extended their flow capabilities to lower flows: the minimum flow set point in the 2003 comparison was 40 sccm; the present minimum is 1 sccm. Flows below 10 sccm are challenging to calibrate due to leaks and temperature effects.

List of Participants, Facilities Used, Circulation Scheme

The participants and the abbreviations used for their labs are given in the list of authors' affiliations on the title page of this report or in Table 1. The transfer standards circulated in a single loop between the participants between June 2018 and August 2019.

Most participants used working standard Molbloc (MB) laminar flow meters (LFMs), either directly upstream or downstream from the transfer standard. Except for the case of Fluke, the working standard MBs are traceable to NIST and/or AFMETCAL gas flow standards via periodic calibrations (dependent traceability). NIST used the 34 L *PVTt* and Rate of Rise (RoR) standards [7, 8], Fluke used their dynamic Gravimetric Flow Standard (GFS) [9], and APSL used its GFS and Constant Pressure Flow Meter (CPFM) [10]. The NIST, Fluke, and APSL standards have independent traceability to mass, time, temperature, pressure, and humidity and their data were used to calculate the comparison reference value.

Transfer Standard and Comparison Protocol

The transfer standard (TS) package included four laminar flow meters (Molbloc-L, manufactured by Fluke) with full scale flows of 10 sccm, 100 sccm, 1000 sccm, and 10 slm (Figure 2). Laminar flow meters use variations of the Hagen-Poiseuille equation to calculate flow from the gas species (in this case nitrogen), the absolute pressure

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* Unless otherwise noted, all uncertainties are expanded, *k* = 2, approximately 95 % confidence level values.

† Certain commercial entities, equipment, or materials may be identified in this document in order to describe an experimental procedure or concept adequately. Such identification is not intended to imply recommendation or endorsement by the National Institute of Standards and Technology, nor is it intended to imply that entities, materials, or equipment are necessarily the best available for the purpose.

Participant	Type of reference standard	Reference flow uncertainty (*k*=2, %)	Date of test	Independent traceability?
NIST	34 L *PVTt* and RoR	0.12 to 0.025	Jun 2018	Yes
AFMETCAL	Downstream MBs	0.24 to 0.14	Aug 2018	No, to NIST/Fluke
APSL	GFS, CPFM	0.1	Sep 2018	Yes
Robins	Upstream MBs	0.35	Nov 2018	No, to AFMETCAL
NPSL	Upstream MBs	0.24 to 0.1	Feb 2019	No, to NIST
Fluke	GFS, Downstream MBs	0.2 to 0.1	Apr 2019	Yes
Tinker	Downstream MBs	0.35	Aug 2019	No, to AFMETCAL

Table 1. Participants, facilities used, reference standard uncertainty, dates of test, and traceability links between participants. See title page for explanation of acronyms.

and temperature of the flowing gas, and the differential pressure between the upstream and downstream side of a narrow flow passage. Instrumentation for pressure and temperature measurements and the flow calculation (a Molbox1+) was shipped as part of the TS to reduce uncertainties that would be introduced by using different instrumentation in each laboratory. The Molbox calculates differential pressure by subtracting absolute pressure measurements made on the downstream and upstream sides of the LFM. The comparison protocol reduced errors in the differential pressure by "taring" the two pressure sensors while flowing at each set point before collecting data. Similar instrumentation and protocols have been used successfully in other comparisons [3, 11, 12].

A control box for mass flow controllers was included with the transfer standard equipment and used to set and maintain the comparison flow set points. Accessories necessary for operating the transfer standard, e.g. pressure regulators, shut-off valves, mass flow controllers, and filters, were also included (Figure 3). The TS pressure regulator was set to 350 kPa and acceptable pressures at the outlet of the TS ranged from 90 kPa to 300 kPa. This allowed the TS to be calibrated with working standard Molblocs positioned either up or downstream of the TS. Figure 4 shows the NPSL upstream MB working standard installed in series with the transfer standard.

The nominal flow set points are listed in Table 2. Note that three of the set points (10 sccm, 100 sccm, and 1000 sccm) were measured by two LFMs, one used at 10 % of its full-scale flow, the other at 100 % of full scale. The 1 sccm set point was outside the operating range of two participants and they tested at 2 sccm instead. The slope of the error curve for the 10 sccm LFM was small enough that the measurements made at 2 sccm could be handled without correction along with the other labs' 1 sccm results without introducing significant uncertainty to the data processing.

The protocol called for 15 min stabilization time at each set point, then five, 60 s long averages were collected using the Molbox averaging capability. This was done on two different occasions. Transfer standard flow, pressure and

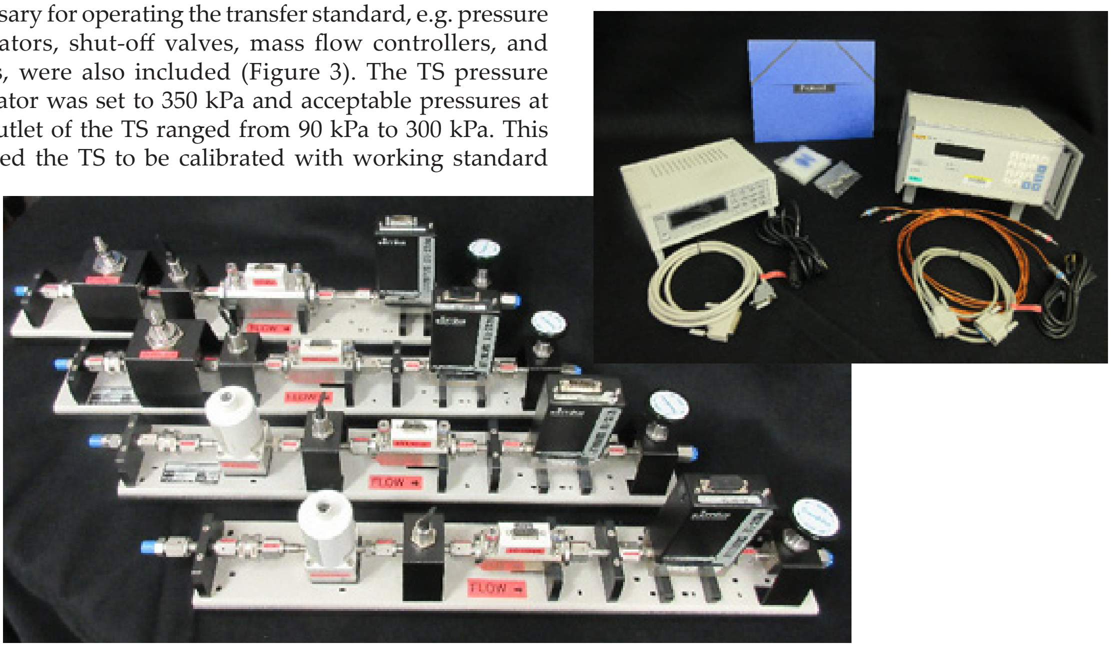

Figure 2. Pictures of the transfer standard package.

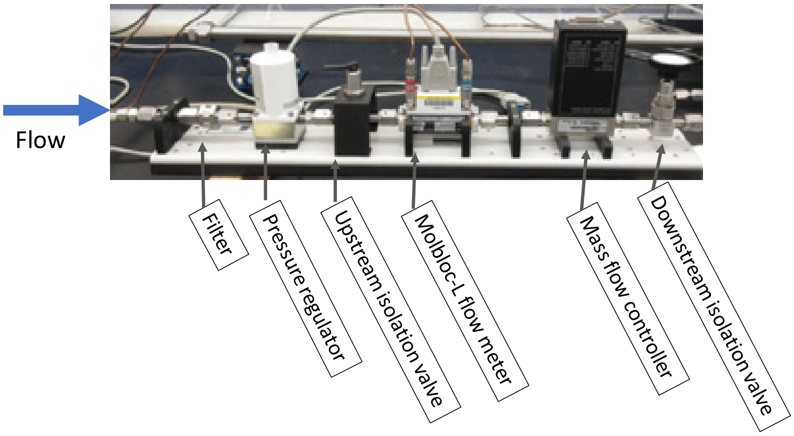

Figure 3. A picture of one of the four transfer standard laminar flow meters. The regulator and mass flow controller maintain reproducible pressure conditions at the laminar flow meter.

temperature, reference flow, and uncertainty data were reported to the pilot lab in a spreadsheet template. The data were not shared between the participants. The percent difference between the reference and the TS was calculated for each 60 s average. The resulting 10 points at each set point were averaged to deliver the data presented in this report. The standard deviation of the mean of the ten 60 s averages was used to quantify the reproducibility (Type A uncertainty) of the averages produced in each lab.

Uncertainty Due to the Transfer Standard

The uncertainty introduced by the transfer standard must be considered in a comparison because TS calibration drift or sensitivities can be mistaken for lab-to-lab differences or accidentally cause labs to seemingly agree with each other when they don't [13]. In practice, much of the work of a comparison involves characterizing the sensitivities of the TS to operating conditions so that the effects of these conditions can be corrected or treated as quantified uncertainties. For this comparison, the variables considered include gas temperature, pressure, gas composition, leaks, and the TS reproducibility.

Reproducibility: The TS uncertainty was primarily due to its long-term calibration instability, particularly at the lowest flow for each LFM (10 % of full scale) where differential pressure uncertainties have the largest effect. The calibration instability was quantified by the standard deviation of four or more calibrations made by the pilot laboratory before and after the TS was shipped between the participants. An example of the multiple calibration sets and their standard deviation at the 10 %, 50 %, and 100 %

Transfer Standard Full Scale	**Low Set Point (sccm)**	**Medium Set Point (sccm)**	**High Set Point (sccm)**
10 sccm	1	5	10
100 sccm	*10*	50	100
1000 sccm	*100*	500	1000
10 slm	*1000*	5000	10,000

Table 2. The flow set points used in the comparison. The italicized values were given less importance for uncertainty reasons (see text).

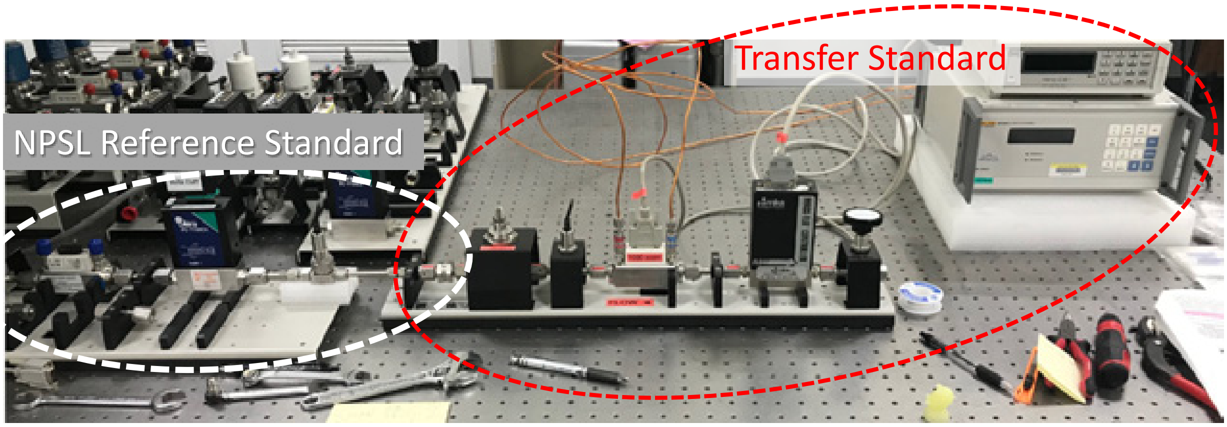

Figure 4. The transfer standard being calibrated with their NPSL upstream Molbloc working standard in February 2019.

of full-scale set points is shown in Figure 5. The Y-axis is the difference between the LFM and the NIST *PVTt* gas flow standard, ε. By design, measurements were made with two different LFMs at the 10 sccm, 100 sccm, and 1000 sccm. The larger LFM operated at 10 % of its full scale (FS); the smaller at 100 % of FS. Because the reproducibility uncertainty was smaller for the LFMs used at 100 % of FS, we focused on those measurements and only considered the 10 % FS results to study the TS performance and, if necessary, as confirmation of conclusions from the 100 % FS measurements. Of course we did not have that option at the lowest set point of the comparison (1 sccm) and accepted the larger TS uncertainty there.

Temperature: Prior research found temperature sensitivity of 0.01 % / °C for a particular Molbloc [14]. We reviewed historic NIST calibration data for several Molblocs over the lab temperature variations that occur in the NIST lab and found temperature sensitivity ranging from negligible levels to as large as 0.01 % / °C. With the exception of one participant, the LFM temperature measurements in each lab were 23.12 °C ± 1.87 °C. One participant's temperatures ranged between 17.76 °C and 21.75 °C. Based on these figures, a temperature sensitivity uncertainty of 0.02 % was assumed at 95 % confidence level.

Pressure: Each TS had a pressure regulator and mass flow controller on either side of the LFM (Figure 3). The regulators maintained consistent pressures for each flow set point, thereby minimizing pressure effects on the TS flow meters. In actuality, the pressure at the MB inlet varied between 198 kPa and 374 kPa depending on the participant and the flow set point. A review of NIST's Molbloc calibration data at various pressures led to an estimated pressure sensitivity uncertainty of 0.03 % at the 95 % confidence level.

Composition: Participants used nitrogen gas cylinders with manufacturer specified purity of 99.995 % or higher. Gas manufacturers list possible contaminants as hydrocarbons, oxygen, carbon dioxide, carbon monoxide, and water. An analysis based on the worst-case impurities determined that their effect on the gas's viscosity and density was negligible compared to other uncertainty components.

Leaks: Participants followed a leak check procedure specified in the comparison protocol. They pressurized the TS to 350 kPa between the closed isolation valves (Figure 3), tared the Molbox, and observed 60 s averages of the flow indicated by the Molbloc. The leak values they measured are listed in Table 3 as a percentage of the minimum flow set point used (50 % of the LFM full-scale except for the 10 sccm LFM). The uncertainty due to leaks attributed to the transfer standard was assumed to be 0.02 % of reading at the 95 % confidence level based on leak tests performed at the pilot lab prior to circulation. In four cases (bold font in Table 3), the leaks measured in the participants' labs were larger than 0.02 %. However, the < 0.02 % leak criterion was achieved for each LFM in several labs. This implies that the larger leaks were in the participants' setups but not in any of the transfer standards.

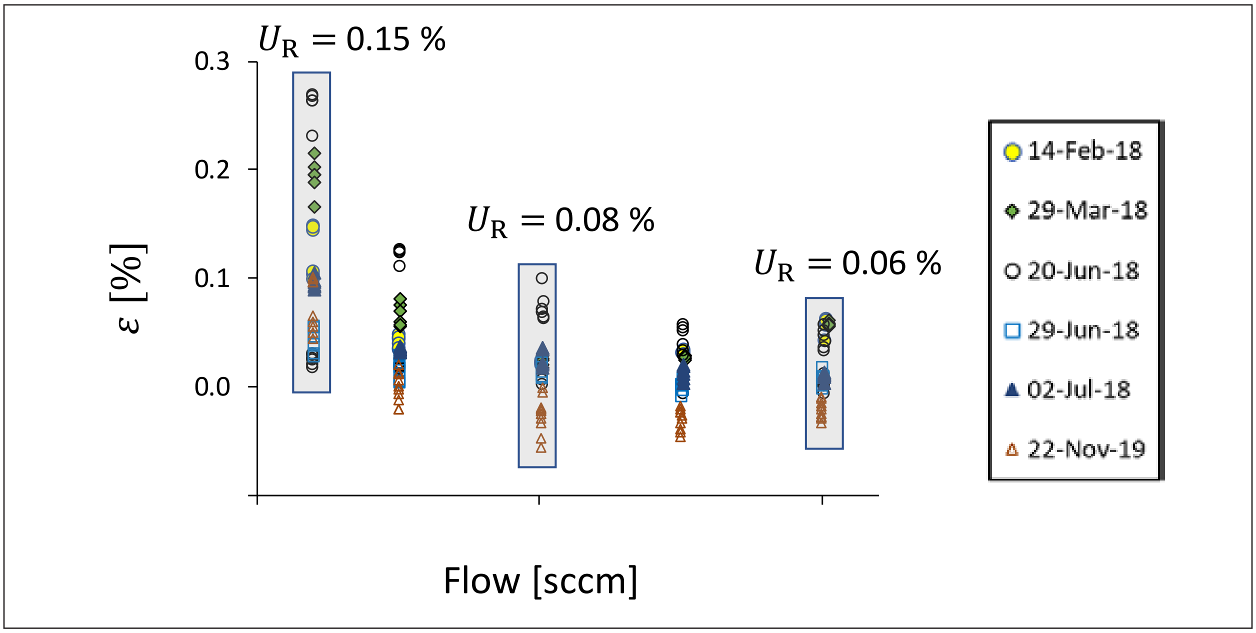

Figure 5. Six calibration data sets measured at NIST for the 10 slm LFM before and after circulation of the transfer standard. The 95 % confidence level reproducibility uncertainty was quantified by doubling the standard deviations of 4 or more calibrations of each LFM at NIST before and after circulation of the TS.

Lab	10 sccm (% of 1 sccm)	100 sccm (% of 50 sccm)	1000 sccm (% of 500 sccm)	10 slm (% of 5 slm)
A	0.020	0.004	0.004	0.004
B	0.010	-0.002	0.000	0.000
C	-0.004	**0.106**	-0.001	0.001
D	**0.050**	0.004	0.002	0.004
E	0.010	0.020	-0.002	0.000
F	0.010	0.001	0.001	0.001
G	0.010	**0.128**	0.006	**0.024**

Table 3. Leaks measured by participants as a percentage of the flow set points.

The uncertainty components for reproducibility, leaks, and temperature and pressure sensitivity were combined by root-sum-of-squares to arrive at the transfer standard uncertainty for each flow set point (U_{TS}) that ranged from 0.37 % to 0.06 %.

Data Processing and Computation of the Comparison Reference Value (CRV)

The protocol, report format, and the data processing for this comparison used templates developed during the past decade for international flow comparisons. The templates are available from NIST for others to use. The data processing followed the methods documented by Cox [15] to calculate the comparison reference value (CRV) using Procedure A (uncertainty weighted mean and χ-squared consistency test). The necessary inputs are the measurements made by the participant, the base uncertainty of the lab*, the reproducibility of the transfer standard for each lab, and the uncertainty of the transfer standard.

Four of the seven participants used reference standards with traceability to NIST or AFMETCAL, so the three labs with independent traceability (NIST, APSL, and Fluke) determined the uncertainty weighted CRV. The uncertainty weighted mean gives greater significance to labs with lower uncertainty estimates. Whether or not a participant has independent traceability impacts the calculation of the comparison reference value and the uncertainty of the degree of equivalence: dependent labs are not included in the reference value calculation because it would be analogous to including the lab that is the source of traceability more than one time in the averaging process. The χ-squared consistency test removes labs from the reference value calculation if their difference from other independent results is larger than expected for their uncertainty estimate. In this comparison, the three independent labs passed the χ-squared consistency test at all flow set points, so there was no need to remove discrepant results.

Results

The comparison results shown in Figure 1 include only the results from duplicate set points made at 100 % of the LFM full scale flow. Table 4 lists the most commonly used means of assessing comparison results, the standardized degree of equivalence for laboratory i, $E_{\mathrm{n},i} = d_i / U_{d_i}$, where d_i is the degree of equivalence and U_{d_i} is the uncertainty of the degree of equivalence [13]. Values of $|E_{\mathrm{n}}| \leq 1$ indicate that a participant's result agrees with the comparison reference value within uncertainty expectations. The same criterion can be applied visually to Figure 1 by seeing whether or not the error bars of each participant cross the $d_i = 0$ line (which represents the CRV).

In this comparison, all results passed the $|E_{\mathrm{n}}| \leq 1$ criterion except Lab B at 1 sccm and Lab E at 100 sccm. We note that because uncertainties are specified at the 95 % confidence level, we would expect 5 % of the comparison results from a proficient lab to fail the $|E_{\mathrm{n}}| \leq 1$ criterion. We also considered other evidence at the set points where $|E_{\mathrm{n}}|$ was greater than 1. We observe that the reference flow standards at Labs F and G are traceable to (i.e., periodically calibrated by) Lab B and Labs F and G both passed the $|E_{\mathrm{n}}| \leq 1$ criterion at 1 sccm. This may indicate that Lab B's result at 1 sccm is not indicative of their typical performance. The 1 sccm set point is the most challenging measurement of this comparison because it has the highest sensitivity to possible leaks and to the transfer standard's stability. Note also that the second Lab E result at 100 sccm that used a Molbloc at 10 % of full scale (not shown in Figure 1) passed the $|E_{\mathrm{n}}| \leq 1$ criterion. These factors and the large fraction of passing results over the range of flows tested lead us to believe that all of the labs are proficient.

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* The base uncertainty of a flow reference does not include the Type A uncertainty for the best existing device, see *WGFF Guidelines for Calibration Measurement Capabilities Uncertainty and Calibration Report Uncertainty*, October 21, 2013, https://www.bipm.org/utils/en/pdf/ccm-wgff-guidelines.pdf.

Set Point [sccm]	Lab A	Lab B	Lab C	Lab D	Lab E	Lab F	Lab G
1	*-0.642*	**-1.063**	0.338	*-0.773*	0.024	0.306	-0.078
5	-0.597	0.048	0.237	-0.237	0.032	0.422	-0.196
10	-0.828	0.134	0.608	-0.095	0.097	0.372	0.584
50	*-0.657*	-0.293	0.157	-0.087	0.536	0.603	0.293
100	0.243	-0.276	-0.202	-0.128	**-2.628**	-0.095	0.383
500	0.050	-0.092	-0.172	0.358	0.090	0.114	-0.160
1000	0.068	-0.598	0.003	0.517	-0.682	-0.076	-0.808
5000	-0.106	-0.552	-0.280	-0.189	-0.641	0.405	-0.073
10000	0.045	-0.775	0.050	0.003	-0.027	-0.104	0.219

Table 4. Standardized degree of equivalence between a lab i and the key comparison reference value, $E_{n,i}$ for the set points plotted in Figure 1. Failed results have bold font, inconclusive results are in italics font.

Inconclusive Results

Members of the Working Group for Fluid Flow (WGFF) have performed over 30 international comparisons during the 21 years since its formation in 2000. The present comparison has followed the recommendations of the WGFF. Specifically, the WGFF and we recognize the importance of quantifying the uncertainty contributed by the transfer standard and including it in the analysis of comparison results. The WGFF observed that if a transfer standard has uncertainty that is large relative to the participants' uncertainties, the $|E_n| \leq 1$ criterion can give passing (or failing) results that should be deemed inconclusive. For many measurands, transfer standards with uncertainties comparable to or better than the reference standards being compared do not exist. In this situation, a comparison using the traditional $|E_n| \leq 1$ criterion, and very large transfer standard uncertainty ensures that all participants will pass, thereby completely undermining the purpose of comparisons.

To deal with the limitations of transfer standards, reference [13] proposed several new approaches that assess comparison results as passing, failing, or inconclusive (instead of only passing or failing) and those approaches were applied to this comparison. "Criterion B" in reference [13] considers the $|E_n| \leq 1$ assessment conclusive if the ratio of TS uncertainty U_{TS} to the participant's base uncertainty is 2 or less. For the results shown in Figure 1, $U_{TS} / U_{base,i} <$ 1.84 except for Lab A and Lab C where it is as large as 3.7. Eight of the 63 entries in Table 4 are inconclusive according to Criterion B. "Criterion D" applies a probability-based approach that finds three results in Table 4 inconclusive: Lab A at 1 sccm and 50 sccm and Lab D at 1 sccm. Labelling these results inconclusive indicates that we should not rely on them to assess the participant's proficiency because of the uncertainty of the comparison process.

Youden Analysis

The measurements made with two different LFMs at 10 sccm, 100 sccm, and 1000 sccm are statistically independent and can be used to generate Youden plots where the degrees of equivalence from one LFM (at 10 % full scale, d_{10i}) are plotted on the x-axis and those from the other LFM (at 100 % of full scale, d_{100i}) on the y-axis. The point (0, 0) corresponds to the comparison reference value and the distance along the diagonal line indicates the difference between the lab and the CRV. A point falling on the diagonal line indicates that a consistent degree of equivalence for that lab was measured by both LFMs. The distance away from the diagonal line is a measure of the randomness of the entire comparison process (due to either or both the reference and transfer standards).

A similar NIST piloted comparison with four of the same participants as this comparison was conducted with a different set of Molblocs between March 2002 and September 2003. The improvement in the reference standards during the past 16 years is apparent in Figure 6. This "comparison of the comparisons" is a Youden plot for the 1000 sccm set point showing only the four common participants. In the 2003 comparison the point at (0, 0) is NIST's result; in 2019, the point at (0, 0) is the uncertainty weighted mean of independent participants. In 2003, the lab farthest from (0,0) was located at (-0.43 %, -0.37 %); in 2019, it is (-0.15 %, -0.13 %), a nearly three-fold improvement in agreement. The average of the degrees of equivalence for these four labs has dropped from -0.14 % to -0.04 %, a more than three-fold improvement. The error bars on the data points represent the standard deviation of the data collected in the participating labs; they show the irreproducibility of the reference and transfer standards. For many of the labs, the error bars for the 2019 comparison have improved and are too small to be visible in this plot.

Summary and Conclusions

We conducted a comparison of seven gas flow calibration laboratories using four Molbloc laminar flow meters that circulated between the labs from June 2018 to August 2019. The range of flows was 1 sccm to 10 slm. The uncertainty components for the TS included pressure and temperature sensitivity, gas purity, leaks, and the long-term calibration stability measured in the pilot lab. To minimize the effects of the transfer standard's uncertainty, the comparison used measurements made at 50 % and 100 % of the transfer standards' full scale (except at the smallest flow set point of 1 sccm). The 95 % confidence level uncertainty of the transfer standard was 0.37 % at the lowest flow and was as small as 0.06 % at the TS full scale flows.

The protocol, spreadsheets for sending data to the pilot lab and performing the comparison calculations, and the format of the comparison report followed templates developed for comparisons between national metrology institutes organized by the Working Group for Fluid Flow and the Consultative Committee for Mass under the guidance of the International Bureau of Weights and Measures (Bureau International des Poids et Mesures). These templates improved the efficiency of the comparison and reduced errors in calculations. These templates are available through the references [2] or directly from the corresponding author of this paper.

The comparison showed agreement between the participants within uncertainty expectations and demonstrated proficiency over the tested range for all participants. The $|E_n| \leq 1$ criterion determined that 2 of the 63 cases were outside of uncertainty expectations. The probability-based criterion from reference [13] also determined that 2 of the 63 cases were outside of uncertainty expectations and 3 cases were inconclusive. None of the > 0.02 % leak values in bold font in Table 3 correlate with the two comparison results that had $|E_n| > 1$.

Upon comparing these results to a similar comparison performed in 2003, we conclude that the improvements to the methods during the past 16 years have improved agreement between the four shared participants by a factor of 3 or more while also reducing maintenance costs in the DOD labs.

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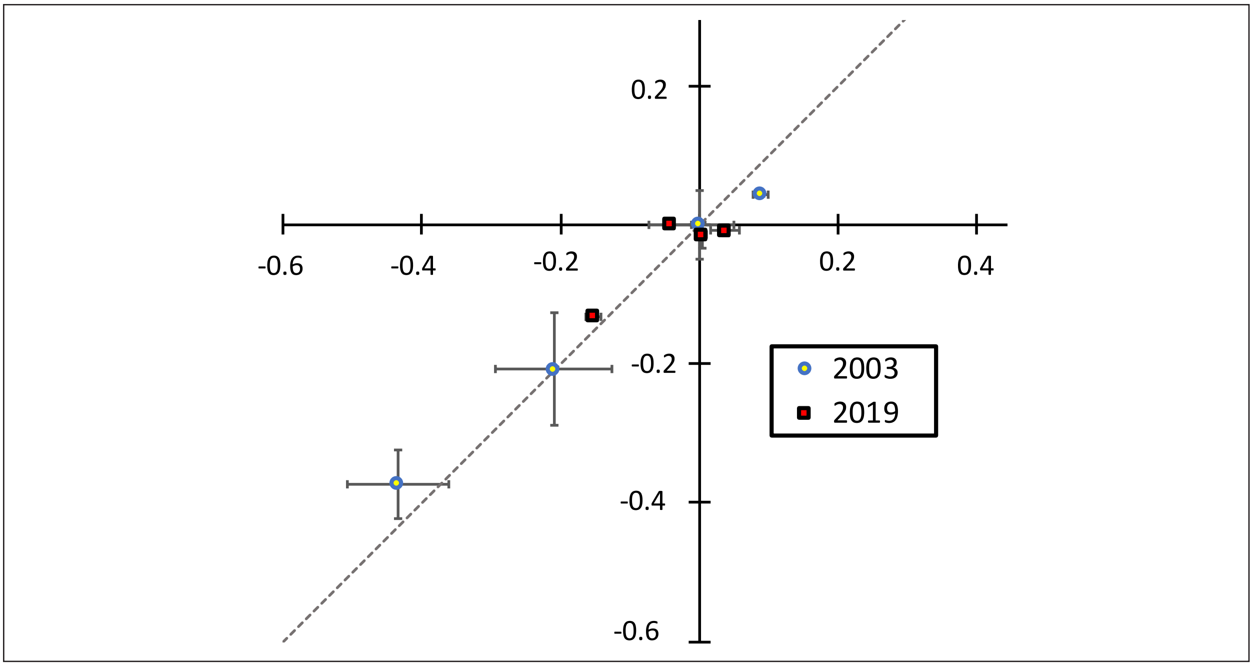

Figure 6. A Youden plot for the 1000 sccm flow set point showing results for the four labs that participated in both the 2003 and 2019 gas flow comparisons.

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