

## WHAT IS THE "BEST" TRANSFER STANDARD FOR GAS FLOW?

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National metrology institutes around the world have embarked on an effort to compare their primary gas flow standards under the supervision of the Comité International des Poids et Mesures and its Working Group on Fluid Flow. As the pilot laboratory for the key comparison of low-pressure gas flows below 1500 L/min (CCM.FF-K6), NIST is designing the transfer standard package with the assistance and advice of the other participants.

A number of topics are studied as part of the transfer standard design process. An analysis of the flow ranges and types of primary standards used by the possible key comparison participants based on their Calibration and Measurement Capabilities is given. The goals of the key comparison and the desirable characteristics of the transfer standard are considered in a general way. Next, a survey of the gas flow comparison literature suggests that laminar flow elements and critical flow venturis are the most practical candidates as transfer standards. These two flowmeter types are compared analytically and experimentally regarding their sensitivity to the external influences of temperature and gas composition. Finally, the calibration stability for these two flowmeter types is considered.

### Introduction

NIST is serving as the pilot laboratory for an international key comparison (KC) in low - pressure gas flow and we are in the process of designing the transfer standard to be used. This paper covers a wide range of topics related to the design of the transfer standard for the KC.

First, we analyze the facilities of the national laboratories that may participate in the KC (or in the regional comparisons that will follow using the same transfer standard design). We also give broad consideration to the goals of the KC and the desirable characteristics of the transfer standard. Due to our experience with critical flow venturis (CFV's) and laminar flow elements (LFE's), we admit a prejudice towards these meter types. They constitute the vast majority of the flowmeters that NIST calibrates for domestic flow traceability purposes and for international comparisons. We give a survey of the literature covering gas flow comparisons, and it confirms our perception that CFV's and LFE's are the most widely used flowmeter types for transfer standards.

We believe that it is practical for the transfer standard to have calibration stability of 0.05 % or better over the duration of the KC, and that this level of performance is necessary given the excellent uncertainties of many of the primary standards that exist today. In order to achieve 0.05 % performance from the transfer standard, we must consider certain flowmeter sensitivities that could safely be considered negligible for previous comparisons.

Therefore, a large section of this paper considers (analytically and experimentally) the sensitivity of LFE's and CFV's to temperature. Experimentally, four LFE and CFV models were subjected to two types of experiments using an oven to test the flowmeter at temperatures up to 314 K. Later we present data regarding the sensitivity of these meters to gas composition, and we answer the question, "Given that the KC is likely to be with dry air, what is the maximum dew point temperature that will support our 0.05 % goal?"

Finally, we discuss the calibration stability of LFE's and CFV's. We pay particular attention to changes in physical dimension (as from a thin layer of dirt on the flow boundary), and we present some calibration stability data from a set of CFV's and LFE's that we have been calibrating periodically over 6 months or more.

Our analysis of the candidate flowmeter types shows that the "best" transfer standard design depends upon the flow range, the types of primary standards compared, and especially on the details of the flowmeter design. The question will not be answered by a simple, analytical comparison of their equations of flow. Therefore, we explore some of the important issues of gas flow transfer standards, present the state of our knowledge about certain flowmeter sensitivities, and offer an invitation for further research and improvements in gas flow transfer standard design.

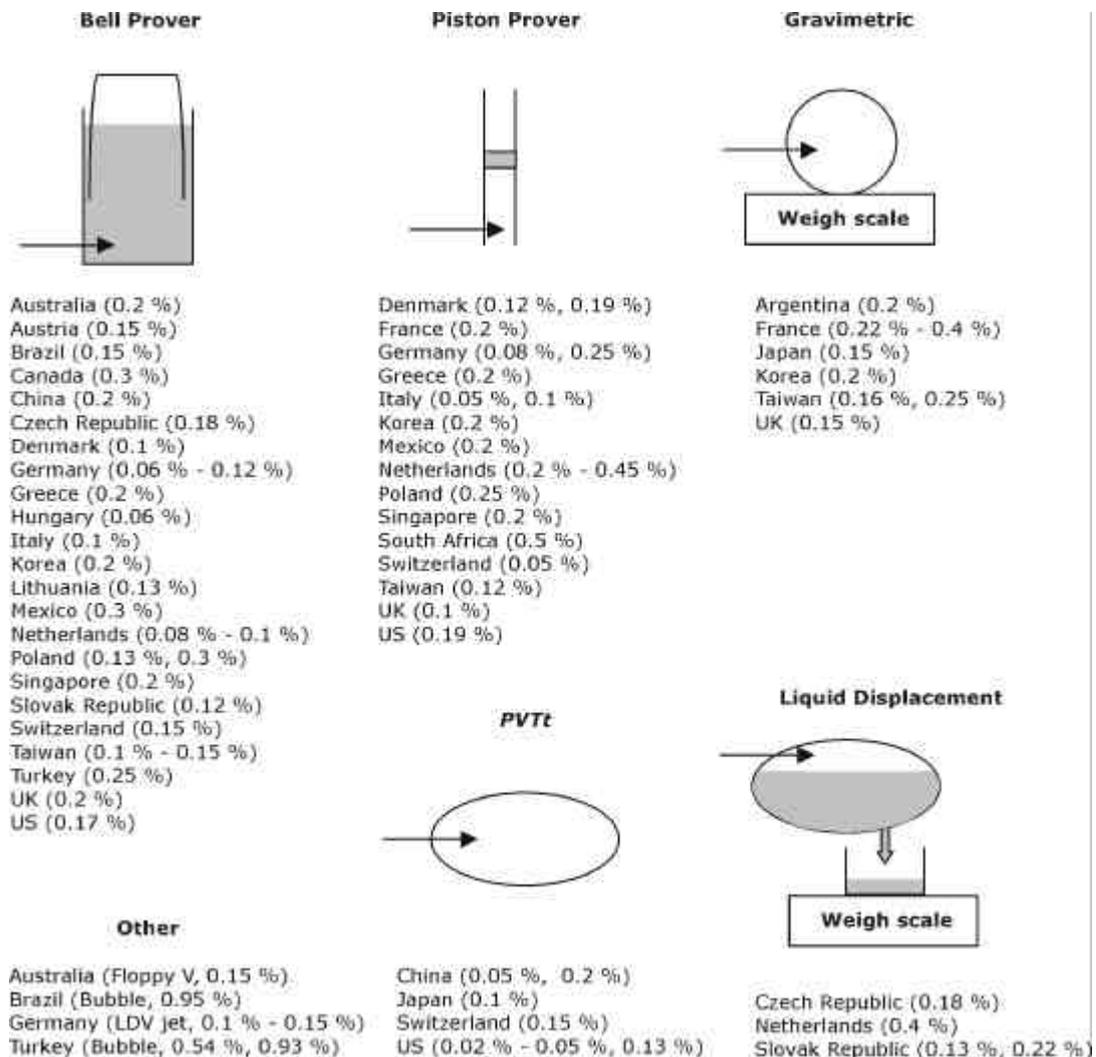


Figure 1. Types of primary flow standards utilized by the potential participating laboratories and their expanded uncertainty ( $k = 2$ ).

Review of Facilities of Potential KC Participants

The Regional Metrology Organizations have gathered the Calibration and Measurement Capabilities (CMC's) of the potential participants in the low -pressure gas flow key comparison. Our review of the CMC's with regard to the types of primary standards, their uncertainties, and their flow ranges is summarized in Figs. 1 and 2. There are six categories of primary gas flow standards: piston prover, bell prover, gravimetric, *PVTt*, liquid displacement, and other (Wright, 2001). The category "other" includes the "floppy" volumetric standard of CSIRO in Australia (Bignell and Choi, 2001), the LDV profiled jet of PTB in Germany (Dopheide et al., 1994), and bubble meters. Air or inert gas will be used in the KC, hence a large gravimetric facility in Canada and a large *PVTt* standard in France (both for natural gas only) are not included.

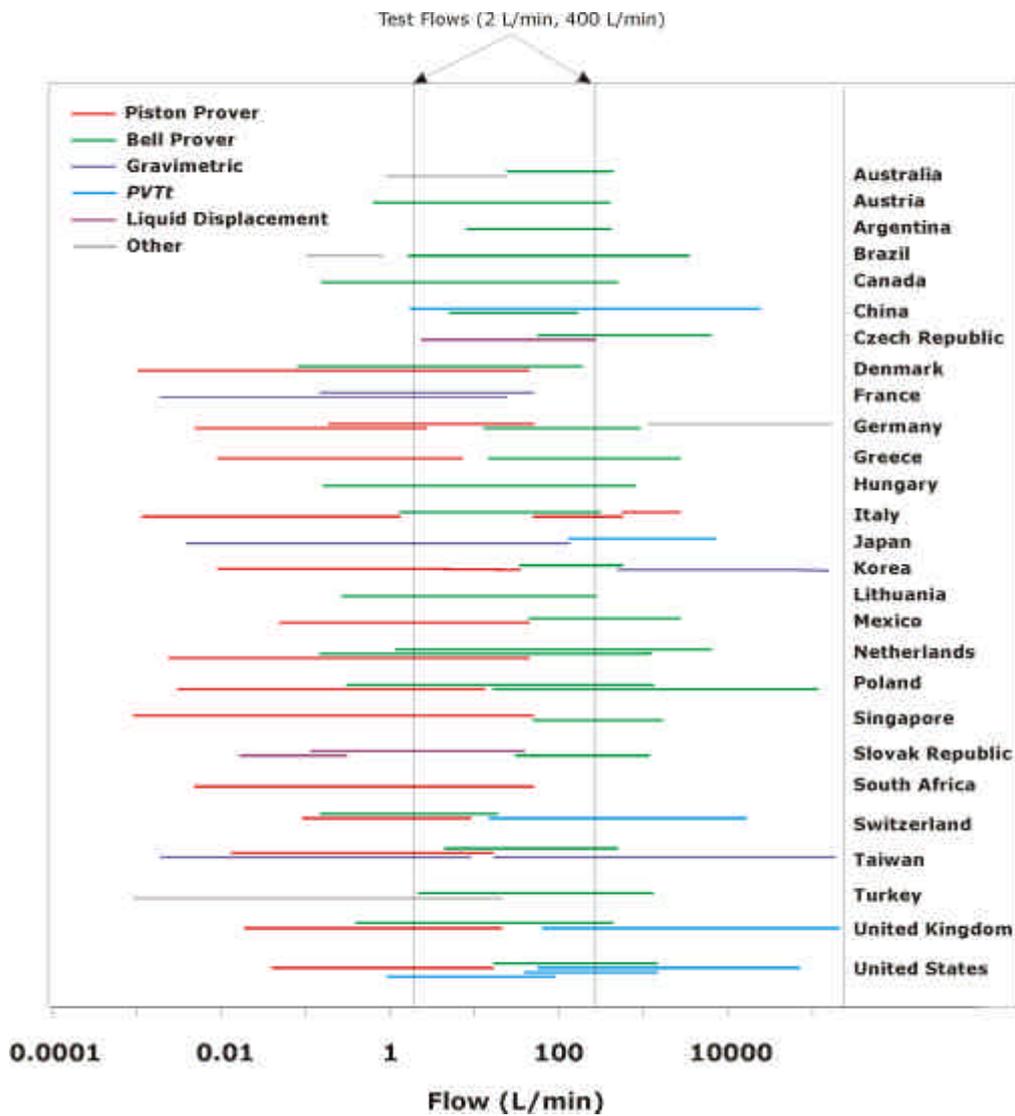


Figure 2. Flow ranges of potential participants based on the CMC's. The proposed test flows of the comparison (2 L/min and 400 L/min) are shown as well.

In our survey, the most commonly used primary standard by the potential participants is the bell prover (found in 23 out of 27 laboratories). The bell prover uncertainty statements range from 0.06 % to 0.3 %, with a mean value of 0.16 %. (All uncertainties stated herein are approximately 95% level of confidence or  $k=2$  values unless otherwise stated.) The nominal flow range covered by the bell provers is from 1 L/min to 50000 L/min, but some handle flows beyond this range. The second most popular primary standard is the piston prover (19 out of 27). The piston prover uncertainty statements range from 0.05 % to 0.5 %, with a mean of 0.19 %, and they cover a nominal flow range from 0.01 L/min to 50 L/min (although some are far above and below this range). Six countries operate gravimetric gas flow standards (0.15 % to 0.4 %), four operate  $PVTt$  standards (0.02 % to 0.2 %), and three operate liquid displacement standards (0.13 % to 0.4 %).

There are seven examples of uncertainty statements of 0.08 % or less. Therefore, it is desirable for the transfer standard to have calibration stability of 0.05 % or better. Even such a transfer standard could not discern differences between the better primary standards.

Many of the working standards listed in the CMC's were been included in the preceding review of laboratory standards because they were not primary standards. For example, a critical nozzle that has been calibrated via a primary standard and subsequently used to calibrate other flowmeters has not been included. Our goal is to perform comparisons using the primary standards, if possible. This is because the primary standard that is the basis of the working standard calibration will necessarily have a lower uncertainty, and we wish to perform the comparison with the laboratory's best flow standard. However, sometimes the design of a primary standard precludes its usage with certain types of transfer standards. In these cases the comparison may have to be done with working standards.

#### Purpose of the Key Comparison

The primary purpose of the low pressure gas flow key comparison is to **compare calibration results** of a gas flow transfer standard circulated between participating laboratories in order to **measure their degree of equivalence**. Secondary purposes of the KC are to:

1. Demonstrate laboratory proficiency, validate laboratory uncertainty statements.
2. Find and (where feasible) correct problems that lead to calibration errors or uncertainty. Compare methods of data collection, reduction, and calculation. Diagnose unknown or uncontrolled influences of the primary standard and its environment on flowmeter calibration results.
3. Give a snapshot of the present state of the art and advance the state of the art in flowmeters (transfer standards) and primary standards.

#### Characteristics of the Transfer Standard

The purposes of the comparison lead to the following list of desirable characteristics from the transfer standard, listed in nominal order of importance:

- **Calibration stability** (0.05 % or better)
- **Well-developed physical model** and well understood external influences. If the flowmeter is susceptible to significant errors due to temperature effects, installation effects, sensitivity to gas composition, etc., we must be well aware of it and either avoid the problem or take steps to quantify and correct the errors.
- **Robustness** through shipping and usage (e.g. shock, over-ranging). A modular design with readily available spare parts is desirable.

- **Compatibility with the primary standards** and measurement conditions in the participating laboratories.
- **Self-diagnostic capabilities, redundancy.** The transfer standard should have two flowmeters in series so that a problem in one can be detected with the other. Redundant instrumentation allows confidence and diagnoses of problems with pressure, temperature, or other measurements necessary for the transfer standard.
- **Ease of use** and familiarity of the participating laboratories with the flowmeter type.
- **Known time to stability** a) upon arrival in the laboratory, and b) upon initiation or change in flow.
- **Facility diagnostic capabilities.** If feasible, the transfer standard should allow examination of potential sources of uncertainty in flowmeter calibration introduced by the primary standard itself or the test environment.

#### Candidate Flowmeter Types / Review of Previous Gas Flow Comparisons

A survey of gas flow comparison publications covering the last 25 years shows that critical nozzles or critical flow venturis (CFV's) have been used most often, followed by laminar flow elements (LFE's), and then turbine flowmeters. Positive displacement and rotary gas have also been used, but to a lesser extent. A detailed review of the literature survey follows.

Laminar flow elements are generally considered the most appropriate meter type for relatively small gas flows ( $10^{-4}$  L/min to 1 L/min), but are commercially available to much higher flows, and have been used successfully in comparisons up to about 30 L/min. Jean Barbe of BNM-LNE in France used DH Instruments' Molbloc<sup>®</sup> LFE's at flows between 0.025 L/min and 10 L/min (Knopf et al., 2001, Nakao et al., 1998). Lin *et al.* (2000) also used Molblocs for comparisons between CMS (Taiwan), NIST (US), and KRISS (Korea) at flows between 0.1 L/min and 1 L/min. The NIST Pressure and Vacuum Group uses a custom made, thermostatted, high-differential LFE as a transfer standard for flows below 1 L/min. The NIST Fluid Flow Group has conducted a domestic comparison (sponsored by the US Department of Defense) between five laboratories at flows from 0.04 L/min to 30 L/min using Molblocs. Data concerning the calibration stability of these LFE's will be presented later in this paper.

Since the early 1990's, numerous groups have used CFV's to perform inter-laboratory comparisons. First we will summarize those CFV based comparisons conducted at flows of 400 L/min or greater.

Takamoto et al. (1993) used six ISO 9300 (ISO, 1990) toroidal throat CFV's to perform comparisons at flows between 417 L/min and 3340 L/min, with participation by NMIJ (Japan), PTB (Germany), and CEESI (a secondary calibration laboratory in the US). The comparison showed agreement between the laboratories was generally within  $\pm 0.05$  %.

In 1995, Gaz de France piloted EUROMET Project #307 which used a single 12.29 mm cylindrical throat nozzle at flows between 1300 L/min and  $10^5$  L/min in at least ten different laboratories (Vulovic, 1997, Vulovic et al., 1999). Laboratories did not necessarily test at the same flows or with the same gas and it seems likely that a flow transition occurred in the

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\* Certain commercial equipment, instruments, or materials are identified in this paper to foster understanding. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

nozzle throat within the tested flow range. Nonetheless the comparison demonstrated agreement between the laboratories of approximately  $\pm 0.3$  %.

In 1994, a division of the Ford Motor Company (now called Visteon Corporation) began a measurement assurance program using a set of three CFV's that permitted comparisons at 5900 L/min, 10900 L/min, and 14900 L/min (Caron et al., 1996, Paik et al., 1998). Two CFV's were used in series and one of the pair was switched so that the comparison data could be analyzed by the techniques developed by Youden. The transfer standard was tested at CEESI, KRISS, NMIJ, CMS, NEL, and twice at NIST more than four years apart (1994 and 1999). The agreement between the participating laboratories was generally better than  $\pm 0.15$  %. The two NIST calibrations agreed within 0.08 % and were generally closer than 0.05 %, demonstrating the very good long-term calibration stability for the CFV's.

Also in the late 1990's, CFV's were used for comparisons between six primarily North American and European natural gas flow laboratories (Karnik et al., 1996).

In the realm of small gas flows, in 1993 George Mattingly used a set of three glass critical nozzles to perform comparisons in twenty-two domestic laboratories (primarily between piston provers) at flows of 0.34 L/min and 0.86 L/min (Mattingly, 1993). The results of several before and after calibrations at NIST indicated transfer standard reproducibility of better than 0.15 %. The transfer standard allowed two nozzles to be used in series and valves permitted nozzle switching for Youden analysis.

In 1996, Shin-Ichi Nakao of NMIJ and Masao Hayakawa of Hirai Co. developed a CFV based transfer standard and began using it to perform comparisons around the world at flows between 0.04 L/min and 50 L/min (Nakao et al., 1996, Hayakawa et al., 1998). This transfer standard uses redundant pressure and temperature sensors and one of a set of CFV's is manually installed in a holder to achieve the desired flow. Comparisons conducted with this transfer standard between five national laboratories between 1996 and 1998 showed agreement generally better than  $\pm 0.2$  % (Nakao et al., 1999). The CFV's used in the transfer standard have shown calibration stability of  $< 0.02$  % (Wright et al., 1998).

Rotary gas meters, positive displacement meters, or Roots type meters were used as transfer standards in EUROMET Projects No. 419 and No. 425. Project No. 425 demonstrated agreement between twelve European laboratory bell provers within  $\pm 0.25$  % (Probert, 2000). Before and after calibrations (two years apart) showed calibration stability of 0.12 % or better for the transfer standard. Dijstelbergen and van der Beek (1998) document calibration stability of 0.03 % over a period of 18 months for a rotary gas meter. Unfortunately, rotary gas meters are subject to changes in leakage past the rotors due to changes in bearing friction, and they can be subject to installation dependent pressure pulsation and resonance problems unless damping systems are used. Also, it is not known how these meters would perform if two were installed in series, a desirable configuration for a transfer standard.

Wet test meters are used as reference standards in the Netherlands and Germany but apparently have not been used as transfer standards for inter-laboratory comparisons, probably due to their lack of robustness during shipping.

For large gas flows ( $> 2000$  L/min), and especially in natural gas, turbine meters are often used as transfer standards (Diritti et al., 1997, van der Beek et al., 1999), primarily due to

their low pressure drop. Occasionally problems in their long-term calibration stability have been observed, probably due to changes in friction in their mechanical bearings (Shaw et al., 2001).

The literature review above confirms that LFE's are generally preferred for small flows, while CFV's are generally used for higher flows. There is a flow range of 0.04 L/min to 30 L/min where both meter types have been used successfully, but a typical break point between the LFE and the CFV transfer standard is 1 L/min. The reasons for this can be understood by considering the weaknesses of the two flowmeter types.

As the throat diameter is made smaller, the assumption of sonic velocities across the entire CFV throat becomes less valid. The viscous boundary layer becomes a more significant portion of the throat cross section and the effects of external temperature conditions via the thermal boundary layer become more significant (Johnson et al., 1998, Bignell and Choi, 2002). Species effects caused by thermodynamic non-equilibrium phenomena (for certain gases) become larger with decreasing throat diameter (Johnson et al., 2000). Critical flow conditions become more difficult to achieve (Nakao et al., 2000, Caron et al., 2000) and less definite, suggesting that the downstream pressure influences the CFV discharge coefficient. As the throat diameter is made smaller, it becomes more difficult to manufacture the CFV in the desired ISO 9300 shape, and any burrs, surface flaws, dirt, or corrosion lead to unexpected performance or calibration changes. The elegance of the CFV lies in the large momentum change from the upstream plenum to the throat that overwhelms installation effects as well as the relative insignificance of viscous phenomena. Hence, at low throat Reynolds numbers, when the viscous forces gain significance, the advantages of the CFV diminish.

On the other hand, the LFE is a flowmeter that performs best at low flows, where viscous forces dominate over kinetic energy effects. The pressure drop across an LFE is primarily due to a combination of viscous (Hagen-Poiseuille) sources and kinetic energy sources (entrance and exit losses, development of the parabolic laminar profile, expansion of gas along the flow path, see Todd, 2001). A well-designed LFE minimizes the effects of kinetic energy relative to its viscous impedance. At high flows this becomes difficult to do in a compact size.

At this point, our search for the "best" transfer standard for gas flow below 1500 L/min has been narrowed to laminar flow elements and critical flow venturis. Many potential external influences on these transfer standards, such as vibration, line voltage stability, installation or velocity profile effects, and pressure stability are either insignificant or are controlled in standard practice. For instance, the length of upstream straight piping or flow conditioners should be a part of the transfer standard in order to minimize installation effects. We can be confident of sufficient pressure stability by including a qualified pressure regulator in the transfer standard package.

We will consider the effects of two external influences that are not so readily controlled by the design of the transfer standard: temperature and gas composition. Regarding temperature sensitivity, our primary concern is that the transfer standard might be used in laboratory "A" under ambient conditions of 23 °C, then calibrated in laboratory "B" at 24 °C. Any differences in the calibration coefficients caused by uncontrolled temperature effects on the transfer standard will be incorrectly attributed to the laboratories' primary standards. This leads to the question: which meter type less sensitive to environmental temperature conditions? We also expect differences in the composition of "dry air" from laboratory to laboratory, particularly for

water content, leading to a similar question regarding the sensitivity of the two meter types to differences in gas composition. These two external influences will be considered via analytical and experimental approaches.

### Temperature Sensitivity

#### Critical Flow Venturis

The effect of temperature on flowmeter performance is a common concern among gas flow metrologists. First order temperature effects are corrected via the flowmeter physical model. For the CFV, the equation for mass flow is:

$$\dot{m}_{CFV} = \frac{C_d C^* A P_0}{\sqrt{R_u T_0 / M}} \quad (1)$$

where  $C_d$  is the coefficient of discharge,  $C^*$  is the critical flow factor (a function of gas properties),  $A$  is the throat area of the CFV,  $M$  is the gas molecular weight,  $P_0$  is the gas stagnation pressure,  $R_u$  is the universal gas constant, and  $T_0$  is the gas stagnation temperature. The discharge coefficient is largely dependent on the throat Reynolds number,  $Re = 4\dot{m}/p d m$  where  $d$  is the throat diameter and  $m$  is the gas viscosity. Second order temperature effects (Bignell and Choi, 2002) enter through (a) the critical flow factor,  $\partial C^*/\partial T$  and (b) the effects of thermal expansion on the throat area,  $\partial A/\partial T$ . There are also changes in the discharge coefficient due to temperature that for our purposes will be divided into two categories: (c) the change in  $C_d$  that follows from the changes in Reynolds number (due to the temperature dependence of viscosity),  $\partial C_d/\partial Re$ , and (d) the changes in  $C_d$  due to changes in the thermal boundary layer thickness and that are not captured by the Reynolds number dependence,  $\partial C_d/\partial T|_{Re}$ . The third category of temperature dependence, (c), can be estimated from a  $C_d$  versus  $Re$  calibration curve measured at nominally constant temperature conditions. The fourth dependence, (d), is not accounted for by the physical model of Eqn. 1 and is a current topic of research.

#### Laminar Flow Elements

For the LFE, the equation for the differential pressure across a single, circular laminar flow tube is approximately:

$$\Delta P = \left[ \left( \frac{8 R_u}{p P M} \right) \left( \frac{C_m m \ell T}{r^4} \right) \right] \dot{m}_{LFE} + \left[ \left( \frac{R_u}{p^2 P M} \right) \left( \frac{C_{KE} T}{r^4} \right) \right] \dot{m}_{LFE}^2 \quad (2)$$

where  $m$  is the gas viscosity,  $\ell$  is the tube length,  $r$  is the tube radius, and  $C_m$  and  $C_{KE}$  are the viscous and kinetic energy calibration coefficients, related to the meter geometry, and normally are determined by flow calibration against a flow standard. The mass flow can be calculated for a given  $\Delta P$ , gas temperature, and pressure via the quadratic formula. Equivalently, LFE calibration data can be cast into the dimensionless viscosity coefficient,  $V_c = \ell^2 r \Delta P / m^2$ , and flow coefficient,  $F_c = \ell^3 \Delta P / (m \dot{V})$ , where  $\dot{V}$  is the volumetric flow and  $\rho$  is the gas density. For the LFE flow calculations presented later in this paper, the midpoint

volumetric flow and density were used ( $P$  equals the average of the upstream and downstream pressures). For the LFE's tested herein, the kinetic energy (second) term is necessary to obtain flow measurements of the desired quality, but  $C_m/C_{KE}$  was  $> 50000$ , so first order LFE behavior can be analytically surmised from the first term alone.

Within the square brackets of Eqn. 2, parentheses separate quantities with and without temperature dependence. Temperature influences the mass flow calculation linearly (as opposed to the square root relationship for the CFV) which leads to the conclusion that for the same temperature uncertainty, the CFV should exhibit half the flow uncertainty of the LFE. As for the CFV, second order effects of temperature enter via (a) gas properties ( $\mu$ ), (b) thermal expansion of the flowmeter materials ( $r$  and  $\ell$ ), (c) the sensitivity of the calibration coefficients ( $C_m$  and  $C_{KE}$ ) to changing gas properties, and (d) the sensitivity of the calibration coefficients to temperature effects on the flow field not covered by the model of Eqn. 2.

#### Analytical Comparison of CFV and LFE Temperature Sensitivity

The first order effects of temperature are accounted for in both meters by making temperature measurements and applying the flow equations given in Eqn. 1 and Eqn. 2. It is common practice to correct for the effects of temperature that enter through the gas properties  $C^*$  and  $\mu$  since correlations that give these quantities as a function of  $P$  and  $T$  are available. It should be noted that for  $P$  between 100 kPa and 800 kPa and at  $T$  of 23 °C, the change in mass flow of the CFV due to  $C^*$  is  $-35 \times 10^{-6} \dot{m} / K$  or less while the change in mass flow of the LFE due to viscosity is  $-2600 \times 10^{-6} \dot{m} / K$ . Therefore, despite corrections to the mass flow made by using temperature to calculate gas properties, a given uncertainty in the measurement of temperature leads to a larger mass flow uncertainty for the LFE than for the CFV.

It is not common practice to make corrections to mass flow calculations for thermal expansion effects in either flowmeter type because they are generally considered too small to be of concern. The thermal expansion of the throat area of a CFV made of stainless steel will lead to a relative mass flow change of  $+35 \times 10^{-6} \dot{m} / K$ . For the LFE, the geometric quantity will lead to a relative mass flow change of  $+5.2 \times 10^{-6} \dot{m} / K$  for a stainless steel LFE.

The thermal effects on calibration coefficients are the most difficult of the three types to predict since they are determined by coupled heat transfer and fluid flow phenomena, but some prior work on this subject is available for CFV's. Johnson *et al.* (1998) have used computational fluid mechanics to study thermal effects on CFV's. They postulated two temperature conditions 1) a "cool" CFV wall temperature (i.e. an adiabatic CFV wall with zero temperature gradient and with the wall  $T$  matching the gas recovery  $T$ ), and 2) a "hot" CFV wall that matches the stagnation gas temperature and the ambient temperature (298.15 K). For the hot wall case, heat transfer from the CFV wall to the gas leads to a thermal boundary layer of lower velocity and density, resulting in a smaller mass flow and lower discharge coefficient. The magnitude of the flow reduction is dependent on the CFV Reynolds number: smaller flows (smaller CFV's) experience larger flow reductions because the thermal boundary layer is a more significant portion of the throat cross sectional area. For the lowest flow considered in the present transfer standard design (2 L/min of air), Johnson *et al.* (1998) computed a worst case change in discharge coefficient of 0.1 %.

In summary, analysis of Eqns. 1 and 2 shows that the LFE is subject to greater first order temperature sensitivity ( $T$  vs  $\sqrt{T}$ ) and larger  $T$  effects from gas properties, thermal expansion, and changes in the independent dimensionless calibration quantity. However, in the experiments that follow, we will see that these analytical sensitivities are sometimes less significant than temperature measurement errors that are the result of temperature gradients within the flowmetering system.

#### Temperature Sensitivity Experiments

Temperature sensitivity tests were conducted with two LFE designs and two CFV designs: (a) a Meriam Model 50MJ10-12 LFE, 11.4 L/min for  $\Delta P = 2$  kPa, (b) a DH Instruments, Model 1E4 Molbloc LFE, 10 L/min for  $\Delta P = 70$  kPa, (c) a Flow-dyne Engineering CFV,  $d = 0.3937$  mm, and (d) an Hirai Model MRS103 CFV,  $d = 0.48$  mm. The Molbloc and the Hirai CFV are equipped with their own  $P$  and  $T$  instrumentation and flow computers that implement their manufacturer's flow calculations based on their calibrations.

The Meriam LFE consists of approximately 200 parallel flow paths that have sinusoidal cross sections, each with hydraulic radius of about 0.2 mm and length of 8 cm. It is made of stainless steel and weighs 1.8 kg. The Model 1E4 Molbloc uses an annular flow crevice about 70 microns wide, 1.5 cm in diameter, and 8 cm long. The Molbloc measures gas temperature by averaging the readings of two PRT's embedded in the flowmeter body (not in direct contact with the gas). The Model 1E4 Molbloc is made of stainless steel and weighs 2.3 kg. The stainless steel, 0.9 kg Flow-dyne CFV follows the ISO 9300 toroidal throat design specifications and used a diverging section half angle of 4 degrees. The Hirai MRS103 also meets the ISO 9300 toroidal throat specifications with a diverging section half angle of 3 degrees, is made of stainless steel, and weighs 1.5 kg. It should be noted that the flowmeter calibration results that follow were performed on only a single meter of each type and do not necessarily represent the performance of all flowmeters of that design. Further, the manufacturers employ different designs for other flow ranges that are likely to show different calibration and temperature sensitivity results.

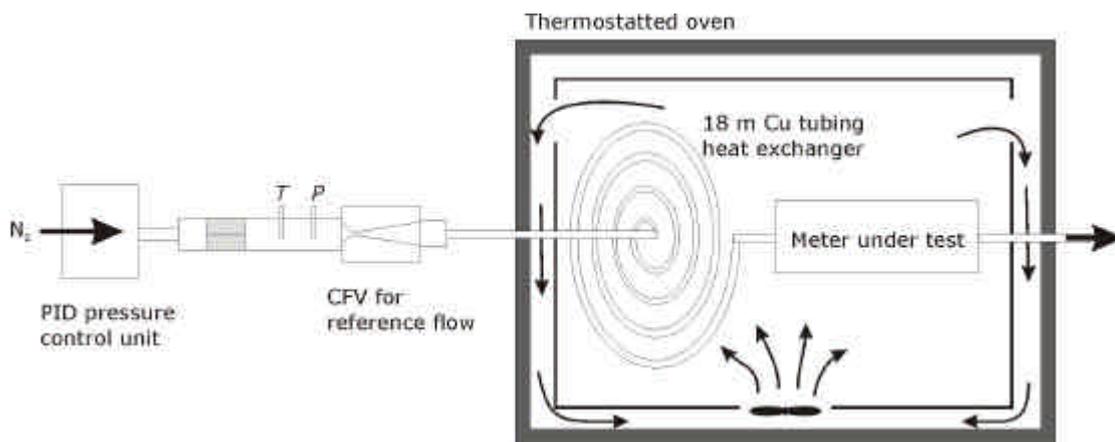


Figure 3. Schematic of the first permutation of temperature sensitivity test performed on two LFE and two CFV designs. In a second permutation, the heat exchanger was outside the thermostatted oven and the meter under test inlet was placed within 15 cm of the oven wall.

There are a number of possible permutations of 1) gas, 2) flowmeter body, and 3) environmental temperature conditions, but two were explored because of their experimental practicality and their bearing on the transfer standard design. The first permutation is the one depicted in Fig. 3 with the heat exchanger in the oven. In this permutation the entrance gas temperature and the flowmeter body temperature were equal within 0.2 K or better at steady state conditions. This test permutation examines the situation where both the transfer standard and the gas entering the meter are at room temperature, but the temperature is different from laboratory to laboratory.

The second test permutation placed the heat exchanger outside the oven. The meter under test was placed as close to the wall of the oven as practical so that there was only a short length (< 15 cm) of tubing upstream of the flowmeter and in the oven. In this manner, gas at nearly room temperature entered the flowmeter while the oven temperature was increased in four hour long steps to approximately 313 K. This permutation examines the case where the flowmeter is at a steady state temperature, but the entering gas is cool either because it comes from another room or has been cooled by expansion through a regulator from a higher pressure. The test examines the question: is a heat exchanger necessary for the transfer standard to achieve the desired level of reproducibility under varying laboratory temperature conditions?

The temperature sensitivity tests were all conducted at a nominal flow of 3.8 L/min. Nitrogen (0.99998 purity) flowed through a PID controlled solenoid regulator (Hirai Model 350A pressure control unit) to a reference 0.2921 mm CFV that was kept at room temperature ( $297 \pm 0.5$  K). The output flow of the reference CFV was then passed through a 18 m long coil of 13 mm diameter copper tubing (which served as a heat exchanger), and then through one of the four meters under test. The meter under test was housed in a thermostatted oven with uniformity better than 0.2 K. The oven temperature was incremented by 2 K every four hours from room temperature up to 313 K. The long heat exchanger tube and the four hours between temperature steps were used to attain good thermal equilibrium throughout. (The stability of the oven temperature was poor below 305 K because there was no active cooling in the temperature control system, hence only data for 305 K to 313 K is shown in the following plots.)

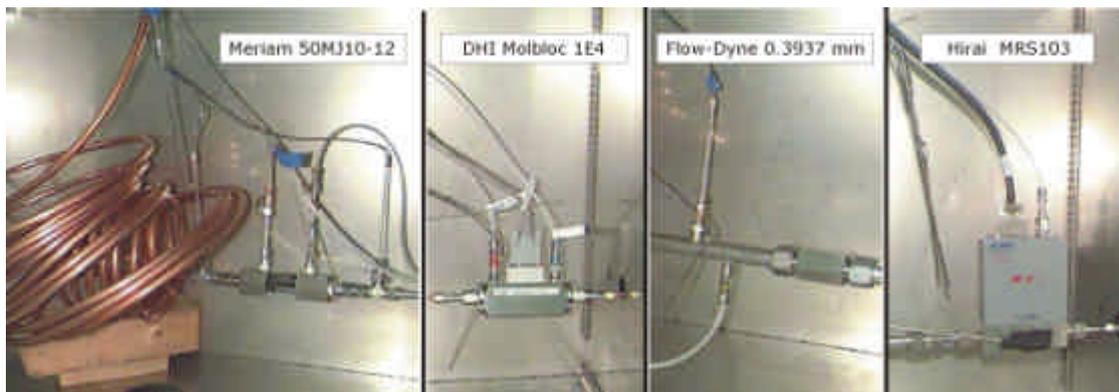


Figure 4. Photographs of the four flowmeters tested in the temperature sensitivity tests (first test permutation with heat exchanger in the oven).

Thermistors in 3 mm stainless steel sheaths with time constants of approximately 10 s and calibration uncertainty of 20 mK were used to measure most of the temperatures. The Molbloc and MRS103 temperatures were measured with an uncertainty of 200 mK by their dedicated sensors. For the Meriam LFE, temperature sensors were installed in the piping immediately upstream and downstream from the LFE and the two values were averaged and used as the LFE gas temperature. Most pressure measurements were made with Paroscientific absolute pressure transducers with uncertainty of 0.04% (based on calibration control charts and calibration uncertainties). The Meriam differential pressure was redundantly measured with two Mensor transducers with an uncertainty of 0.2%. The MRS103 pressure was measured with a Yokogawa sensor with 0.02 % uncertainty. The uncertainty of the mass flow provided by the reference CFV was 0.05%.

The uncertainties listed above are given for completeness, but it should be noted that the temperature sensitivity experiments are relative in nature. We are looking for changes in the ratio of two flowmeter outputs with one flowmeter held at room temperature ( $\pm 0.5$  K) and the other at the oven temperature. Therefore the stability of the sensors used to calculate flow (much smaller than the uncertainties given above) and the uncertainty of the temperature sensors used in the oven are the principle concerns because the pressures are nearly constant throughout the test and the drift in pressure sensor calibration during the test was very small.

As previously stated, the Molbloc LFE and the Hirai CFV are equipped with their own  $P$  and  $T$  instrumentation and flow computers that implement their manufacturer's flow calculations based on their calibrations. The raw  $P$  and  $T$  data are also available, so flow calculations can also be made based on Eqns. 1 and 2 and a calibration against our flow standards. The results of both flow calculations are presented herein. Only flows from our calibrations and calculation methods are presented for the Meriam LFE and Flow -dyne CFV. More specifically, before the temperature sensitivity testing, the meter under test was calibrated at room temperature against the reference CFV over a range of flows spanning the temperature sensitivity test flow of 3.8 L/min. The room temperature data were used to calculate dimensionless calibration curves. The calibration curve was used, along with the appropriate gas properties calculated from  $T$  and  $P$  measurements made during the oven to calculate the mass flow of the meter under test. Except where otherwise stated, properties in this paper were calculated from the NIST "Refprop" database (Lemmon et al., 2002). Therefore, the customarily implemented corrections have been made in the flow calculations: the first order effects of temperature in Eqns. 1 and 2, as well as effects that enter through the gas properties and through changes in the independent variable of the calibration curve ( $V_c$  and  $Re$ ). The second order effects of thermal expansion and any other thermal phenomena unaccounted for in the physical models of Eqns. 1 and 2 remain uncorrected in the following results (except perhaps in the manufacturer's flow calculations).

#### *Meriam LFE Temperature Sensitivity*

In Figure 5, the ratio of the mass flow measured by the Meriam LFE to the reference CFV mass flow (the "flow ratio") is plotted versus elapsed time. Also plotted (on the secondary axis) are the air temperature within the oven and the LFE inlet and outlet gas temperatures. The upper plot in Fig. 5 shows the results for the first permutation temperature test. With the heat exchanger in the oven, the LFE inlet and outlet temperatures and the oven gas  $T$  agree within 0.08 K, once steady state is achieved. The differences in the flow ratio at steady state seen in Figure 5 ( $-80 \times 10^{-6} \dot{m}/K$ ) represent our measurement of the sensitivities of the LFE to

temperature that are not customarily accounted for by users. Note that corrections for thermal expansion of the flowmeter materials ( $+50 \times 10^{-6} \dot{m}/\text{K}$ ) have not been made.

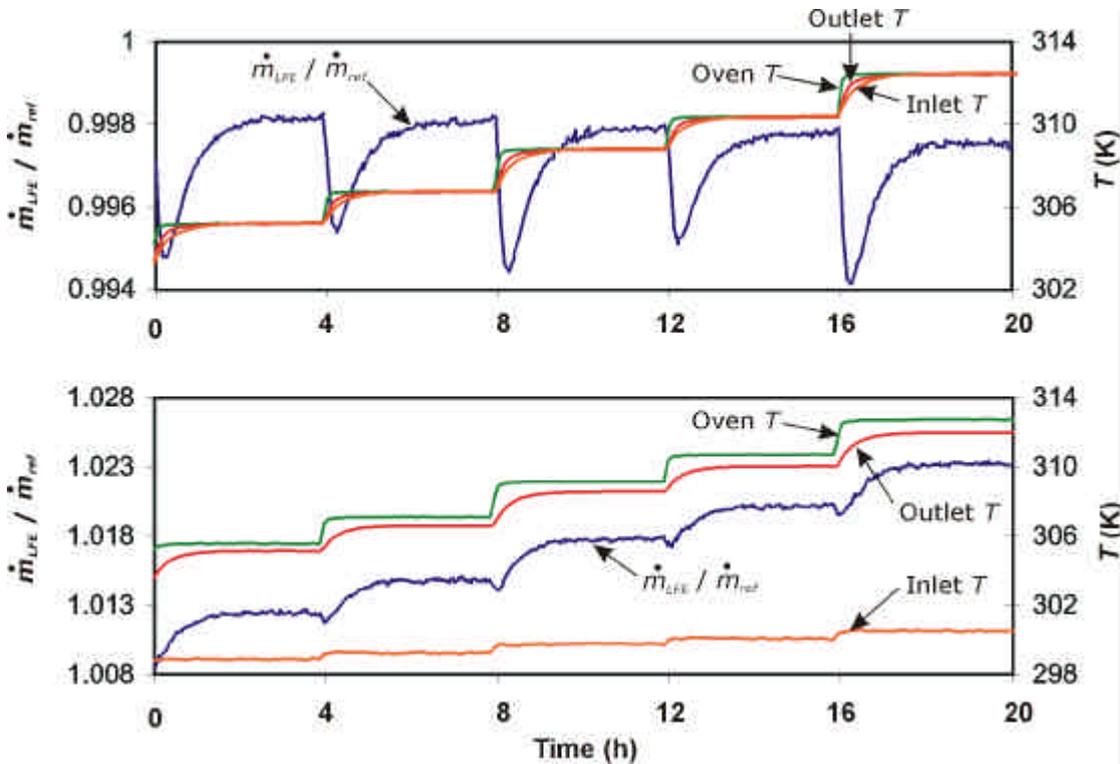


Figure 5. Meriam LFE temperature sensitivity test results. First permutation (above) with heat exchanger inside oven (gas  $T =$  meter  $T =$  environmental  $T$ ), second permutation (below) with heat exchanger outside oven (gas  $T <$  meter  $T <$  environmental  $T$ ).

The lower half of Fig. 5 gives the second test permutation results for the Meriam LFE. The LFE inlet gas  $T$  is as much as 8 K cooler than the oven  $T$ . The flowmeter body heats the gas as it flows through the LFE. The average of the LFE inlet and outlet  $T$ s was used in the LFE flow calculation. The upstream gas temperature increased about 2 K during the course of the test due to heat transfer through the short length of tube upstream of the flowmeter and inside the oven. The gas was heated as much as 7 K as it flowed through the flowmeter. However, the gas did not reach equilibrium with the oven temperature as it flowed through the LFE as evidenced by the difference between the oven temperature and the downstream gas temperature (0.4 K to 0.9 K). The LFE mass flow changed by  $1800 \times 10^{-6} \dot{m}$  per degree of difference between the oven and inlet gas temperatures. Thus a difference of 2 K between the environmental and the inlet gas temperatures leads to a 0.36 % change in the mass flow reported by the flowmeter. The reason for the mass flow over prediction is probably due to “sampling errors” in the gas temperature measurement: the gas temperature in the LFE is closer to the outlet temperature than to the inlet temperature and the average of these two temperatures is inadequate.

The time constant for the air temperature within the oven for this and all of the temperature tests is 0.1 h. The time constant of the flowmeter gas temperature and the flow ratio is

approximately 0.47 h. Steady state is considered achieved after five time constants (2.4 h in this case).

DH Instruments 1E4 Molbloc LFE Temperature Sensitivity

The results of the  $T$  sensitivity tests for the DH Instruments 1E4 Molbloc LFE are shown in Fig. 6. The mass flow ratio for both the manufacturer's proprietary flow output (from the Molbox) and the flow calculated via Eqn. 2 are shown in the figure. Also shown (on the secondary axis) are the oven air temperature and the Molbloc temperature (the average of two sensors located in the flowmeter body). For the first test permutation, the manufacturer's model shows thermal sensitivity of  $100 \times 10^{-6} \dot{m} / K$  while the Eqn. 2 model sensitivity is  $30 \times 10^{-6} \dot{m} / K$ . The time constant for the Molbloc was approximately 0.6 h. At steady state, the temperature difference between the oven air temperature and the Molbloc temperature measurement increased from 0.03 K to 0.2 K over the range of temperatures tested (perhaps due to  $T$  sensor calibration uncertainty).

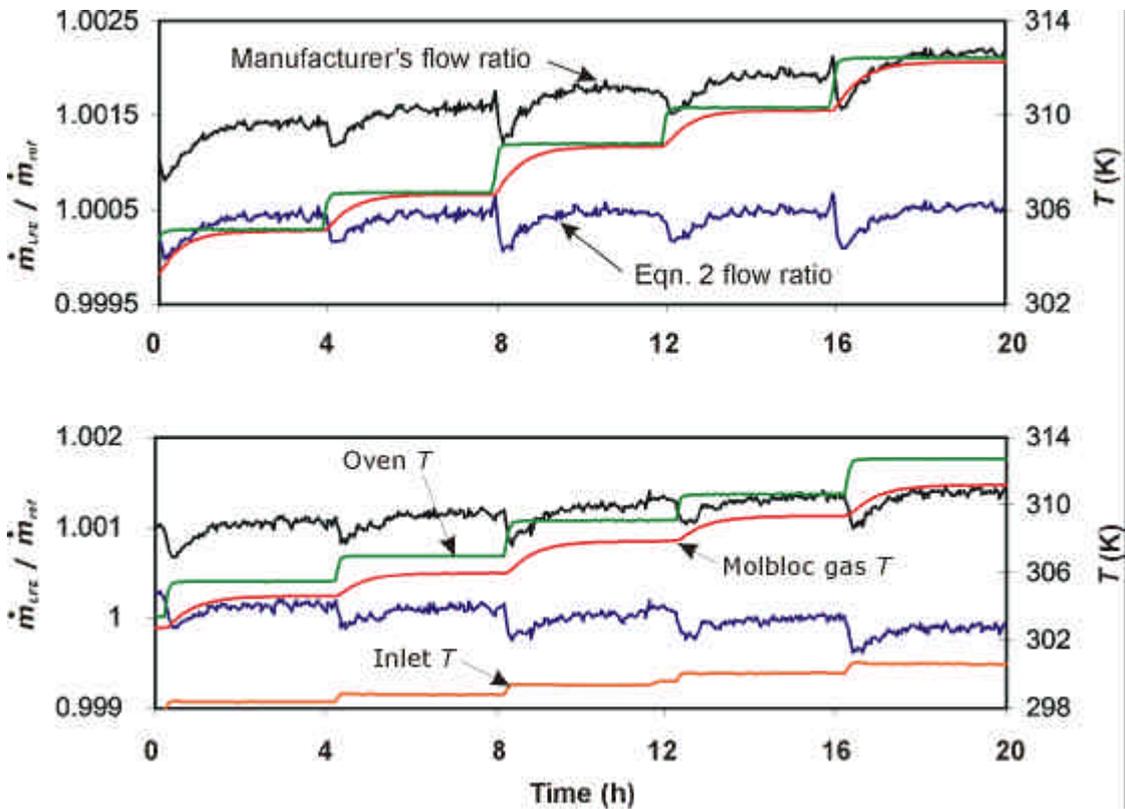


Figure 6. DH Instruments Model 1E4 Molbloc temperature sensitivity results.

The second permutation test results for the Molbloc 1E4 LFE are shown in the lower half of Fig. 6. The manufacturer's flow changed by  $70 \times 10^{-6} \dot{m}$  per degree of difference between the oven and inlet gas temperatures. The flow calculated via Eqn. 2 changed by  $-45 \times 10^{-6} \dot{m}$  per degree of difference between the oven and inlet gas temperatures. These very small temperature sensitivities are apparently due to the excellent heat transfer characteristics of the annular laminar flow path. The Molbloc patent (Delajoud, 1995) specifically mentions this as a design feature of the flowmeter. The narrow annular crevice has a short thermal entrance

length relative to the LFE length, therefore the laminar flow path acts as a heat exchanger, "thermalizing" the inlet gas and minimizing the errors due to temperature gradients and gas  $T$  measurement errors.

Flow-dyne 0.3937 mm CFV Temperature Sensitivity

The Flow-dyne CFV  $T$  sensitivity results are presented in Figure 7. A 3 mm hole drilled into the CFV body allowed a temperature sensor to be inserted so that the body temperature could be measured, and this data is plotted along with the inlet gas  $T$  (used as  $T_0$  in Eqn. 1) and oven air temperature. The temperature sensitivity for the first permutation is  $-20 \times 10^{-6} \dot{m} / K$ . Thermal expansion is expected to cause  $-30 \times 10^{-6} \dot{m} / K$ , therefore the effects of the thermal boundary layer on the discharge coefficient are  $-55 \times 10^{-6} \dot{m} / K$ .

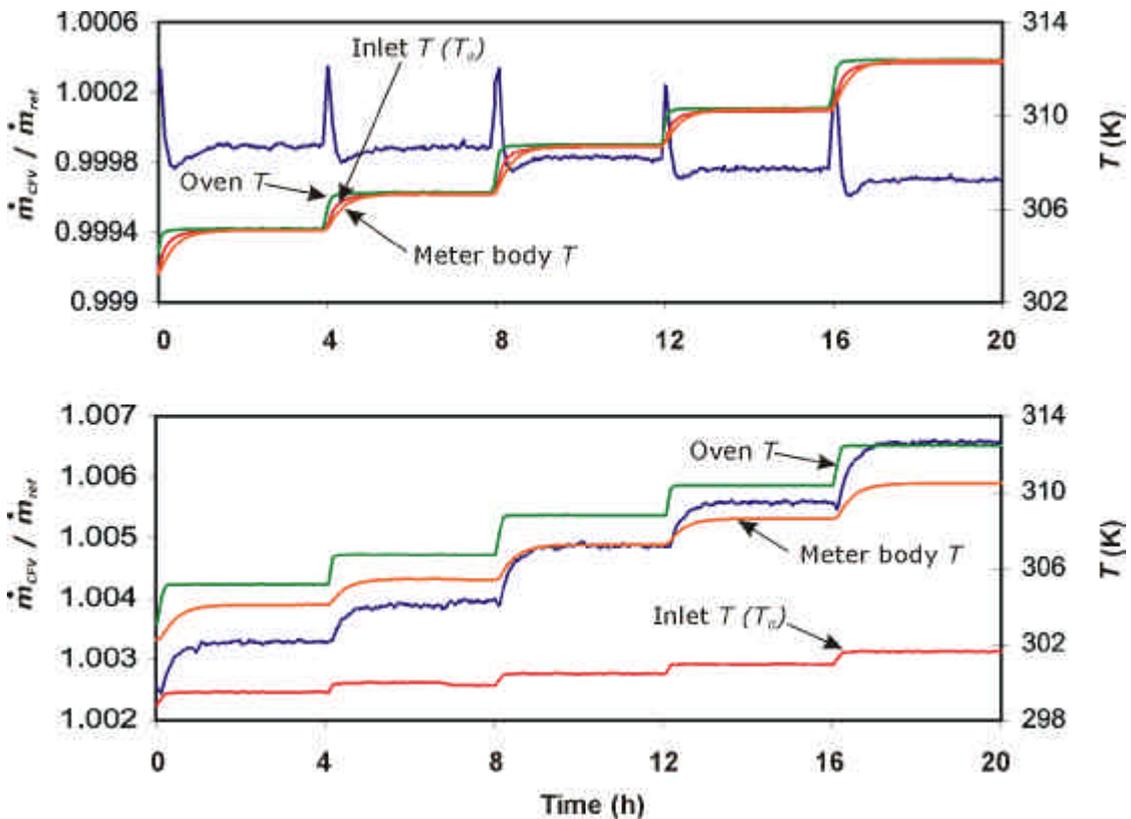


Figure 7. Flow -dyne 0.3937 mm CFV temperature sensitivity results.

The time constants of the flowmeter gas and the CFV body temperatures were 0.33 h. The positive transients in the CFV flow ratio seen during the temperature step changes are caused by the CFV pressure sensor having a shorter response time than the CFV temperature sensor.

In the second test permutation, the CFV gas temperature was measured 10 cm upstream from the CFV throat and the rather large sensitivity of the CFV mass flow to temperature ( $700 \times 10^{-6} \dot{m}$  per degree of difference between the oven and inlet gas temperatures) is most likely caused by the gas temperature increasing before it reaches the throat due to heat transfer from the pipe walls. A problem of this sort could be diagnosed (and corrected) by placing two temperature sensors different distances upstream from the throat. Significant

differences between these  $T$  sensors would indicate significant heat transfer and erroneous CFV temperature measurements. Alternatively, a single  $T$  sensor could be placed closer to the throat or a heat exchanger could be used upstream.

Hirai MRS103 CFV Temperature Sensitivity

The temperature test results for the Hirai CFV are shown in Fig. 8. For the first test permutation, the manufacturer's flow calculations showed a sensitivity of  $-25 \times 10^{-6} \dot{m}/K$  and the flow calculations based on our calibration and Eqn. 1 showed a sensitivity of  $-55 \times 10^{-6} \dot{m}/K$ . An exposed bead PRT 3.5 cm upstream from the CFV throat measures the gas temperature in the MRS103. The flowmeter body temperature was measured with a NIST thermister in good thermal contact with the stainless steel body (see Fig. 4). The flowmeter body temperature matched the MRS103 gas temperature measurement within 0.1 K at steady state conditions and therefore they appear to be a single trace in the top half of Fig. 8.

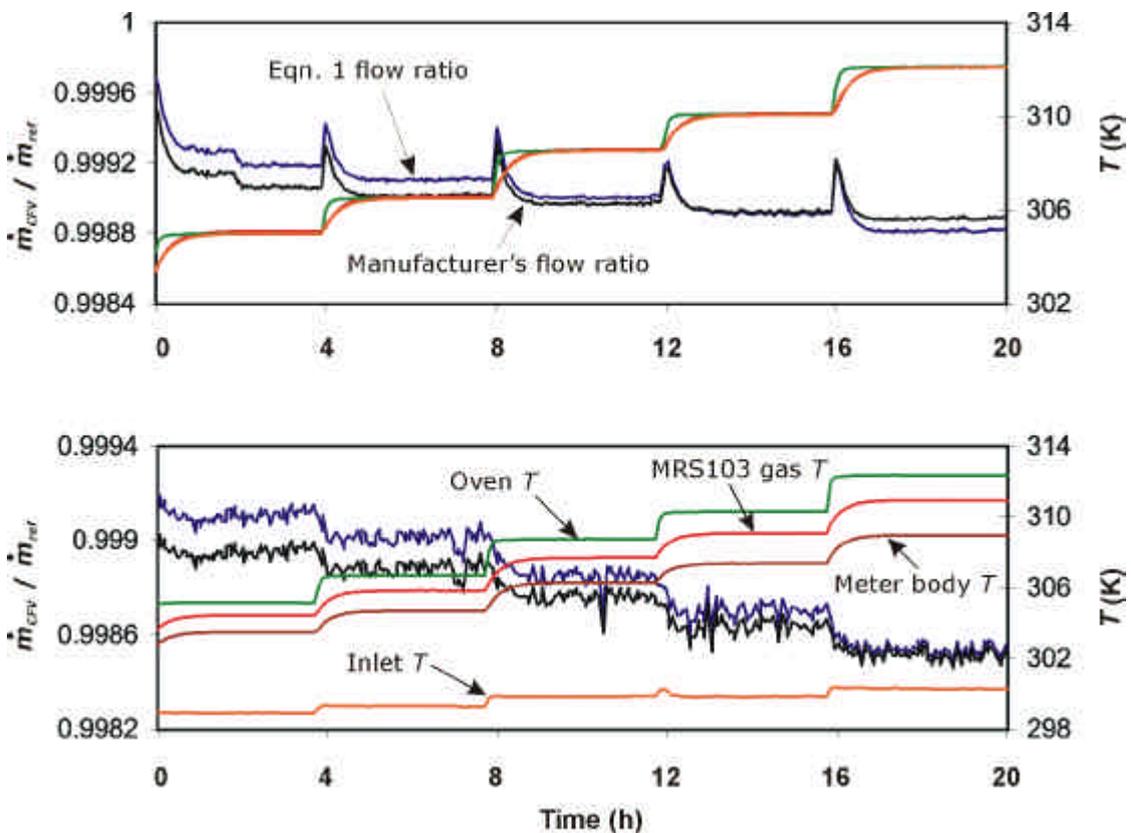


Figure 8. Hirai MRS103 CFV temperature sensitivity data.

In the second test permutation, the MRS103 gas temperature was greater than the flowmeter body temperature by 0.5 K to 2 K, indicating that significant heat transfer was occurring along the flow path. However the MRS103 showed temperature sensitivity of only  $-80 \times 10^{-6} \dot{m}/K$  (manufacturer) and  $-95 \times 10^{-6} \dot{m}/K$  (Eqn. 1). The close proximity of the temperature sensor to the CFV throat, as compared to the Flow-dyne CFV separation of 8 cm, leads to a considerable improvement in the CFV temperature sensitivity. The time constants for the gas temperature and the flowmeter body temperature were 0.4 h.

A summary of the temperature sensitivity test results for the four flowmeters tested is given in Table 1. The time constant for the flowmeter to reach equilibrium (flow equilibrium and thermal equilibrium) was approximately proportional to the mass of the flowmeter. The time constants of the gas temperature used to calculate flow, the flowmeter body temperature, and the mass flow ratio were nominally equal for each of the flowmeters. The time constants ranged from 0.33 h to 0.6 h.

The temperature sensitivity was less than  $100 \times 10^{-6} \dot{m}/K$  for all four flowmeters in the first test permutation. For the second test permutation, the Molbloc and the MRS103 both showed sensitivities less than  $100 \times 10^{-6} \dot{m}/K$  because their gas temperature measurements experienced relatively small errors. These two flowmeters either incorporate heat transfer concerns into the flowmeter design or place the temperature sensor in good locations so as to minimize temperature "sampling" errors. The Meriam LFE and the Flow-dyne CFV (both instrumented with NIST sensors) showed temperature sensitivities of  $1800 \times 10^{-6} \dot{m}/K$  and  $700 \times 10^{-6} \dot{m}/K$  respectively, probably because of temperature sampling errors. The temperature sensitivities of both of these flowmeters could be improved by adding a heat exchanger upstream and by placing the temperature sensor closer to the flow element.

Table 1. Temperature sensitivities and time constants for the four flowmeters at 3.8 L/min. Manufacturers' flow ratio sensitivities are in parentheses.

Flowmeter Model	Meter mass (kg)	Time constant (h)	1 <sup>st</sup> permutation sensitivity $\dot{m}/K$ ( $\times 10^6$ )	2 <sup>nd</sup> permutation sensitivity $\dot{m}/\Delta K$ ( $\times 10^6$ )
Meriam 50MJ10-12	1.8	0.47	-80	1800
DH Instruments 1E4 Molbloc	2.3	0.60	30 (100)	-45 (70)
Flow-dyne 0.3937 mm CFV	0.9	0.33	-20	700
Hirai MRS103	1.5	0.35	-55 (-25)	-95 (-80)

#### Gas Composition Sensitivity

In this section we will consider the effects of gas composition on the uncertainty of flow measurements made with CFV's and LFE's. First we will survey the state of knowledge about species effects on the two flowmeter types and present experimental data for the 0.3937 mm Flow-dyne CFV and the 1E4 Molbloc with pure nitrogen, air, argon, helium, and carbon dioxide. Then we will consider the error introduced by moisture in air that is assumed to be perfectly dry.

#### Species Effects in the CFV

In the case of the CFV, changes of 2 % or more in discharge coefficient have been observed for certain gases (e.g. CO<sub>2</sub> and SF<sub>6</sub>) at small Re (i.e. throat diameters) due to the significant energy in their molecular vibrational states at room temperatures (Johnson et al., 1998, 2000). Experiments showing the anomalous behaviors of CO<sub>2</sub> and SF<sub>6</sub> were published by Nakao et al. (1997). The CFV discharge coefficients for four of the same gases used by Nakao et al. (nitrogen, argon, helium, and carbon dioxide) and also in dry air were recently measured at NIST with our 34 L PVTt system (Wright et al., 2003). The results of the NIST calibrations and of Nakao et al. are shown in Fig. 9. Nakao et al. used a CFV with throat diameter of 0.5935 mm and the NIST data were collected for a CFV with nominal  $d = 0.3937$  mm (the

same CFV used in the thermal sensitivity tests). The data of Nakao et al. is only plotted over the range of Re tested (no extrapolation). The NIST CFV throat diameter has been adjusted to achieve matching  $C_d$  values for nitrogen. NIST  $C_d$  measurements in dry air have been included although no air data was available in the Nakao et al. publication. The differences between the dry air calibration and the nitrogen calibration are less than 0.05 %. As previously stated, species effects are larger at small Re (i.e. large  $Re^{-1/2}$ ) due to greater significance of the boundary layer. For the smallest proposed flow of the key comparison (2 L/min),  $Re^{-1/2}$  is about 0.01.

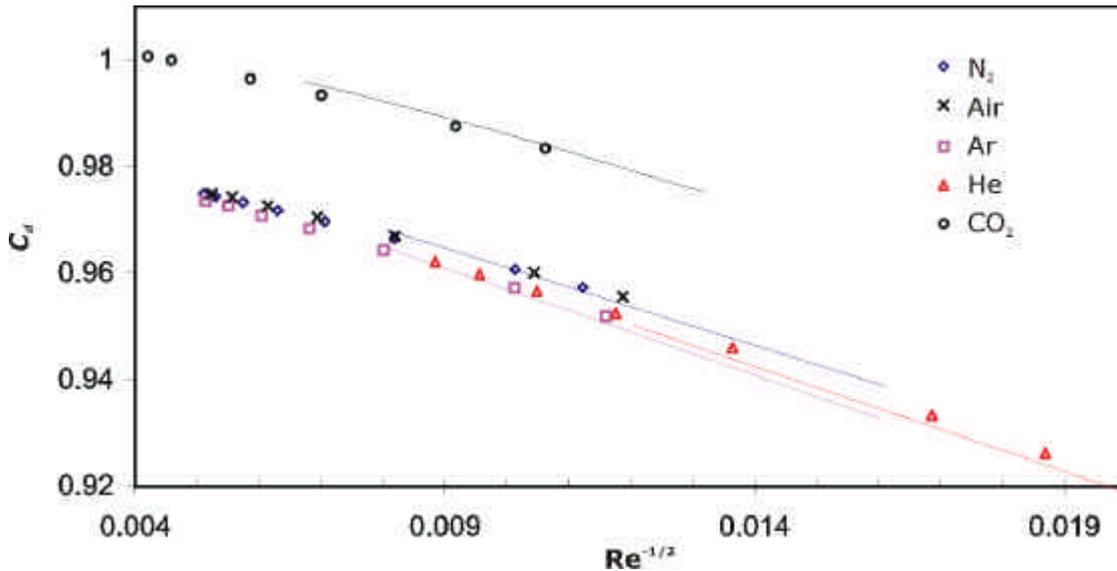


Figure 9. The discharge coefficient versus Reynolds number for various pure gases. Solid lines are fits to data of Nakao et al. (1997) and symbols are NIST data.

The agreement between the two data sets is excellent with the differences in the overlapping Re ranges never greater than 0.25 %. The agreement verifies the systematic effects of gas species on  $C_d$ , not just the large vibrational relaxation effects (>2 %) found with carbon dioxide, but smaller systematic differences between (a) the noble gases argon and helium and (b) nitrogen and air (-0.4 %). These differences have been considered previously by Johnson et al. (1998) and are consistent with their physical explanation that gases with larger specific heat ratio lead to thicker thermal boundary layers and therefore smaller  $C_d$  values. These mechanisms are not incorporated into the relatively simple physical model represented in Eqn. 1 and therefore manifest themselves as the species effects or gas composition effects seen in Fig. 9. Fortunately, these sensitivities are significant only at small Re, and more importantly, are negligible for the level of gas composition uniformity anticipated during our key comparison as evidenced by the agreement between the nitrogen and dry air calibration results.

#### Species Effects in the LFE

The 0.3937 mm CFV, now calibrated in five gases, was used as a reference flowmeter to check the species sensitivity of the same Molbloc 1E4 LFE that was used during the thermal sensitivity studies. The test consisted of applying upstream pressures between 200 kPa and 700 kPa to the 0.3937 mm CFV with the CFV output connected to the LFE inlet. We then

waited 15 minutes for conditions to stabilize and acquired pressure and temperature measurements from both flowmeters. Figure 10 presents the calibration data in the dimensionless  $V_c$  and  $F_c$  format. The dimensionless data collapse to a single calibration curve within a  $\pm 0.3\%$  band for all five gases. The argon results are approximately 0.3% higher than the other four gases, probably due to error in the viscosity of argon. The ratio of the room condition viscosities of argon and nitrogen is 1.2670 for Refprop (Lemmon, 2002, used for Fig. 10) while other researchers (Vogel, 1984) have measured the ratio to be 1.2709. The data of Fig. 10 leads to a viscosity ratio of 1.2708. Hence, using Vogel's argon viscosity values would result in all five gases agreeing within  $\pm 0.15\%$ . The difference in calibration between dry air and nitrogen is less than 0.15 %.

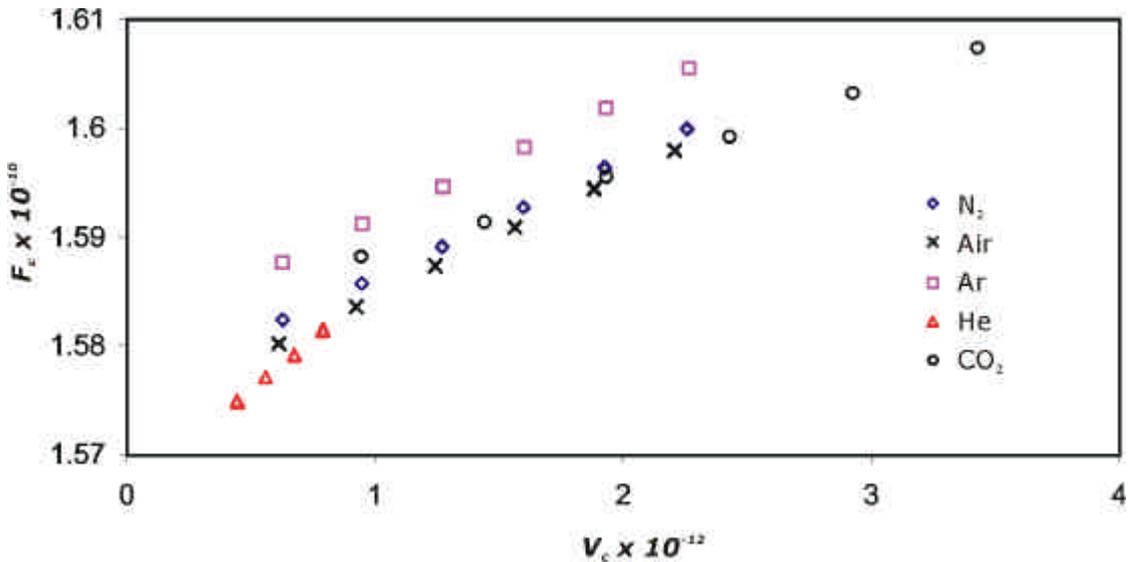


Figure 10. Calibration data for the Model 1E4 Molbloc LFE in five gases.

The Effect of Moisture in "Dry Air"

We anticipate "dry air" as the medium for the key comparison, but variations in performance of driers attached to air compressors means that we can expect composition variations between the participants. We will assume that the species effects described above are negligible for the small composition changes considered now, and hence we will rely on the models of Eqns. 1 and 2 in the following analysis.

The first order sensitivities of a CFV flow measurement to gas composition can be derived from Eqn. 1. Gas composition enters via the molecular weight and critical flow factor in the form  $C^* \sqrt{M}$ . Hence an estimate of the effects of moisture content on CFV flow can be made by examining  $(C^* \sqrt{M}) / [(C^* \sqrt{M})]_{dry}$ . An analogous approach to the LFE flow equation yields the ratio  $(M/m) / [(M/m)]_{dry}$ . In Fig. 11,  $(C^* \sqrt{M}) / [(C^* \sqrt{M})]_{dry}$  and  $[(M/m)]_{dry} / (M/m)$  are plotted for air at constant pressure and temperature and dew point temperatures ( $T_{dp}$ ) from 230 K to 280 K. (The inverse of the composition sensitivity for the LFE has been plotted to allow simpler visual comparison between the curves for the two meter types.) The sensitivities of the two meter types are opposite in sign, but quite similar in magnitude. If a user were to assume completely dry air when the actual dew point temperature was 250 K, the CFV would give an error of

$-100 \times 10^{-6} \dot{m}$  while the LFE would have an error of  $125 \times 10^{-6} \dot{m}$ . Although  $\partial \dot{m} / \partial T_{dp}$  is 20 times larger than  $\partial C^* / \partial T_{dp}$ , cancellation occurs with  $\partial M / \partial T_{dp}$  for the LFE. Hence there is not a significant advantage for one meter type over the other with respect to the first order effects of gas composition, but it is clear that both meter types will suffer significant inter-laboratory differences if moisture content is not controlled or corrected for in flow calculations. Based on Fig. 11, a maximum  $T_{dp}$  of 250 K is desirable during the key comparison.

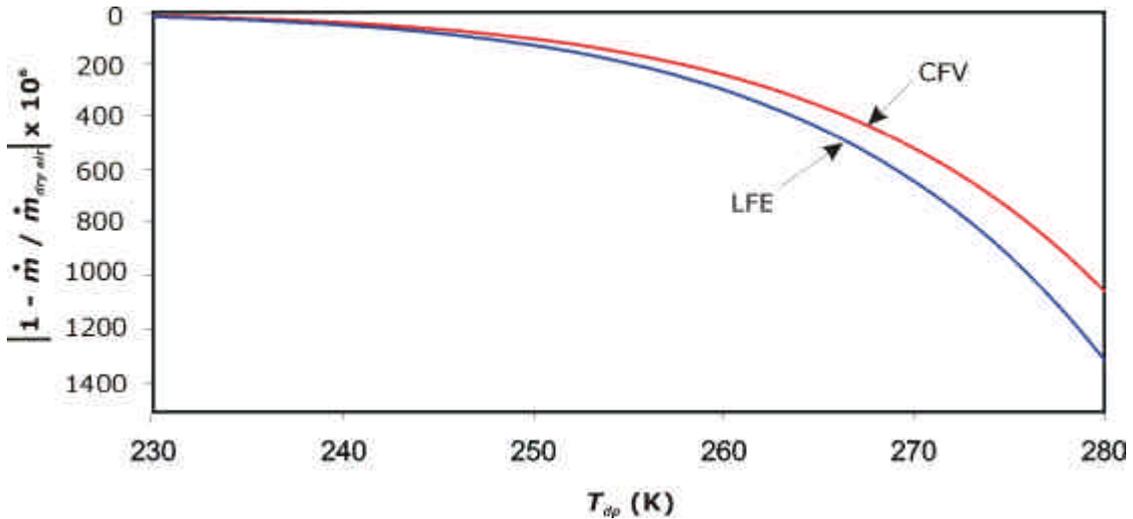


Figure 11. Sensitivities of CFV and LFE flow measurements to moisture content of air.

There is a wide range of composition of "air" sold in compressed cylinders. Air in compressed cylinders is often mixed from the component gases, and the tolerance on the mole fraction of the constituents is not necessarily tightly controlled. These differences in air composition will have a larger influence on  $M/m$  (on the LFE) than on  $C^* \sqrt{M}$  (on the CFV).

In summary, the CFV exhibits sensitivity to gas composition through reasonably well understood mechanisms, vibrational relaxation and thermal boundary layer phenomena, which are not incorporated into the widely used physical model of Eqn. 1. The most significant problem for the LFE with respect to gas composition is uncertainty of viscosity. In fact uncertainty of viscosity is an impediment to more careful experimental study of LFE species effects and hampers improvements in the physical model of Eqn. 2. The physical model for both flowmeter types are valuable for assessing the effects of the most likely composition issue, the moisture content of "dry air". If it is possible to maintain a dew point temperature of 250 K or less for dried, atmospheric composition air, the uncertainty introduced by gas composition sensitivity will be  $125 \times 10^{-6} \dot{m}$  or less. If gas from compressed cylinders is to be used, its composition must be tightly controlled to match that of dried, atmospheric air, or significant errors will be introduced, especially for an LFE.

Calibration Stability

The ability of a flowmeter to maintain a constant calibration through the rigors of shipment and over long periods of time is perhaps the most important characteristic of a transfer standard. Based on our review of the CMC's, transfer standard calibration stability of 0.05 %

or better is desired. Fortunately, this level of stability seems attainable from both the CFV and the LFE. Neither meter type has any moving parts, such as bearings, to wear or change in friction with usage. Therefore the long-term changes in calibration of the LFE and CFV are due to changes in the associated pressure and temperature sensors or in the physical dimensions of the flow passages within the flowmeter primary element. The calibration stability of commercially available pressure and temperature sensors is quite good based on our calibration experience. Barring shock damage during shipment, we can expect stability as good as 0.02 % for  $P$  and  $T$ . Also, stability and damage can be assessed and controlled by making redundant  $P$  and  $T$  measurements. Therefore these measurements do not pose a serious impediment to our 0.05 % stability goal.

We will now consider the effects of dimensional changes on the two flowmeter types, particularly the effects of a coating on the flow path surfaces. Both meter types should be used with clean, filtered gas. But, over time, a thin layer of oil or fine dirt may be deposited by dirty gas flowing through the flowmeter. Can we make any generalizations about the effects of such a coating on the flow measurements? CFV mass flow is proportional to the square of the radius of its throat, while the LFE flow is proportional to the fourth power of its hydraulic radius. Therefore, if one postulates a coating of given thickness on each flowmeter type, the decrease in flow will be twice as large for the LFE as it is for the CFV, i.e.

$$\left[\frac{\partial \dot{m}}{\partial r}(r/\dot{m})\right]_{LFE} = 2 \left[\frac{\partial \dot{m}}{\partial r}(r/\dot{m})\right]_{CFV}.$$

Another generalization regarding coatings can be made about the LFE. If the LFE is designed to have a smaller hydraulic radius in order to generate a larger and more easily measurable differential pressure, the LFE will be more sensitive to a given sized coating. Using the approximate dimensions previously given for the three flowmeters used during the thermal sensitivity tests, a coating of 1 micron would cause an indicated flow change of 8 % in the Molbloc, 2 % in the Meriam LFE, and 1 % in the CFV. For the range of LFE designs with differential pressure levels that are practical to measure, a CFV with the same flow range will have a larger minimum dimension and will be less subject to fouling by dirt. It also seems that sonic velocities at the throat are more likely to keep the CFV clean.

A previous paper examined CFV and LFE calibration stability over periods as long as 30 years by examining the records of meters periodically sent to NIST for calibration (Wright, 1998). In this study, there were examples of both meter types that maintained their calibration for decades within the uncertainty of the calibration system ( $\sim 0.3\%$ ). Given the relative simplicity of the flowmeter measurement systems and their lack of moving parts, it seems likely that some of these flowmeters were more stable than the primary standards used to calibrate them.

Our ability to experimentally examine the calibration stability of CFV's has been dramatically improved by the introduction of a new  $PVTt$  primary standard that measures mass flow of nitrogen gas with an uncertainty of 0.02 % ( $k = 2$ ) (Wright et al., 2003). Additional uncertainties of 0.04 % for both the CFV pressure and temperature lead to discharge coefficient or CFV mass flow uncertainty of 0.05 %. The  $PVTt$  standard was used to perform periodic calibrations on a set of Flow-dyne CFV's at flows ranging from 1 L/min to 80 L/min. Some of the calibration results for four of the CFV's over an eight month period are presented in Fig. 12. The plots show the difference between the mass flow calculated from the CFV using a  $C_d$  function best-fitted to all of the calibration data and the mass flow as measured by the  $PVTt$  system on a particular date. Averages of numerous, repeated calibration measurements

are plotted in Fig. 14 to reduce clutter. The standard deviation of the data averaged to obtain the individual points plotted in Fig. 12 was typically  $20 \times 10^{-6} \dot{m}$ . The standard deviation of the changes in mass flow of the individual calibration points (before averaging) varied between  $46 \times 10^{-6} \dot{m}$  and  $154 \times 10^{-6} \dot{m}$ . Calibration results for some dates are not plotted in Fig. 14 (particularly for the 0.2921 mm CFV) because they differed by as much as  $700 \times 10^{-6} \dot{m}$  from the data shown here. We suspect that these errant results were caused by small leaks in the  $37^\circ$  flare fittings on the CFV's. None of the CFV's show a monotonic trend in calibration drift over time. The measured changes are well below the uncertainty of the test and the most likely sources of the variations are the pressure transducer used to measure the CFV pressure and leaks. We conclude that the four CFV's show calibration stability better than our ability to measure it (0.05 %) and probably better than 0.02 %.

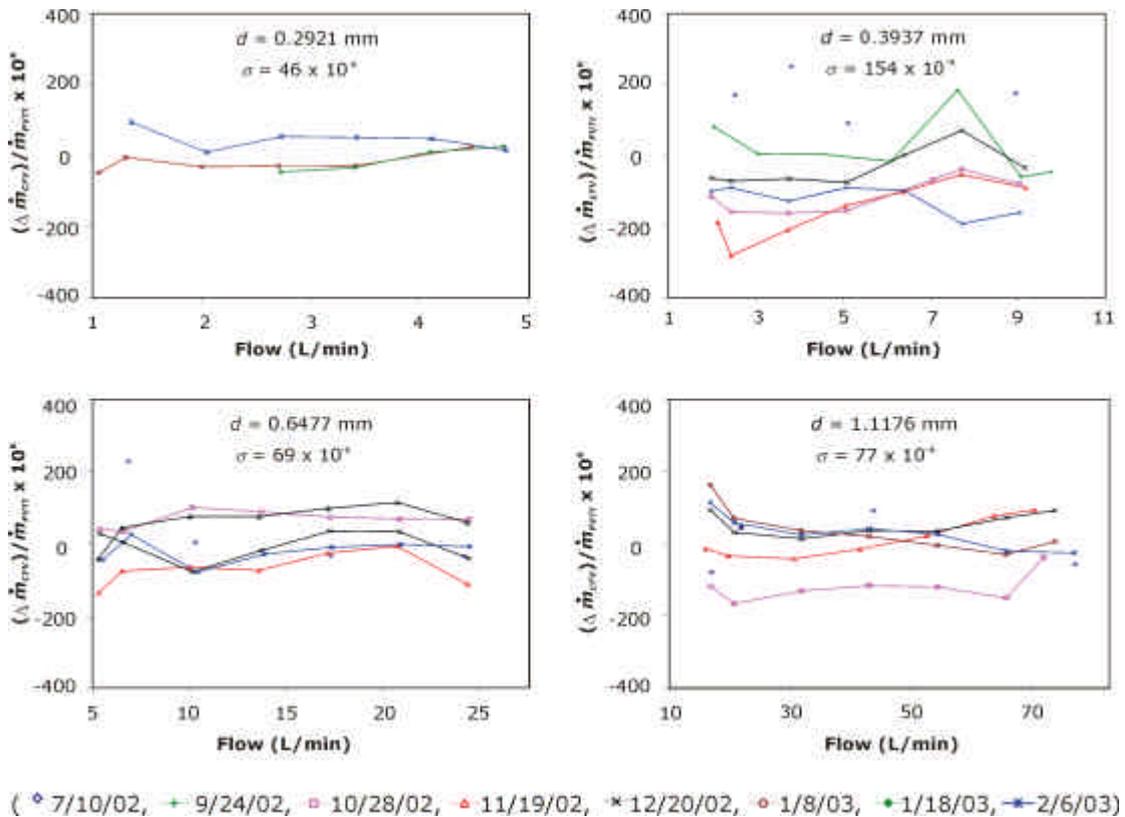


Figure 12. Change in calibration for four CFV's calibrated repeatedly between 7/02 and 2/03.

Some of the CFV's considered in Fig. 12 were used as working standards to perform periodic calibrations on four Molblocs and their mated Molboxes (two 1E4's and two 3E4's) over a six month period. The results for two of the Molbloc systems are plotted in Fig. 13. A single CFV (0.3937 mm) was used to calibrate the 1E4 Molbloc between 2 L/min and 10 L/min while two CFV's (0.3937 mm and 0.6477 mm) were used for the 3E4 to achieve flows from 2 L/min to 25 L/min. There is an overlapping flow region in the 3E4 data (2.6 L/min to 9.2 L/min) where both CFV's were used and using the Molbloc as a transfer standard between them, the agreement of the two CFV's is 0.05 % or better. Both Molblocs show a concave up error curve, but the data is within the manufacturer's specifications (1E4:  $\pm 0.2$  % of reading,  $\pm 0.02$  % of

full scale under 10 % full scale, 3E4:  $\pm 0.3$  % of reading,  $\pm 0.03$  % of full scale under 10 % full scale).

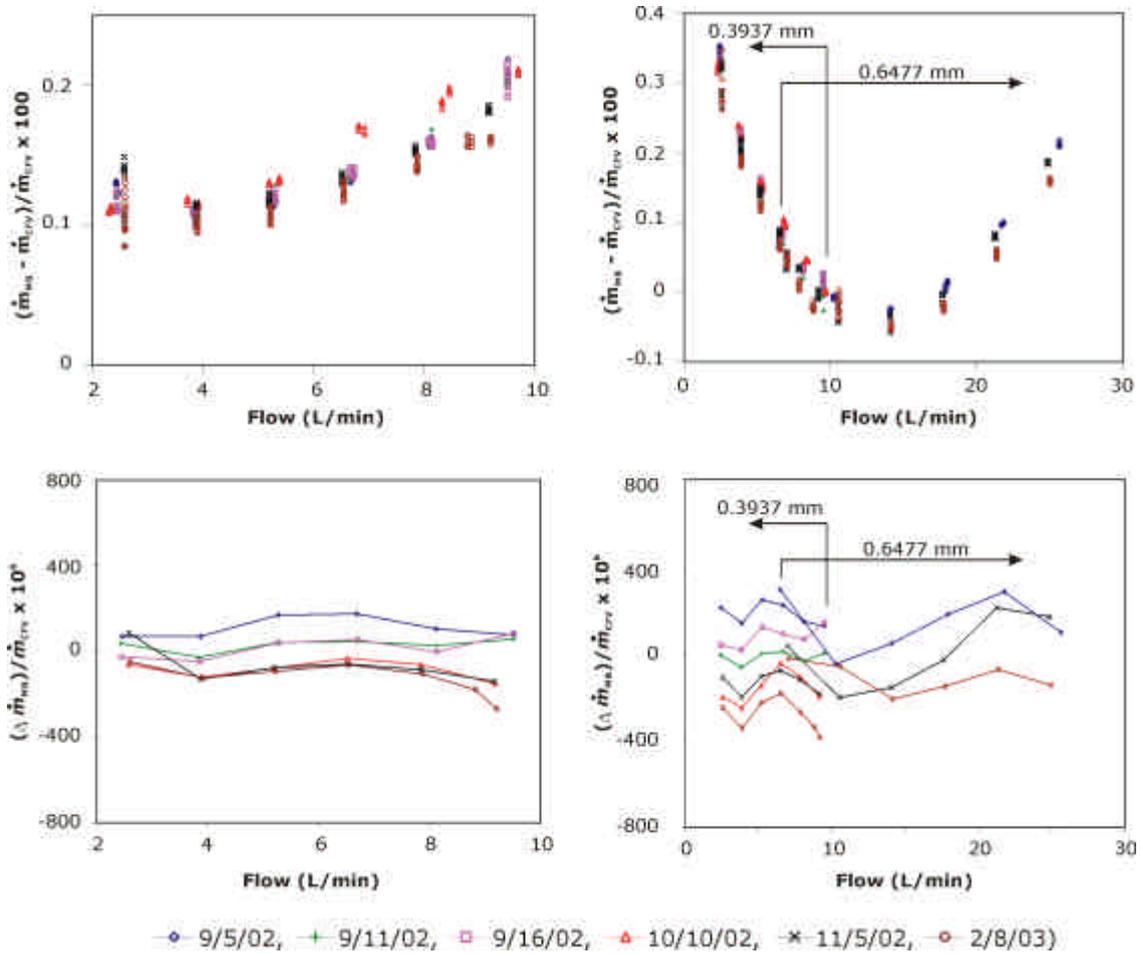


Figure 13. Calibration data for a 1E4 Molbloc and a 3E4 Molbloc. Top shows the difference between the manufacturer's calculated flow and the reference CFV flow. Bottom shows the changes in calibration over time.

The lower two plots of Fig. 13 show the changes in Molbloc calibration over the 6 month test relative to a best fit calibration through all of the data (the same approach as used above for the CFV's). Both Molblocs show good stability: the standard deviations of the change in calibration plots are  $110 \times 10^{-6} \dot{m}$  (1E4) and  $190 \times 10^{-6} \dot{m}$  (3E4). Both Molblocs show a trend of decreasing flow over time as compared to the reference CFV. Over the 6 month test, the Model 1E4 Molbloc calibration decreased by about  $200 \times 10^{-6} \dot{m}$  and the 3E4 Molbloc decreased by about  $450 \times 10^{-6} \dot{m}$ . Again, these calibration stabilities are within the manufacturer's specifications. We have not yet made the necessary calibration checks of the Molbloc *P* and *T* sensors that would allow us to state what is responsible for these calibration changes. The calibration changes are not due to the reference flow since the CFV calibration data (Fig. 12) showed no similar trend over time.

### Conclusions

The capabilities of potential participants in the low pressure gas flow key comparison (CCM.FF-K6) are summarized in Figs. 1 and 2. Bell provers (23 of 27 labs) and piston provers (19 of 27 labs) are the most common types of primary standards and they typically have uncertainties  $< 0.2\%$ . Seven laboratories have primary standards with uncertainty statements  $< 0.08\%$ .

The desired characteristics of the transfer standard (in approximate order of importance) are: calibration stability  $< 0.05\%$ , a well developed physical model, robustness, compatibility with primary standards, self-diagnostic capabilities (redundancy), ease of use, short time to stability, and facility diagnostic capabilities.

Our literature survey shows that CFV's and (to a lesser extent) LFE's are the most commonly selected flowmeter types for transfer standards. In the comparisons surveyed, the agreement between laboratories varied between  $0.05\%$  and  $0.3\%$ , and the calibration stability of the transfer standards ranged between  $0.02\%$  and  $0.15\%$ . LFE's are more appropriate for small flows, CFV's for large flows, with a typical break point of 1 L/min, but there is a wide range where either flowmeter works well.

The temperature sensitivities of the CFV and LFE have been considered analytically (i.e. through their flow equations). The CFV has smaller first order, gas property, and thermal expansion temperature sensitivities. Examples of both flowmeter types have been examined via two temperature sensitivity experiments. All of the flowmeters showed  $< 100 \times 10^{-6} \dot{m} / K$  sensitivity in the first test permutation (gas T = meter T = environmental T). The second temperature test permutation demonstrated that the details of the particular flowmeter design (not the flowmeter type) are most important. In this test, where gas T  $<$  meter T  $<$  environmental T, the meter which considered heat exchange in its design (the Molbloc) showed sensitivity of  $100 \times 10^{-6} \dot{m} / K$  or less while the others showed  $700 \times 10^{-6} \dot{m} / K$  or more. Therefore heat exchange issues must be considered in a transfer standard design intended to attain  $0.05\%$  performance.

The effects of gas composition are well understood for both the LFE and the CFV, however the known influences of vibrational non-equilibrium and thermal boundary layer effects are not incorporated into the commonly used CFV flow model. For the LFE, data gathered with five gases collapse to a single non-dimensional curve within the uncertainty of the gas viscosity data. Fortunately, if the dew point temperature of the dry air to be used in the KC is maintained below 250 K, composition effects will be  $< 125 \times 10^{-6} \dot{m} / K$  for both flowmeter types.

Calibration data from four CFV's showed calibration stability of better than  $200 \times 10^{-6} \dot{m}$  over an eight-month period. Over six months of periodic calibrations, one Molbloc showed a downward drift in calibration of  $200 \times 10^{-6} \dot{m}$ , and a second one fell  $450 \times 10^{-6} \dot{m}$ .

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