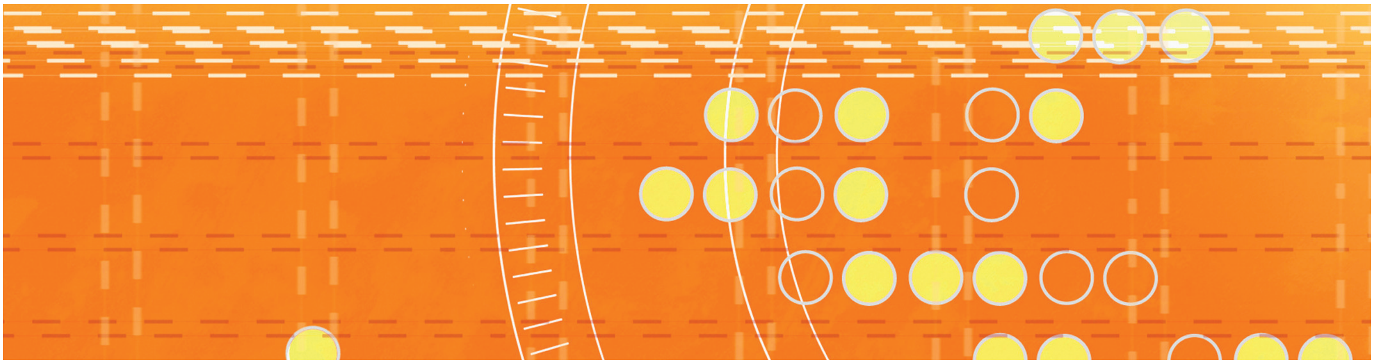

Interlaboratory Comparison of Helium Low Gas Flow Measurements in the Range 10^{-13} to 10^{-11} mol/s (10^{-9} to 10^{-7} cm^3/s)

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Abstract: An interlaboratory comparison (ILC) of helium low-flow measurement capability was recently completed. The ILC was piloted by the National Institute of Standards and Technology (NIST). The majority of the data was taken over a period of approximately two years; a final round of data was taken by the pilot laboratory approximately three years after the data from the other participants were collected. The participants included a mix of ten industrial and metrological calibration laboratories within the United States. The comparison was performed using three helium permeation leak artifacts having different flow rates within the range of 10^{-13} mol/s to 10^{-11} mol/s (10^{-9} to 10^{-7} cm^3/s at 0 °C and 101.33 kPa). Each participant laboratory was required to measure the helium flow rate of all three artifacts at a nominal artifact temperature of 23 °C, and to submit a report of their results, including a complete uncertainty analysis, to the pilot laboratory. The pilot laboratory made measurements at the beginning, end, and at three other times during the course of the comparison. The reference values used to compare the flow measurements from each laboratory were defined as the weighted linear fit to the pilot laboratory measurements. Analysis of the comparison results is presented along with an assessment of each participant's equivalence to the reference value for each flow artifact. The goal of this work is to help the participant laboratories to quantify their capability in the area of helium low-flow measurements. Information of this sort is often required for quality system documentation and certification by organizations such as the American Association for Laboratory Accreditation (A2LA) and the National Voluntary Laboratory Accreditation Program (NVLAP).

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1. Introduction

Calibrated helium leak artifacts are often used as flow standards in a variety of industrial and research applications. For example, one common use of a calibrated helium leak artifact is as a calibration standard for a helium leak detection system. A helium leak artifact is typically a small, pressurized bottle from which helium continually leaks or diffuses through, for example, an orifice, capillary, polytetrafluoroethylene (PTFE) element, or glass element. Many private and government laboratories within the United States provide calibration services for helium leak artifacts. These laboratories are capable of measuring the leak rate of a helium leak artifact to within a specified uncertainty.

An interlaboratory comparison (ILC) was begun in November 2003 to appraise and compare laboratory performance in measuring helium flow rates in the range of 10^{-13} mol/s to 10^{-11} mol/s (1 mol/s = 2.241×10^4 cm^3/s at 0 °C and 101.33 kPa). A total of ten calibration laboratories participated in this comparison, including the National Institute of Standards and Technology (NIST), which served as the pilot laboratory for the study. The participants included calibration laboratories from the aerospace industry, vacuum technology companies, and government research laboratories, all located within the United States. Table 1 lists the participants of this interlaboratory comparison.

In what follows, the protocol, procedures, analysis, and results of this interlaboratory comparison are presented. With the exception of NIST, the data from the participating laboratories will not be identified.

2. Comparison Protocol

The pilot laboratory developed a protocol for the comparison that explicitly stated the procedures that each participant was to follow. Each participant was encouraged to comply with the details of the protocol as closely as possible so that the leak artifacts were handled and calibrated in a consistent manner. The following is a brief summary of the protocol and the mechanics of running the comparison.

Three glass-element permeation leak artifacts were used for this comparison; two of these artifacts had a calibration history of approximately ten years in the NIST leak calibration service, while the third artifact was new. Since the flow rate from this type of helium permeation leak artifact is very temperature dependent (3 % to 4 % change in flow rate per °C change in temperature), it was important that the reported leak rate from each participant be referenced to the same temperature. A temperature of 23.0 °C (296.15 K) was chosen as the reference temperature for this study. The flow from a permeation leak as a function of temperature may be modeled by: [1]

$$F = ATe^{-B/T} \,. \tag{1}$$

In equation (1), F is the flow rate of helium in mol/s, A and B are empirically determined constants, and T is the absolute temperature of the artifact. The pilot laboratory measured the temperature dependence of the flow rate for each artifact to determine the B-coefficients, and corrected the data from the participating laboratories that was reported at temperatures other than 23.0 °C.

The three leak artifacts used for the ILC were identified as NIST 10, NIST 11, and NIST 12 by labels secured to the artifacts. The participants were told only that the artifacts had leak rates in the range of 10^{-13} mol/s to 10^{-11} mol/s at 23 °C (10^{-9} cm^3/s to 10^{-7} cm^3/s at 0 °C and 101.33 kPa). From November 2003 to August 2008, the pilot laboratory performed five calibrations on each of the three leak artifacts: three calibrations used a constant-pressure flowmeter as a working standard to generate a known flow of helium to compare against the artifacts [2, 3], and two comparison calibrations that used a previously calibrated leak artifact as a working standard. [4] The later comparison calibrations that used leak artifacts as working standards typically had a higher uncertainty than did the comparisons to the flowmeter, but served as a fast check of the health of the leak artifacts. In addition, the NIST artifact comparison system, which is capable of determining the leak rate as a function of temperature, was used to establish the B-coefficients used in equation (1). The uncertainty of the B-coefficients was typically better than 0.2 % ($k = 2$). The pilot laboratory used the three flowmeter comparisons to establish the reference value for the flow, F, at ambient temperature over the time period of the ILC. The reference values for flow were temperature corrected to 23 °C using the B-coefficients, as described above.

Participant Laboratory	Location
Boeing Company	Anaheim, CA
Helium Leak Testing	Northridge, CA
LACO Technologies	Salt Lake City, UT
LDS Vacuum Products, Inc.	Altamonte Springs, FL
NIST (Pilot Laboratory)	Gaithersburg, MD
Raytheon Company	El Segundo, CA
Sandia National Laboratories	Albuquerque, NM
Vacuum Instruments Corporation	Ronkonkoma, NY
Vacuum Technology, Inc.	Oak Ridge, TN
Varian, Inc.	Lexington, MA

Table 1. List of interlaboratory participants and their locations.

The artifacts were transported between the laboratories in a foam-padded aluminum box that was designed to minimize mechanical shock during transit. To facilitate mounting the artifacts on each participant's calibration system, an assortment of common mounting adapters were included in the shipping container along with extra gaskets, protective caps, clean aluminum foil, and extra mounting hardware and tools. Prior to calibration, the protocol required that the artifacts be pumped for a period of 24 hours to allow for equilibrium of both temperature and helium flow rate. As mentioned earlier, the protocol required that the flow rate for each artifact be measured and reported as close to 23 °C (296.15 K) as possible. No restriction was placed on the number of flow measurements that could be made on each artifact; the only requirement was that sufficient data be taken so that the quality was satisfactory to the participant. After completing all measurements, the participants were asked to remove the artifacts from their calibration system, cover the flow port of each artifact with a piece of "oil free" aluminum foil (provided in the shipping container) along with a plastic dust cover, and return each artifact to its designated place in the shipping container. The participant was then required to ship the container to the next participant via ground transportation. The calibration schedule followed a "modified star pattern" depicted in Fig. 1.

After completing measurements, the ILC protocol required the participants to submit a short report to the pilot laboratory containing the following information: a description of the measurement method used, the measured flow rate at 23 °C, the raw data, and a complete uncertainty evaluation including Type A and Type B uncertainties, or a combined expanded uncertainty ($k = 2$) which contains both Type A and Type B uncertainties. [5] Any deviations from the testing protocol or anomalous behavior were required to be reported to the pilot laboratory and disclosed in the report.

The final round of data taken by the pilot laboratory was acquired about three years after the last data were taken by the other participants. Although this delay was not intentional, it did have the added benefit of allowing an accurate determination of the depletion rate of all three leak artifacts.

The International System of Units (SI) will be used throughout this paper, and the unit of gas flow specified for reporting the measured helium flow in the ILC is the mole per second (mol/s). Although the unit of cm^3 per second at STP (standard temperature and pressure) is commonly used in the helium leak detection industry, this unit requires the specification of a "standard" temperature and pressure, usually 0 °C and 101.33 kPa (1 cm^3/s at 0 °C and 101.33 kPa equals 4.46×10^{-5} mol/s); unfortunately, there are instances of different standard temperatures used in the literature. Since the flow from a helium permeation leak artifact is strongly dependent on temperature, confusion over which temperature is "standard" will have a profound effect on the reported leak rate. The unit of moles per second is in accord with SI units [6] and is independent of temperature and pressure and therefore unambiguous. [7, 8] It is common nomenclature to use Q to represent throughput so that molar flow rate is $F = Q/RT$, where R is the molar gas constant and T is the absolute gas temperature. In this report, F will represent the molar flow rate in mol/s; units of throughput will not be used. Conversion factors can be found in Ehrlich and Basford [8] and in Moss [9].

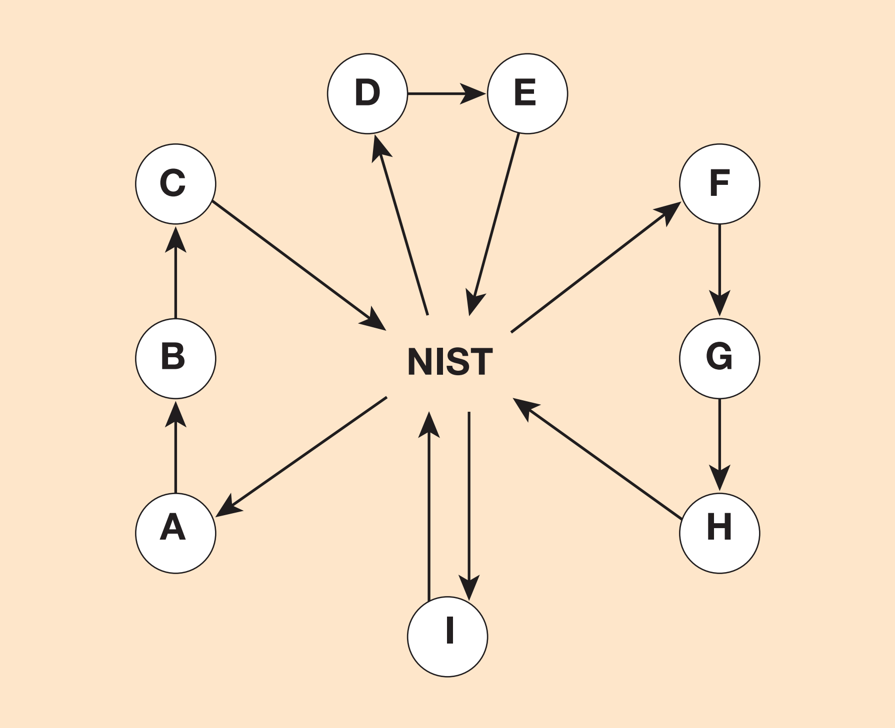

Figure 1. Artifact shipping pattern for the ILC. Each letter represents a different participating laboratory.

3. Leak Measurement Techniques

Several different techniques for measuring leak rates were employed by the participant laboratories. The pilot laboratory (NIST) used two systems for determining the flow from leaks: The NIST Primary Leak Standard (PLS) and the NIST Leak Comparison System (LCS) [3]. Note that the name PLS is used because the apparatus utilizes a primary technique for leak calibration, defined in Ehrlich and Basford [8] as one which depends on mass, pressure, volume, temperature, and time measurements, and also to be consistent with previous references to the apparatus in the literature. Neither the PLS, nor any of the other techniques employed in this study by the participants, are true primary standards because they utilize calibrated instruments. A brief summary of these techniques is given below, and additional details can be found in Ehrlich and Basford [8].

As stated above, NIST used two systems to determine the flow from the leak artifacts: The PLS and the LCS. The PLS employed a method of direct comparison of an unknown gas flow to that of a known gas flow generated by a constant-pressure flowmeter [2]. The flowmeter was used to generate a known flow of helium that was approximately the same flow as that of the leak artifact being calibrated. Flow from the leak artifact and the flowmeter were alternately introduced into a the upper portion of the PLS high vacuum chamber, where the partial pressure of helium was measured with a high quality quadrupole mass spectrometer tuned to mass 4 (helium). A small orifice separated the upper and lower portion of the PLS chamber such that the partial pressure was much greater in the upper portion than in the lower portion. By comparing the spectrometer signal due to the known flow from the flowmeter to that of the helium artifact with an unknown flow, the unknown flow from the artifact was determined.

In addition, a "flow-ratio" or "flow-division" method was used to determine the leak rate from leak artifacts with particularly low flow rates. In this technique, the known flow from the flowmeter was directed into the lower chamber and was alternatively compared to the unknown flow from the leak artifact, which was directed into the upper chamber. In this case, the generated flow from the flowmeter must be greater than that of the unknown flow from the leak artifact in order to produce a similar partial pres-

		NIST 11		NIST 10		NIST 12	
Lab Code	**Date**	**F_{Lab} (mol/s)**	**U_{Lab} (%)**	**F_{Lab} (mol/s)**	**U_{Lab} (%)**	**F_{Lab} (mol/s)**	**U_{Lab} (%)**
NIST 1	**Nov-03**	**9.508E-12**	**1.1**	**4.366E-12**	**1.8**	**8.677E-13**	**1.2**
A	Jan-04	9.401E-12	0.7	4.289E-12	0.7	8.570E-13	0.9
B	Feb-04	9.465E-12	2.8	4.381E-12	2.8	8.672E-13	1.5
C	Mar-04	9.188E-12	3.9	4.460E-12	3.9	9.143E-13	3.9
NIST 2	**Apr-04**	**9.460E-12**	**1.0**	**4.343E-12**	**1.0**	**8.668E-13**	**1.1**
D	May-04	9.9E-12	10.0	4.4E-12	10.0	9.1E-13	10.1
E	June-04	8.462E-12	7.3	3.830E-12	6.9	7.688E-13	10.2
NIST ×1	**Aug-04**	**9.427E-12**	**1.3**	**4.315E-12**	**1.5**	**8.550E-13**	**1.2**
F	Nov-04	9.096E-12	4.0	4.105E-12	3.9	8.563E-13	4.6
G	Mar-05	9.4E-12	12.0	4.3E-12	14.0	8.5E-13	14.0
H	Apr-05	6.57E-12	20.0	4.55E-12	20.0	8.68E-13	20.0
NIST ×2	**May-05**	**9.424E-12**	**1.4**	**4.300E-12**	**0.8**	**8.556E-13**	**1.3**
I	Jul-05	9.710E-12	4.0	4.257E-12	4.1	8.912E-13	3.9
NIST 3	**Aug-08**	**9.331E-12**	**0.7**	**4.268E-12**	**0.7**	**8.627E-13**	**0.9**

Table 2. Summary of ILC flow measurement data for all participants. F_{Lab} is the measured flow, and U_{Lab} is the expanded uncertainty (k = 2). Measurements reported at temperatures other than 23 °C where adjusted to 23 °C using equation (1). The reported uncertainties are rounded to one decimal place for this table.

sure of helium in the upper chamber. The ratio of flows between the lower and upper chamber that yield the same partial pressure in the upper chamber was known from a separate measurement and was used to adjust the final measured leak rate. NIST employed both of these techniques when using the PLS in this study, as will be discussed further in Section 4. These techniques are discussed in detail by Abbott and Tison. [3]

A variation of the above direct comparison technique involves using a previously calibrated leak artifact as the known gas flow. This artifact then serves as a transfer standard and is used in conjunction with a quadrupole mass spectrometer to calibrate a leak artifact whose flow is unknown. The NIST LCS employs this technique. During the ILC, the LCS utilized leak artifacts previously calibrated on the NIST PLS as transfer standards. In addition, a leak artifact mounted on NIST LCS could be temperature controlled over a range of 0 °C to 50 °C, thus allowing the determination of the B-coefficient in equation (1). Many of the participants in the study also employed this technique, although they did not necessarily have the ability to determine the B-coefficient. These laboratories utilized standard leaks that had been calibrated by NIST or another calibration laboratory.

Another calibration method that was used by the participants in the study was the "V-delta-P," or "pressure rate-of-rise," technique. This procedure involves directing the flow of a leak artifact into an initially evacuated vessel whose volume is accurately known. By measuring the pressure as a function of time as the gas flows into the volume, the flow is calculated by making use of the time derivative of the ideal gas equation at constant volume:

$$F = (RT)^{-1} \cdot V \cdot {}^{\Delta P}\!/_{\Delta t}\,, \tag{2}$$

where ΔP is the pressure rise in the vessel with volume V in a time interval Δt.

Finally, the "accumulate-dump" technique was also used by participants in the ILC. This technique utilizes a mass spectrometer to compare the partial pressures of helium from the unknown artifact with a series of gas samples generated by expansion from a known pressure of gas contained in a known volume. The gas from the sample is "dumped" to the mass spectrometer and the resulting partial pressure is measured. In the same way, gas from the unknown leak artifact is collected for a specified period of time and then dumped to the mass spectrometer, where the partial pressure is measured. The flow from the leak artifact is calculated from the partial pressure of helium, the volume, the temperature, and the collection time.

4. Results and Analysis

4.1 Participant Data and Reference Values

The flow measurement results for all participants are listed in Table 2. To ensure confidentiality of the reported data, each par-

	NIST 11			NIST 10			NIST 12		
	U_A (%) ($k = 1$)	U_B (%) ($k = 1$)	U_{Lab} (%) ($k = 2$)	U_A (%) ($k = 1$)	U_B (%) ($k = 1$)	U_{Lab} (%) ($k = 2$)	U_A (%) ($k = 1$)	U_B (%) ($k = 1$)	U_{Lab} (%) ($k = 2$)
NIST 1	0.24	0.50	1.10	0.16	0.89	1.81	0.28	0.51	1.16
NIST 2	0.10	0.46	0.94	0.15	0.47	0.98	0.23	0.51	1.11
NIST 3	0.09	0.31	0.65	0.11	0.31	0.66	0.30	0.31	0.86

Table 3. Type A, U_A, and Type B, U_B, uncertainties for the NIST measurements made with the PLS.

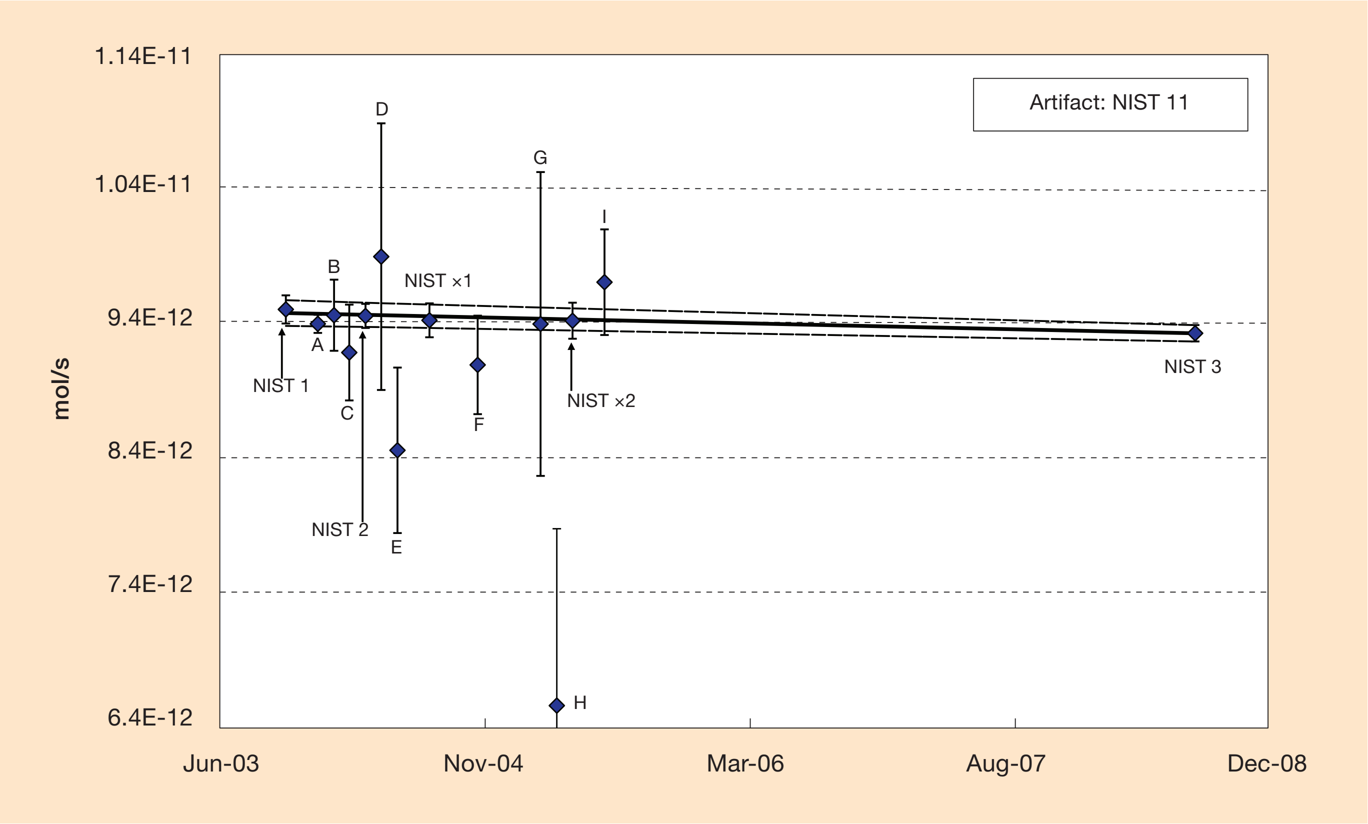

Figure 2. Summary of ILC flow measurements for the NIST 11 leak artifact. The error bars represent the expanded uncertainty ($k = 2$) for each participant. The heavy horizontal line is the reference value. The two dashed lines represent the expanded uncertainty of the reference value.

ticipant has been assigned a laboratory code (Lab A through Lab I). Flow rates that were reported at a temperature different than 23 °C were adjusted to 23 °C using equation (1). The laboratory code "NIST" represents the five calibrations performed by the pilot laboratory. NIST 1, NIST 2, and NIST 3 designate calibrations performed using the PLS, while the labels NIST ×1 and NIST ×2 designate calibration performed using the LCS, as discussed above. Table 2 lists the flow measurements, F_{Lab}, the expanded uncertainty, U_{Lab}, in the measured leak rate, and the month and year when the calibration was performed for all participants. The expanded uncertainty represents the combined Type A and Type B uncertainties to a coverage factor of $k = 2$, corresponding to a confidence level of approximately 95 % (assuming a normally distributed measurement). Table 3 lists the Type A and Type B contributions for the NIST PLS measurements.

From Table 2, it is clear that the total uncertainties for the NIST 1 measurements were larger than those for NIST 2 and NIST 3. Although the PLS was used to make leak measurements in all three rounds, the NIST 1 measurements employed the direct comparison technique for the NIST 10 and NIST 11 artifacts, whereas the split-flow technique was used for all the artifacts during the NIST 2 and NIST 3 measurements. For the NIST PLS, the Type B uncertainty typically increases as the helium leak rate becomes smaller. The split-flow method allows a larger known flow to be used, but adds an additional uncertainty component associated with the flow-ratio that must be used to calculate the flow rate. For the flow rates covered in this comparison, the split-flow method typically yielded a smaller combined uncertainty that did the direct comparison method. In addition, the turbo-molecular pump on the NIST PLS was replaced between the NIST 2 and NIST 3 measurements with a faster pump. This increased the flow-ratio and resulted in a lower uncertainty for the NIST 3 measurements.

As stated in Section 2, the comparison measurements (LCS) typically had a higher uncertainty than did the PLS but, as can be seen in Table 2, this was not the case for the comparison measurements

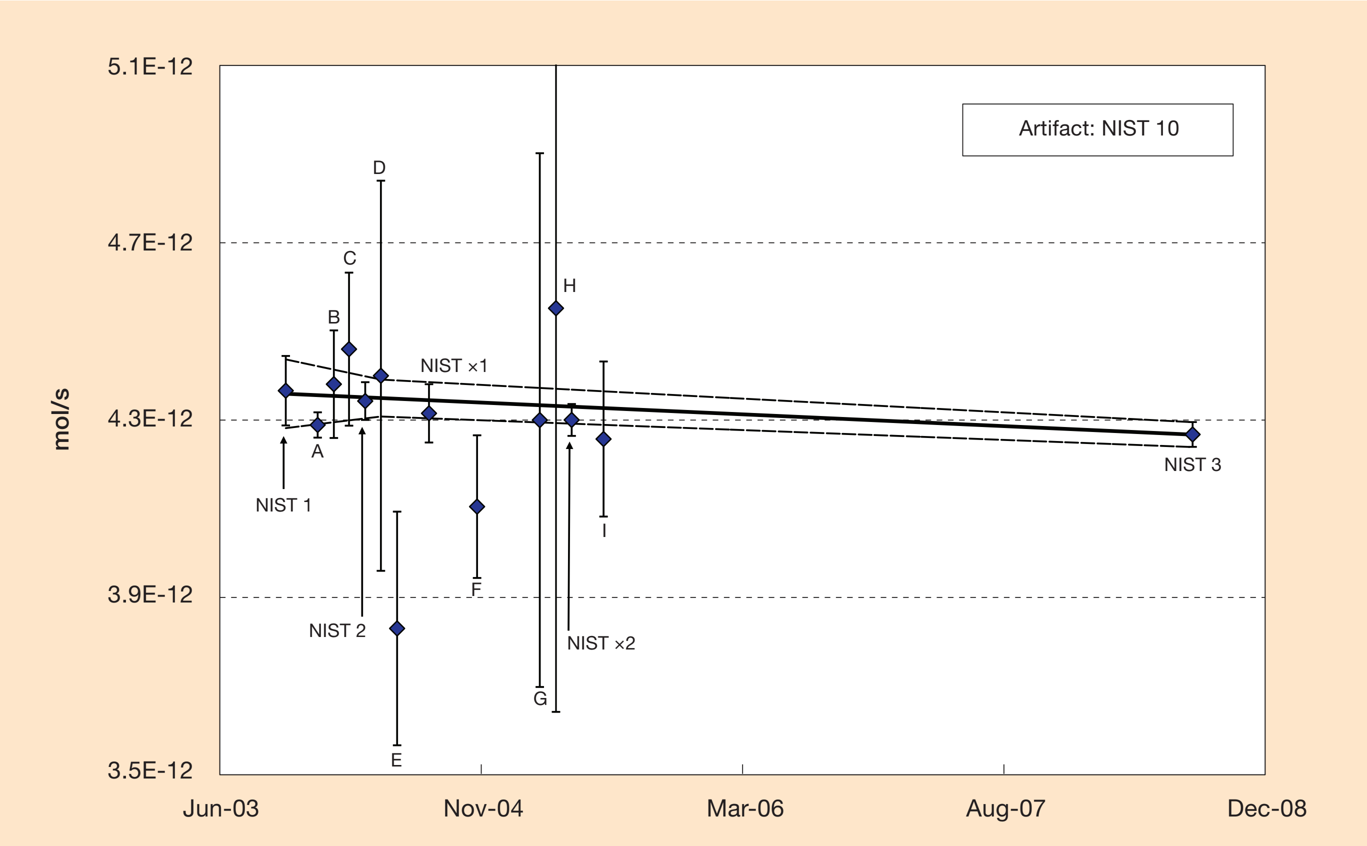

Figure 3. Summary of ILC flow measurements for the NIST 10 leak artifact. The error bars represent the expanded uncertainty ($k = 2$) for each participant. The heavy horizontal line is the reference value. The two dashed lines represent the expanded uncertainty of the reference value.

NIST ×1 and NIST ×2 for the artifact NIST 10. This resulted from an unusual set of circumstances, so an explanation is warranted here. The NIST LCS utilized calibrated helium leak artifacts as standards. The uncertainty in the flow rate from the calibrated leak standard was a significant component of the total LCS uncertainty. Like the leak artifacts in this study, the flow rates for these leak standards were determined utilizing the NIST PLS. Therefore, one would expect that the total combined uncertainty for the LCS measurements to be greater than that for the PLS. However, the leak artifact used as a standard in the LCS was measured with the PLS using the split-flow technique, which had a much lower uncertainty than the direct measurements of NIST 1. This is the primary reason that both the NIST ×1 and NIST ×2 measurements have smaller uncertainties than does the NIST 1 measurements. In addition, while the leak standard used in NIST ×2 – NIST 10 comparison had a similar flow rate to the NIST 10 artifact, it was not the same flow rate. As discussed in McCulloh et al. [2], pressure measurements contribute significantly to the uncertainty of the flows determined with the PLS. Different pressure gauges were used during the PLS calibration of the leak standard than for the NIST 10 artifact. These pressure gauges had different uncertainty contributions; consequently, in this particular case, the total combined uncertainty of the NIST ×2 measurements are slightly lower than the NIST 2 measurements for the NIST 10 artifact.

Figures 2 thru 4 are graphs of the data listed in Table 2. Each figure shows the measured flow rate reported by each participant with error bars representing the expanded uncertainty.

The flow from a helium leak artifact will become lower with time as the helium depletes from the reservoir. In general, the flow rate will reduce exponentially with time for a leak artifact kept at constant temperature. Given the low depletion rates of the artifacts (< 2 %/yr) and the relatively small time period covered by this study (as compared to the depletion rate), the depletion rate for the leak artifacts used in this study can be considered to be linear with time to a good approximation. Therefore, the reference lines and values were determined from a weighted linear least-square fit of the three data points NIST 1, NIST 2, and NIST 3 for each artifact. It is appropriate to choose the components of the uncertainty which are random and uncorrelated between the three data points as weights in the fit. Clearly the Type A uncertainties fall into this category. The Type B components of uncertainty may contain effects which are systematic or random; however, it is difficult or impossible to completely sort these out. A conservative approach is to assume that the Type B uncertainties cannot be reduced by making additional measurements and to omit these from the weights. Therefore, the inverse squared Type A uncertainties associated with each of these points were used as the weighting factors for the fit. The determined slopes, a, and intercepts, b, are listed in Table 4. The Type A uncertainties used to calculate the weights for the fit are listed in Table 3.

In order to facilitate a comparison of the measurements of the participating laboratories with the reference value, an uncertainty must be assigned to the reference value. The expanded uncertainty ($k = 2$) of the reference values are represented by dashed lines in Figs. 2 thru 4. The uncertainty of the reference

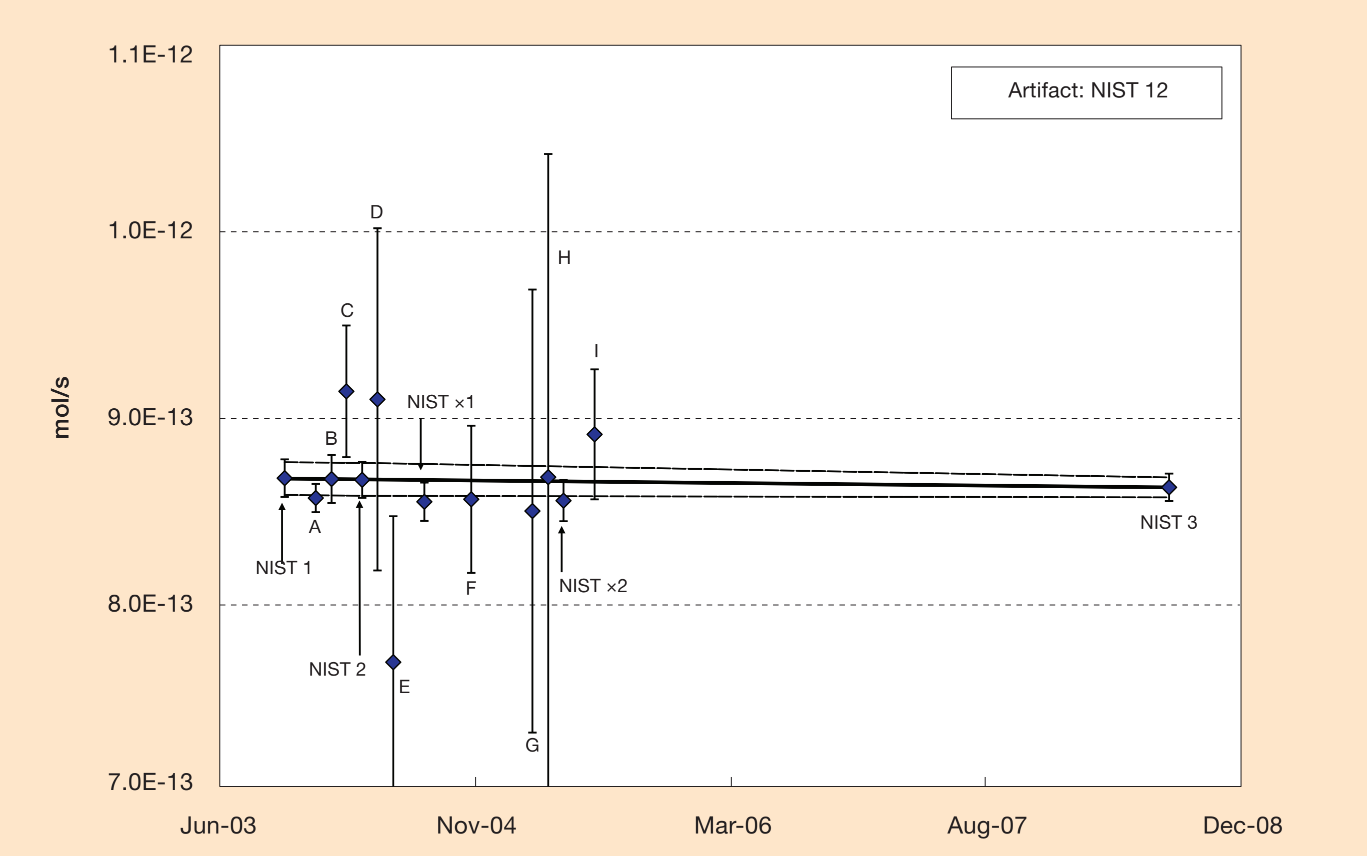

Figure 4. Summary of ILC flow measurements for the NIST 12 leak artifact. The error bars represent the expanded uncertainty ($k = 2$) for each participant. The heavy horizontal line is the reference value. The two dashed lines represent the expanded uncertainty of the reference value.

Artifact	*a* (Slope) [(mol/s)/day]	*b* (Intercept) [mol/s]	Depletion Rate [%/year]	Max U_{Ref} (%) ($k = 2$)	Min U_{Ref} (%) ($k = 2$)
NIST 11	-8.6749E-17	9.4812E-12	0.3	1.01	0.65
NIST 10	-5.3123E-17	4.3594E-12	0.4	1.78	0.66
NIST 12	-2.7259E-18	8.6743E-13	0.1	1.01	0.91

Table 4. Reference values for the leak artifacts. The reference value is determined from $F_{Ref} = at + b$, where t is the time in days since November 1, 2003.

value, U_{Ref}, is determined at a particular time by combining the Type A uncertainty, calculated from the fit using standard statistical methods, with the linear interpolation of the Type B uncertainty from the measurements of the pilot laboratory. All NIST 1 data were taken during November 2003 and the time, t, is taken to be the number of days since November 1, 2003; this day was arbitrarily chosen for $t = 0$ and all NIST 1 data were assumed to be taken on that day for simplification of the analysis. The fit also allowed the depletion rate of each artifact to be determined, which is also given in Table 4.

Since the reference value uncertainty is different for each laboratory, it is instructive to consider the maximum and minimum values of the reference value uncertainty over the course of the study. These are given in Table 4. The choice to use a linear interpolation to determine the Type B uncertainty at any given time was somewhat arbitrary; however, it is clear that the total uncertainty must be between the maximum and minimum values given in Table 4. With the exception of Lab A, the use of the maximum or minimum uncertainty in place of U_{Ref} has little impact on the comparison results. This will be discussed further in Section 5.

4.2 Proficiency Test

The comparison of the reference flow rates with measured flow rates can be evaluated by computation of the measurement error normalized with respect to the uncertainty of the measurements. This computation is accomplished by making use of the normalized error defined by:

$$E_n = \frac{F_{Lab} - F_{Ref}}{\sqrt{U_{Lab}^2 + U_{Ref}^2}} . \qquad (3)$$

The molar flow rate of the participant laboratory is represented by F_{Lab} with an associated expanded uncertainty U_{Lab}, as given in Table 2, converted to units of flow. The reference flow rate, F_{Ref}, was calculated from the linear fit parameters of Table 4. An acceptable measurement and reported uncertainty would

Lab Code	NIST 11			NIST 10			NIST 12		
	$\frac{F_{Lab}-F_{Ref}}{F_{Ref}}$ (%)	*U*(%)	E_n	$\frac{F_{Lab}-F_{Ref}}{F_{Ref}}$ (%)	*U*(%)	E_n	$\frac{F_{Lab}-F_{Ref}}{F_{Ref}}$ (%)	*U*(%)	E_n
NIST 1	0.28	1.49	0.19	0.16	2.53	0.06	0.03	1.54	0.02
A	-0.79	1.22	-0.65	-1.54	1.64	-0.94	-1.18	1.35	-0.88
B	-0.09	2.95	-0.03	0.61	3.09	0.20	0.00	1.80	0.00
C	-2.99	3.99	-0.77	2.46	4.06	0.59	5.44	4.00	**1.29**
NIST 2	-0.09	1.34	-0.07	-0.19	1.47	-0.13	-0.03	1.50	-0.02
D	4.59	10.04	0.44	1.16	10.05	0.11	4.97	10.15	0.47
E	-10.58	7.32	**-1.61**	-11.92	6.95	**-1.94**	-11.31	10.25	**-1.24**
NIST ×1	-0.32	1.62	-0.20	-0.68	1.79	-0.38	-1.35	1.56	-0.87
F	-3.74	4.12	-0.94	-5.42	4.03	**-1.42**	-1.17	4.72	-0.25
G	-0.41	12.03	-0.04	-0.78	14.03	-0.06	-1.86	14.03	-0.14
H	-30.41	20.02	**-2.18**	5.09	20.02	0.24	0.25	20.02	0.01
NIST ×2	-0.10	1.67	-0.06	-0.69	1.22	-0.57	-1.20	1.59	-0.76
I	2.99	4.13	0.70	-1.62	4.20	-0.39	2.94	4.02	0.71
NIST 3	0.00	0.92	0.01	0.01	0.93	0.01	0.00	1.06	0.00

Table 5. E_n numbers used as a proficiency test. The first column represents the numerator of equation (3), expressed as a percentage, and the second column represents the denominator of equation (3), expressed as a percentage.

result in an E_n value of between –1.0 and +1.0. This metric is derived from *ISO/IEC Guide 43* [10], and is associated with testing the proficiency of a laboratory. A flow measurement with an E_n value falling outside of ±1.0 fails the proficiency test. The E_n number was computed for each participant utilizing the measured flow rate and reported uncertainty, along with the associated reference value and uncertainty from Table 4. A summary of E_n numbers for each laboratory is presented in Table 5. Values in bold represent E_n values which fail the proficiency test. The total uncertainty, *U,* is represented by:

$$U=\sqrt{U_{Lab}^2+U_{Ref}^2}\ . \qquad (4)$$

For completeness, E_n values for NIST are also included in Table 4 even though these have limited meaning and are not an indication of the proficiency of the NIST laboratory since the reference values were calculated from the NIST measurements alone.

A graphical representation of the E_n data is shown in Fig. 5. This shows the percent difference between the laboratory and reference measurements (the numerator of equation (3) divided by the reference value) for each participating laboratory with error bars representing the total expanded uncertainty, *U,* from Table 5 (the denominator of equation (3)). These values are also summarized in Table 5. Looking at the data this way gives some information which can be obscured in the E_n values: laboratories with large uncertainties may have small E_n values, while it is possible that other laboratories have measurements that are close to the reference value, but with understated uncertainties, will have large E_n values. Note that since the reference values were taken from a fit, the difference between the pilot laboratory and the reference value is small but non-zero.

5. Discussion

Table 2 along with Figs. 2, 3, and 4 represent all data reported for this ILC. The dark line in each figure represents the reference flow value from Table 4. Each figure shows the measurement results of each participant along with their associated measurement uncertainty as a function of the date when the data were taken. Note that artifact NIST 11 had the highest leak rate of the three artifacts in the study, followed by the NIST 10 artifact, and, finally, the NIST 12 artifact had the lowest leak rate of the three. The E_n values of Table 5 were used to evaluate the proficiency of each laboratory.

Figure 3 represents data for artifact NIST 10. Two participating laboratories, Lab E and Lab F, had measurements which were smaller than the reference value by at least their expanded uncertainties.

Two laboratories failed the proficiency test for the NIST 11 artifact. The measurements of Lab E and Lab H were more than one expanded uncertainty below the reference value.

Finally, for the NIST 12 artifact, the measurement of Lab C was larger than the reference value, whereas Lab E was slightly below. Although the NIST 12 artifact represented the lowest leak rate in the study, it did not seem to present any more difficulty than the other two artifacts.

Overall, Lab A, Lab B, Lab D, Lab G, and Lab I consistently measured leak rates within their stated uncertainties and passed the proficiency test for all the leak artifacts measured. Table 5

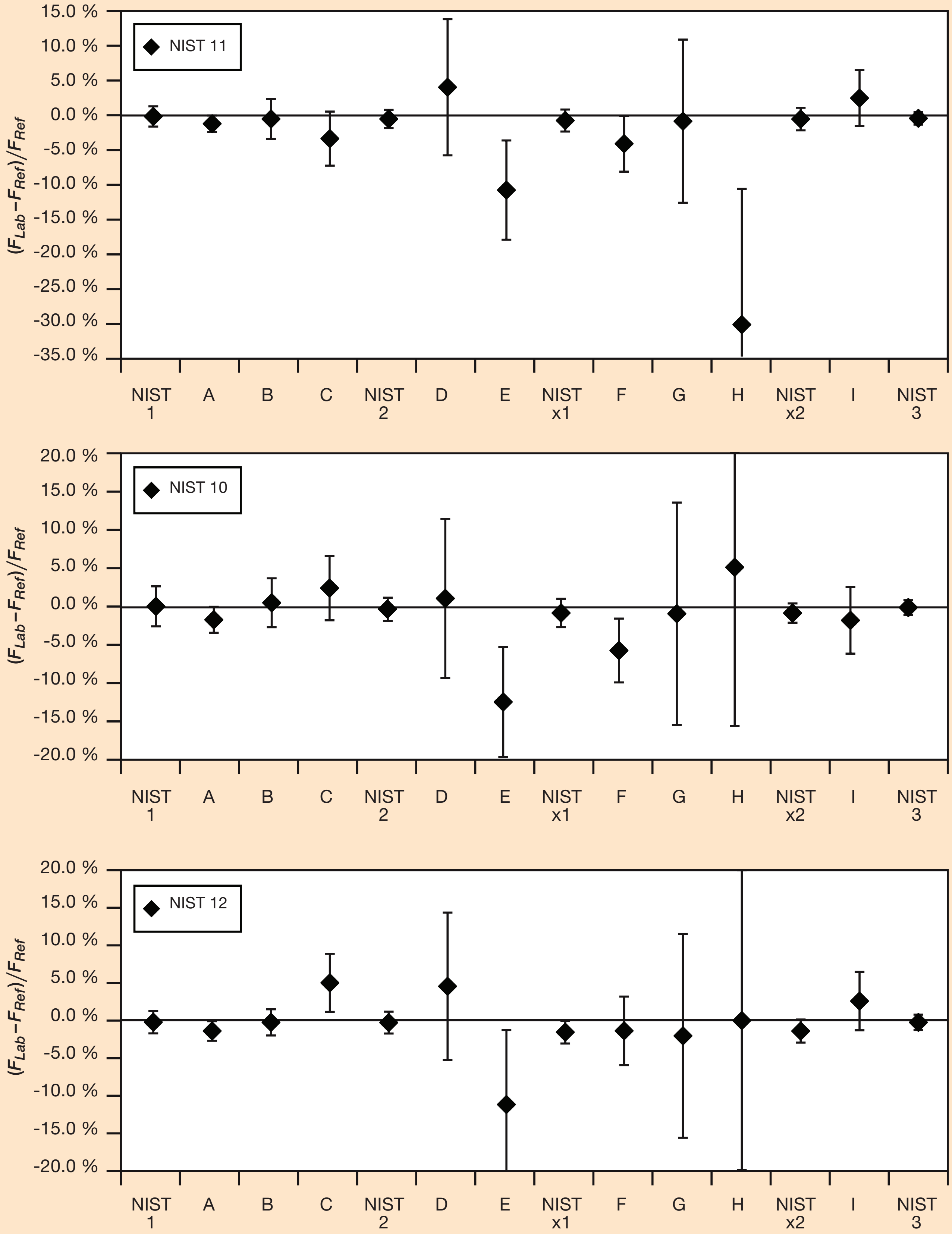

Figure 5. The percent difference between the participating laboratory measurements and the reference value. The error bars represent the combined total uncertainty ($k = 2$) of the laboratory measurement and the refrence value.

and the difference graph of Fig. 5 are useful in evaluating these laboratories. Lab A, Lab B, and Lab G were consistently within 2 % of the reference value for all the artifacts measured. In light of this, it would seem that the reported uncertainties for Lab G, which were between 12 % and 14 %, may be very conservative. The uncertainties of Lab D may also have been slightly overstated as well: the measurements of Lab D were within 5 % of the reference value for all the leak artifacts measured, but their stated uncertainty was about 10 % in all cases.

Three laboratories passed the proficiency test for two of the three artifacts. Lab C passed the proficiency test for artifacts NIST 10 and NIST 11, but failed on artifact NIST 12 with an E_n of about 1.3. Lab F passed the proficiency test for NIST 11 and NIST 12, but failed on the NIST 10 artifact with an E_n of about –1.4. Finally, Lab H passed the proficiency test for artifacts NIST 10 and NIST 12, but failed on artifact NIST 11 with an E_n of about –2.2.

Lab E failed the proficiency test in that their E_n values were less than –1.0 for all the artifacts measured. All of the measurements of Lab E were consistently low, suggesting that there were systematic problems, or that the uncertainties may have been understated. Lab E used a pressure rate-of-rise technique, discussed in Section 3, to measure the leak rate. In comparison, the other laboratories that had failures, Labs C, E, and F, all used the comparison technique to measure the leak rate. Since these measurements were made, Lab E has reported improvements in their calibration techniques and equipment, including a more accurate pressure gauge and a lower uncertainty assigned to their calibrated volume.

As mentioned in Section 4.1, choosing to use the minimum or maximum U_{ref}, from Table 4 to calculate the E_n values would have had little impact on the results of the proficiency test for any of the participating laboratories with the exception of Lab A. Choosing the minimum U_{ref} would have caused Lab A to fail the proficiency test for NIST 10. Lab A had the smallest uncertainty statement of any of the participating laboratories, and the data were taken close in time to the NIST 1 measurements. In fact, the reported uncertainties of the Lab A measurements were smaller than those of the NIST 1 and NIST 2 measurements. Given the small uncertainties of Lab A, it would seem unreasonable to compare the Lab A measurements to the minimum U_{ref} from Table 4, which corresponds to the uncertainty in the NIST 3 measurements. Therefore, the preceding discussion of the proficiency test, based on the U_{ref} shown in Figs. 2 thru 4, should be an accurate representation of the capabilities of all of the participating laboratories at the time the data were taken.

6. Summary and Conclusions

NIST performed an interlaboratory comparison of helium flow rates from three leak artifacts with ten participants. The leak artifacts had nominal helium flows of 8.6×10^{-13} mol/s, 9.4×10^{-12} mol/s, and 4.3×10^{-12} mol/s. NIST performed flow measurements of all of the artifacts three times during the course of the study. From these measurements, a reference value was determined and used to facilitate a comparison to the participating laboratories. The normalized error, E_n, was used as a proficiency test to evaluate the measurement capabilities of the participants. An E_n value between –1.0 and +1.0 indicates that a laboratory performed a measurement that was within their stated expanded uncertainty. Five of the laboratories, Lab A, Lab B, Lab D, Lab G, and Lab I, consistently showed good proficiency with E_n values between –1.0 and +1.0. Lab A, Lab B, and Lab G produced measurements that were within 2 % of the reference value for all the artifacts measured. Lab A had stated expanded uncertainties of less than 1 % and passed the proficiency test for all three artifacts. Lab D and Lab I were within 5 % of the reference values for all the artifacts measured. Lab C, Lab F, and Lab H passed the proficiency test for two of the three artifacts. One of the laboratories, Lab E, consistently produced measurements smaller than the reference value by more than their stated expanded uncertainty, thus producing E_n values less than –1.0.

It should be noted that this study reflects the practices of the participants during the time of the study: January 2004 to July 2005. Improvements or other changes of equipment or operating procedures could alter the measurement capability of any of the participating laboratories. As of this writing, Lab E has informed the authors that they have made significant changes in their apparatus and procedures which could affect the accuracy and precision of their measurements. Considering the length of time required for a round-robin style of comparison, such as presented here, one may suspect that a bi-lateral proficiency test between an individual laboratory and NIST may be a more efficient way to evaluate the measurement capability of a laboratory. At present, such a program does not formally exist at NIST, but would be considered upon request.

7. References

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