A Survey of Reference Standards for Gas Flow Calibrations

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Introduction

- Describe gas flow standards that are used as references to calibrate flow meters, to produce a *meter calibration factor*.
- Uncertainty examples.
- Can be classified as 1) primary standards or 2) working standards
- *Primary gas flow standards* are traceable to the international system of units (SI) through length, mass, time, pressure, or temperature and have the lowest available uncertainty. They are not calibrated versus other flow references.
- *Volumetric* or *gravimetric*, *static* or *dynamic*.
- A *working standard* flow reference is a flow meter with demonstrated long-term calibration stability used to calibrate other flow meters.
- Filling a tank with pressurized gas is dangerous! Relief valves, periodic inspection, overheating, limited fill / empty cycles.

List of Variables

m	Mass [kg]
'n	Mass flow [kg/s]
Р	Absolute pressure [kPa]
Т	Absolute temperature [K]
V _C	Volume of collection tank [m ³]
VI	Inventory (connecting) volume [m ³]
ρ	Density [kg/m ³]
t	Time [s]

Volumetric Flow

 $Q = u_{avg}A$

 u_{avg} – average fluid velocity A – cross sectional area



SI unit: *m³/s*, *m³/min*, *m³/hr* Other units: liters/min, cm³/min,...

NOT conserved!





Dopheide, Strunck, and Krey, *Three-Component Laser Doppler Anemometer for Gas Flowrate Measurements up to* 5500 m³/h, Metrologia, 30, pp. 453–469, 1994.

Mass Flow

$$\dot{m} = \frac{\mathrm{d}m}{\mathrm{d}t} = \rho u_{\mathrm{avg}} A = \rho Q$$

 $u_{\rm avg}$ – average fluid velocity A – cross sectional area ho – fluid density



SI Unit for Mass Flow: kg/s, kg/min, kg/hr, g/s, g/min, g/hr

Conserved!



Density of Gases



Equation of State

$$\rho = \frac{P\mathcal{M}}{ZR_{\rm u}T}$$

- $R_{\rm u} = 8.314462 \, \text{J mol}^{-1} \, \text{K}^{-1}$ (Universal gas constant)
- \mathcal{M} = Molar Mass
- Pure gas: *M* determined using reliable reference
 - Gas Mixture: $\mathcal{M} = x_1 \mathcal{M}_1 + x_2 \mathcal{M}_2 + \cdots + x_N \mathcal{M}_N$
- T Temperature in absolute units Kelvin (K)
- T(K) = T(°C) + 273.15

Density of Gases

Equation of State

$$\rho = \frac{P\mathcal{M}}{ZR_{\rm u}T}$$

 $\succ P$ - Pressure

- Measured with absolute pressure transducer
- Gauge pressure measurement & barometer
- $> Z = Z(T, P, x_k)$ is the compressibility factor
 - Requires that composition is known
 - Equal to unity for an *ideal* gas (*i.e.*, pressure approaching vacuum conditions)



REFPROP (https://www.nist.gov/srd/refprop)

- REFPROP is an acronym for REFerence fluid PROPerties
- Based on the most accurate pure fluid and mixture models currently available
 - Maintained by NIST (Eric W. Lemmon)
 - Continuously updated (next version is being developed)
 - More than 50 pure fluids
 - > Flexibility to create your own mixture (e.g., wet air, natural gas)
- REFPROP Platform and Interface Capabilities
 - Stand alone graphical user interface (GUI)
 - Compatible with Excel, Fortran, Visual Basic, C++, MatLab, LabVIEW
- Computes over 75 Thermodynamic properties (gas, liquid, and two phase)
 Density, Specific Heat, Enthalpy, Compressibility Factor, etc.

Calculating Fluid Density Using REFPROP



	В	С	D	E	F	G
0		Independent				
3	Gas	Variables	Units	T	P	ρ
4	[]	[TP]	[]	[K]	[kPa]	[kg/m ³]
5	Nitrogen	TP	mks	=25+273.15	500	=Density(B5,C5,D5,E5,F5)

Calibrating a Flow Meter

- Source of Flow (e.g., compressor/pump or vacuum pump)
- Flow meter to be calibrated or Meter Under Test (MUT)
- Connecting Piping (e.g., flow conditioners, straight piping)
- Reference Standard (RS) (e.g., Primary standard, working standard)
- Auxiliary Equipment (e.g., Freq. Counters, Data Acq., Temp. Sensors)



- Goal: to relate the known RS flow to the unknown MUT flow
- Question: Under what conditions does $\dot{m}_{MUT} = \dot{m}_{RS}$?





- General Conservation of Mass $\sum \dot{m}_{in} - \sum \dot{m}_{out} = \frac{dM_{cv}}{dt}$
- Applied to flow measurement mass balance $\dot{m}_{MUT} = \dot{m}_{RS} + \dot{m}_{Leak} + \frac{dM_{cv}}{dt} \leftarrow \text{Instantaneous mass flows}$
- Time averaged over measurement interval of Δt

$$\tilde{\tilde{m}}_{MUT} = \tilde{\tilde{m}}_{RS} + \tilde{\tilde{m}}_{Leak} + \frac{M_{CV}^{f} - M_{CV}^{i}}{\Delta t}$$
Line Pack or

$$\tilde{\tilde{m}}_{MUT} = \tilde{\tilde{m}}_{RS} + \tilde{\tilde{m}}_{Leak} + \frac{\Delta \rho_{CV} V_{CV}^{f}}{\Delta t} + \frac{\Delta V_{CV} \rho_{CV}^{i}}{\Delta t}$$
Mass Storage



1) Leaks

- 2) Density change in the connecting volume between start and stop of measurement
 - a) Generally caused by temperature changes
 - b) Could be caused by pressure or composition changes
 - c) More significant as V_{cv} increases
- 3) Volume changes in connecting volume due to pressure or temperature

Pressure Decay Leak Test



Pressure Decay Leak Test

The connecting volume between a meter under test and a flow reference was pressurized with nitrogen gas and isolated using valves at the inlet and outlet of the volume. Pressure and temperature sensors installed in the connecting volume were used to produce a time trace shown here. The pressure and temperature at 10 minutes were 403.440 kPa and 297.042 K respectively. The pressure and temperature at 30 minutes were 403.237 kPa and 296.898 K respectively. The isolated volume was estimated to be 250 cm³ (2.5 x 10⁻⁴ m³). What is the leak rate? Assume that the ideal gas law applies for density calculations.



1. Calculate the density of the gas in the isolated volume at 10 min $\rho_i(P_i, T_i)$ and 30 min $\rho_f(P_f, T_f)$.

$$\rho_{\rm i}(P_{\rm i},T_{\rm i}) = \frac{P_{\rm i}\mathcal{M}}{\mathcal{R}T_{\rm i}} = \frac{403440\,\text{Pa} \cdot 0.0280135\,\text{kg/mol}}{8.314462\,\frac{\text{J}}{\text{mol}\cdot\text{K}}\cdot297.042\,\text{K}} = 4.57609\,\text{kg/m}^3 \,,$$

$$\rho_{\rm f}(P_{\rm f},T_{\rm f}) = \frac{P_{\rm f}\mathcal{M}}{\mathcal{R}T_{\rm f}} = \frac{403237\,\text{Pa} \cdot 0.0280135\,\text{kg/mol}}{8.314462\,\frac{\text{J}}{\text{mol}\cdot\text{K}}\cdot296.898\,\text{K}} = 4.57600\,\text{kg/m}^3 \,.$$

2. Calculate the leak flow.

$$\dot{m}_{\text{leak}} = V_{\text{c}} \frac{\rho_{\text{f}}(P_{\text{f}},T_{\text{f}}) - \rho_{\text{i}}(P_{\text{i}},T_{\text{i}})}{t_{\text{f}} - t_{\text{i}}} = 2.5 \times 10^{-4} \text{m}^3 \frac{4.57600 \text{ kg/m}^3 - 4.57609 \text{ kg/m}^3}{30 \text{ min} - 10 \text{ min}},$$

$$\dot{m}_{\text{leak}} = 1.125 \times 10^{-4} \text{ kg/min}.$$

References

- Roger C. Baker, *Flow measurement handbook: Industrial designs, operating principles, performance, and applications*, Cambridge University Press, 2000
- D. W. Spitzer, editor, *Flow Measurement: Practical Guides for Measurement and Control*, 2nd edition, ISA, 2001
- International Vocabulary of Basic and General Terms in Metrology, International Standards Organization, 1993

Primary Gas Flow Standards



Bucket (a) and stop-watch (b) methods for measuring the volumetric flow of *left*: liquid and *right*: gas to calibrate a flow meter under test (c). An inverted graduated cylinder (d) and a trough filled with liquid (e) form a collection tank for the gas flow.

Primary Gas Flow Standard Methods



Bubble Meter



- Soap solution reservoir at the bottom of a bubble meter burette
- Water vapor from soap solution
- < 10 % uncertainty

Barr, G. *Two Designs of Flowmeter and a Method of Calibration*, J. Scient. Instrum., 11 (10) pp. 321 to 324, 1934.

For uncertainty of < 0.1 %, one must consider:

- maintaining stable conditions (pressure, temperature, and flow),
- correcting buoyancy effects on mass,
- using low uncertainty instrumentation,
- a well-designed flow *diverter* to switch flow on and off,
- density changes in connecting volumes.

Inventory Volume, Connecting Volume, Storage Effects



A generic primary gas flow standard with: (a) meter under test (MUT), (b) a pressure regulator or critical nozzle to set the upstream boundary of the inventory volume (c, shaded region), and (d) a collection tank with volume $V_{\rm C}$. The small open circle represents the regulator control valve used to maintain constant pressure at the outlet of the MUT if the MUT is not a critical nozzle.

Constant Pressure (or Density)



Piston Provers (8000 slm to 0.01 sccm)



Wright, J. D. and Mattingly, G. E., *NIST Calibration Services for Gas Flow Meters: Piston Prover and Bell Prover Gas Flow Facilities*, NIST Special Publication 250-49, August 1998.

Mercury-Sealed Piston Prover



Model 1050 mercury sealed piston provers manufactured by George K. Porter Inc. / Books Instrument Division that were used as national standards at NIST from 1962 to 2003.

"Clearance-Sealed" Piston Prover (10 µm)



Damped pressure fluctuations caused by closing the bypass valve on a clearancesealed piston prover. The start sensor should be located high enough on the cylinder that these fluctuations decay.

Padden, H., Uncertainty Analysis of a High-Speed Dry Piston Flow Prover, International Symposium on Fluid Flow Measurement, Anchorage, Alaska, USA, 2002.
Kutin, J., Bobovnik, G., and Bajsic, I., Dynamic Effects in a Clearance-Sealed Piston Prover for Gas Flow Measurements, Metrologia, 48, pp. 123 to 132, 2011.

Uncertainty Analysis Review

Consider a process that has an output measurand y, based on N input quantities x_i with the generic basis equation:

 $y = y(x_1, x_2, \dots, x_N).$

For uncorrelated uncertainty components, standard uncertainties are combined by root-sum-of-squares (RSS) to calculate the uncertainty of the measurand u(y):

$$u(y) = \sqrt{\sum_{i=1}^{N} \left(\frac{\partial y}{\partial x_i}\right)^2 u^2(x_i)},$$

- $u(x_i)$ are the standard uncertainties for each input quantity,
- $\frac{\partial y}{\partial x_i}$ are the sensitivity coefficients.

Uncertainty Analysis Review

It is convenient to use 1) relative (fractional or percentage) uncertainties $u(x_i)/x_i$ rather than absolute or dimensional uncertainties $u(x_i)$ and 2) normalized sensitivity coefficients S_i :

$$S_i = \frac{x_i}{y} \frac{\partial y}{\partial x_i},$$

instead of $\partial y / \partial x_i$ during uncertainty calculations.

$$\frac{u(y)}{y} = \sqrt{\sum_{i=1}^{N} S_i^2 \left(\frac{u(x_i)}{x_i}\right)^2}$$

For linear basis equations, the normalized sensitivity coefficients are the exponent of the input variables x_i and can be evaluated by inspection.

Uncertainty Example: Volume of a Cylinder

$$y = y(x_1, x_2, \dots, x_N)$$
$$V = h \pi r^2$$

$$S_{i} = \frac{x_{i}}{y} \frac{\partial y}{\partial x_{i}}$$
$$S_{h} = 1, \ S_{r} = 2$$

Г



$$\frac{u(h)}{h} = 1$$
 %, $\frac{u(r)}{r} = 1$ %

$$\frac{u(y)}{y} = \sqrt{\sum_{i=1}^{N} S_i^2 \left(\frac{u(x_i)}{x_i}\right)^2}$$
$$\frac{u(V)}{V} = \sqrt{S_h^2 \left(\frac{u(h)}{h}\right)^2 + S_r^2 \left(\frac{u(r)}{r}\right)^2} = \sqrt{1^2(1^2) + 2^2(1^2)} = \sqrt{5} = 2.24\%$$

Large Components Dominate RSS

$$\sqrt{(2^2) + (1^2)} = \sqrt{5} = 2.24 \%$$

$$\sqrt{(3^2) + (1^2)} = \sqrt{10} = 3.16\%$$

$$\sqrt{(4^2) + (1^2)} = \sqrt{17} = 4.12\%$$

Correlated Uncertainties: NOT RSS

$$(y) = \sqrt{\sum_{i=1}^{N} \left(\frac{\partial y}{\partial x_i}\right)^2 u^2(x_i) + 2\sum_{i=1}^{N-1} \sum_{j=i+1}^{N} \frac{\partial y}{\partial x_i} \frac{\partial y}{\partial x_j} u(x_i) u(x_j) r(x_i, x_j)}$$

References

- Evaluation of Measurement Data An Introduction to the "Guide to the Expression of Uncertainty in Measurement" and Related Documents, Joint Committee for Guides in Metrology, (JCGM) 104: 2009, <u>https://www.bipm.org/utils/common/documents/jcgm/JCGM_104_2009_E.pdf</u>.
- Guide to the Expression of Uncertainty in Measurement, JCGM 100: 2008, <u>https://www.bipm.org/utils/common/documents/jcgm/JCGM 100 2008 E.pdf</u>.
- Evaluation of Measurement Data –Supplement 1 to the "Guide to the Expression of Uncertainty in Measurement"-Propagation of distributions using a Monte Carlo Method, JCGM 101: 2008, https://www.bipm.org/utils/common/documents/jcgm/JCGM_101_2008_E.pdf.
- Coleman, H. W. and Steele, W. G., *Experimentation and Uncertainty Analysis for Engineers*, John Wiley and Sons, 2nd edition, 1999.

Uncertainty of the 4.44 cm diameter piston prover for 1 L/min.

Input Variable (x_i)	Notes	x_i Value	$u(x_i)/x_i$ [%]	S _i [-]	Contrib. [%]
Cylinder volume, V _C	а	0.71 L	0.016	1	6
Gas density, $\rho_{\rm C,f}$	b	1.14 g/L	0.053	1	68
Collection time, $(t_{\rm f} - t_{\rm i})$	с	38 s	0.009	-1	2
Inventory mass change, $V_{\rm I}(ho_{\rm I,f}- ho_{\rm I,i})$	d	$2.8 \times 10^{-5} \text{ g}$	0.007	0.5	~ 0
Leaks	e	0.01 sccm	0.010	1	2
Type A (s)	f	0.03 sccm	0.030	1	22

Expanded uncertainty (95 %) $U(\dot{m})/\dot{m} = 0.13$ %

- a) Based on 25 evenly spaced internal diameter measurements, start to stop sensor length, piston rocking, thermal expansion caused by environmental temperature changes.
- b) Includes pressure, temperature calibration, temperature errors due to spatial nonuniformity, equation of state, gas composition.
- c) Timer calibration, timer actuation.
- d) From inventory volume and density uncertainty.
- e) Measured via a floating piston as described in text.
- f) Based on calibration data from laminar flow meters.



O-Ring-Sealed Piston Prover



O-ring sealed 1200 L piston prover at IMGC Italy. Gas from volume (a) is expelled through outlet (b) by descending piston (c) that is sealed at (d). Piston position is measured with laser interferometer (g).

Bellinga, H. and Delhez, F. J., *Experience with a High-Capacity Piston Prover as a Primary Standard for High-Pressure Gas Flow Measurement*, Flow Meas. and Instrum., Vol. 4, No. 2, pp. 85-89, April, 1993.

O-Ring-Sealed Piston Prover



A piston prover with O-ring seals for flows as low as 0.01 sccm.

Berg, R. F., Gooding, T., and Vest, R. E., *Constant Pressure Primary Flow Standard for Gas Flows from 0.01 cm3 min-1 to 100 cm3 min-1 (0.007–74 \mumol s-1)*, Flow Meas. Instrum. **35** 84–91, 2014.



Schematic of a bell prover from Burick, T. E., *Automatic Prover Controls*, Proceedings of the 22nd Annual Appalachian Gas Measurement Short Course, WVU Bulletin, Series 65, No. 8-1, pp. 95 to 99, February, 1965.

One of three bell provers used as national standards at NIST until 2003.



Pressure and velocity time traces for a 48 L bell prover during a collection at a flow of 90 L/min. Note the initial damped oscillation of the bell velocity due to the change in pressure when the bypass valve is closed and the resulting oil level fluctuations. The increase in bell pressure during the collection is caused by imperfect counterbalancing of buoyancy effects on the bell's weight as it rises out of the sealing liquid.

Uncertainty of the 140 L bell prover for 100 L/min.

Input Variable (x_i)	Notes	x_i Value	$u(x_i)/x_i$ [%]	S _i [-]	Contrib. [%]
Tank volume, V _C	a	114 L	0.059	1	48
Gas density, $\rho_{C,f}$	b	1.14 g/L	0.045	1	28
Collection time, $(t_{\rm f} - t_{\rm i})$	с	68.3 s	0.009	-1	1
Inventory mass change, $V_{\rm I}(ho_{\rm I,f}- ho_{\rm I,i})$	d	$1.3 \times 10^{-3} \text{ g}$	0.011	0.09	~ 0
Leaks	e	0.01 slm	0.010	1	1
Type A (s)	\mathbf{f}	0.04 slm	0.040	1	22
Expanded uncertainty (95 %)		$U(\dot{m})/\dot{m} = 0.17$	%		

- a) Based on outside diameter (strapping) and wall thickness measurements, start to stop sensor displacement, tilting (rocking) of the bell, sealing liquid film drainage and thickness, thermal expansion caused by environmental temperature changes.
- b) Includes spatial non-uniformity of temperature, pressure and temperature calibration, equation of state, gas composition.
- c) Timer calibration and actuation.
- d) From inventory volume and density uncertainty.
- e) Based on leak tests performed by raising the bell under zero flow conditions.
- f) Based on calibration data from critical nozzles meters.

Constant Volume


Pressure-Volume-Temperature-time (PVTt)



Wright, J. D., Moldover, M. R., Johnson, A. N., and Mizuno, A., *Volumetric Gas Flow Standard with Uncertainty of 0.02 % to 0.05 %*, ASME J. of Fluids Engineering, **125**, 1058-1066, 2003.



The 13 m³ *PVTt* system of NMIJ/AIST is surrounded by a recirculating, temperature-controlled water jacket.

Ishibashi, M., Takamoto, M., and Watanabe, N., *New System for the Pressurized Gas Flow Standard in Japan*, Proceedings of International Symposium on Fluid Flow Measurement, 1990.



A photograph of the NIST 34 L and 677 L *PVTt* collection tanks submerged in a temperature-controlled water bath stable and uniform to ± 2 mK.



The equilibration of pressure and temperature immediately following a filling of the NIST 34 L *PVTt* standard at 25 L/min.

Determining Tank Volume

gas gravimetric method:

$$V_{\rm C,grav} = \frac{m_{\rm f} - m_{\rm i}}{\left(\rho_{\rm C,f} - \rho_{\rm C,i}\right)} - V_{\rm extra}$$

gas expansion method:
$$V_{C,exp} = \frac{(\rho_{1,f} - \rho_{1,i})V_1}{(\rho_{C,f} - \rho_{C,i})} - V_{extra}$$

Wright, J. D., Johnson, A. N., and Moldover, M. R., *Design and Uncertainty Analysis for a PVTt Gas Flow Standard*, J. Res. Natl. Inst. Stand. Technol., **108**, 21–47, 2003.



Uncertainty of mass flow measurements from the NIST 34 L and 677 L *PVTt* standards plotted versus flow. A circle marks the case detailed in the example *PVTt* uncertainty analysis.

Uncertainty of the NIST 677 L PVTt flow standard at 1000 slm.

Input Variable (x_i)	Notes	x_i Value	$\begin{array}{c} u(x_i)/x_i \\ [\%] \end{array}$	S _i [-]	Contrib. [%]
Tank volume, $V_{\rm c}$	a	677.92 L	0.0041	1	26
Initial gas density, $\rho_{\rm C,i}$	b	$2.27 \times 10^{-4} \text{ g/L}$	5.0	-0.0002	2
Final gas density, $\rho_{\rm C,f}$	b	1.14 g/L	0.0037	1	22
Inventory mass change	с	1.3	3.2	0.0017	44
Collection time, $(t_{\rm f}-t_{\rm i})$	d	40 s	0.0034	-1	0
Leaks	e	8×10^{-6} slm	~ 0	1	0
Type A (s)		0.04 slm	0.0020	1	6

Expanded uncertainty (95 %) $U(\dot{m})/\dot{m} = 0.016$ %

- a) Measured by gas gravimetric method. Largest component is full tank density due to pressure uncertainty.
- b) Pressure, temperature, equation of state, and gas composition.
- c) Used estimated uncertainties, verified by comparison and multiple diversions.
- d) 3000 Hz data acquisition rate, rectangular distribution, applied for both start and stop times.
- e) Periodically measured by pressure decay test.

Rate of Rise (RoR)



Wright, J. D., Johnson, A. N., Moldover, M. R., and Kline, G. M., *Errors in Rate-of-Rise Gas Flow Measurement from Flow Work*, Proceedings of the 10th International Symposium on Fluid Flow Measurement, Queretaro, Mexico, March 21 to 23, 2018.



Example RoR flow calculations from a least squares regression, and for different moving averages (MA = 1, 10, 100). The filtered data allows one to check data for flow stability before applying least squares regression and allows monitoring the flow as data is collected.

Flow Work or Heat of Compression



Pressure in the 34 L tank versus time at the end of a collection for an air flow of 5 slm. The gas is initially warm (and therefore at higher pressure) due to flow work during filling.

Wright, J. D. and Johnson, A. N., *Uncertainty in Primary Gas Flow Standards Due to Flow Work Phenomena*, FLOMEKO, Salvador, Brazil 2000.

Errors Due to Flow Work



$$T_{\rm err} = T - T_{\rm H_2O} = T_{\rm H_2O} \left[\frac{(T_{\rm in}/T_{\rm H_2O})\gamma - 1}{1 + \Gamma} \right] \left[1 - \left(\frac{\dot{mt}}{m_{\rm i}} + 1\right)^{-(1+\Gamma)} \right]$$

$$\Gamma \frac{\left(T - T_{\rm H_2O}\right)}{T} = \left[\frac{hA}{c_V \dot{m}}\right] \left[\frac{\left(T - T_{\rm H_2O}\right)}{T}\right]$$



Uncertainty (k = 2, approximately 95 % confidence level) versus flow for the NIST 34 L rate of rise flow standard. A circle marks the case detailed in the example RoR uncertainty analysis.

Uncertainty of the NIST 34 L Rate of Rise flow standard at 100 sccm

Input Variable (x_i)	Notes	x_i Value	$u(x_i)/x_i$ [%]	S _i [-]	Contrib. [%]
Tank volume, $V_{\rm C} + V_{\rm I}$	a	34.13 L	0.028	1	39
Gas density change, $\Delta \rho_{C}$	b	0.352 g/L	0.013	1	48
Slope calculation and flow stability	с	~ 0	~ 0	1	0
Collection time $(t_{\rm f} - t_{\rm i})$	d	5588 s	~ 0	-1	0
Leaks	e	5.0×10^{-4} sccm	~ 0	1	0
Type A (s)	f	8.2×10^{-3} sccm	0.008	1	13
Expanded uncertainty (95 %)		$U(\dot{m})/\dot{m}=0.00$	55 %		

- a) Measured by gas gravimetric method. Largest component is full tank density due to pressure uncertainty.
- b) Includes pressure, temperature calibration, temperature errors due to flow work, resolution, equation of state, gas composition.
- c) Data is prequalified for stability, N is large.
- d) Collection time must be long enough to ensure low uncertainty pressure change.
- e) Periodically measured by pressure decay test as a function of tank pressure. Leaks are significant at lower flows, see Fig. 21.
- f) Based on calibration data from laminar flow meters.

Gravimetric



Static Gravimetric Method with Flying Start/Stop



$$\dot{m}_{\rm SG} = \frac{\left(m_{\rm C,f} - m_{\rm C,i}\right) + V_{\rm I1}\left(\rho_{\rm I1,f} - \rho_{\rm I1,i}\right) + V_{\rm I2}\left(\rho_{\rm I2,f} - \rho_{\rm I2,i}\right)}{t_{\rm f} - t_{\rm i}}$$

Park, J. T., Behring, K. A., and Grimley, T. A., *Uncertainty Estimates for the Gravimetric Primary Flow Standards of the MRF*, International Symposium of Fluid Flow Measurement, San Antonio, USA, 1995.

Mass with Buoyancy Corrections

$$m = m_{\rm A} + \rho_{\rm air} V_{\rm C} = \frac{m_{\rm A}}{\left(1 - \frac{\rho_{\rm air}}{\rho_{\rm C}}\right)} \approx m_{\rm A} \left(1 + \frac{\rho_{\rm air}}{\rho_{\rm C}}\right)$$

Typical density values for the air and the weighed tank are 0.001 g/cm³ and 1 g/cm³ respectively, so buoyancy corrections are approximately 0.1 %



Cylinder volume depends on the pressure and temperature:

$$V_{\rm C} = V_{\rm C,ref} [1 + \lambda (P - P_{\rm ref})] [1 + 3\alpha (T - T_{\rm ref})]$$

 $\lambda = 1.59 \times 10^{-10}$ 1/Pa, $\alpha = 2.4 \times 10^{-5}$ 1/K, negligible for most applications.



A picture of the test section and diverter unit of the static gravimetric system of NMIJ/AIST.



The balances of the static gravimetric system of NMIJ/AIST.

Nakao, S. and Takamoto, M., *Development of the Calibration Facility for Small Mass Flow Rate of Gases and the Sonic Venturi Nozzle Transfer Standard*, JSME International Journal, Series B, vol.42, no.4, pp.667-673, 1999.

Uncertainty of the NMIJ/AIST flying start/stop static gravimetric flow standard

Input Variable (x_i)	Notes	x_i Value	$u(x_i)/x_i$ [%]	S _i [-]	Contrib. [%]
Tank mass change $(m_{C,f} - m_{C,i})$	a	1.8 g	0.028	1	28
Inventory mass change	b	~0 g	0.020	1	14
Collection time $(t_{\rm f} - t_{\rm i})$	с	30 s	0.000	-1	~ 0
Leaks	d	0.013 Pa/min	0.000	1	~ 0
Type A (s)	e	~0	0.040	1	58

Expanded uncertainty (95 %) $U(\dot{m})/\dot{m} = 0.105 \%$

- a) Includes balance resolution, buoyancy corrections via reference tank.
- b) Includes density and inventory volume components.
- c) 14 ms out of 30 s minimum collection.
- d) Measured by pressure decay test.
- e) Measured during calibration of best existing device. For this and other uncertainty examples in this chapter, the sample standard deviation *s* is used instead of the standard deviation of the mean s/\sqrt{n} because the uncertainty is for a single measurement made with the reference flow standard.

Static Gravimetric Method with Standing Start/Stop





A picture of a standing start/stop static gravimetric standard at NIST being used to calibrate a laminar flow meter. Flow is from right to left.

Integration Errors

$$m_{\text{MUT}} = \int_{t_{i}}^{t_{f}} \dot{m}_{\text{MUT}} dt = \sum_{i=1}^{N} \frac{\dot{m}_{\text{MUT},i-1} - \dot{m}_{\text{MUT},i}}{2} \Delta t$$



A time-trace of the mass flow indicated by a laminar flow meter under test. The nominal flow is 4.3×10^{-5} g/s (2 sccm).

Uncertainty of the NIST static, standing start/stop gravimetric flow standard

Input Variable (x_i)	Notes	x_i Value	$u(x_i)/x_i$ [%]	S _i [-]	Contrib. [%]
Tank mass change $\left(m_{ extsf{C,f}}-m_{ extsf{C,i}} ight)$	a	9.4 g	0.048	1	31
Inventory mass change	b	0.001 g	0.010	1	1
Integration errors	с	0.006 g	0.064	1	55
Collection time $(t_{\rm f} - t_{\rm i})$	d	235620 s	0.004	-1	~ 0
Leaks	e	1 Pa/min	0	1	~ 0
Type A (s/\sqrt{N})	f	~0	0.03	1	12

Expanded uncertainty (95 %)

 $U(\dot{m})/\dot{m} = 0.171 \%$

- a) Includes balance resolution, reference mass uncertainty, buoyancy corrections.
- b) Based on making no inventory correction, nitrogen.
- c) Assumed a quarter of a triangular area covering the stop transient.
- d) 10 s out of 3927 min integration.
- e) Measured by pressure decay test.
- f) Measured during calibration of best existing device.

Dynamic Gravimetric Method



Barbe, J., Couette, J., Picault, J., and Marschal, A., *Traceability of Standard Gas Mixtures Prepared by the Dynamic Gravimetric Method*, Bulletin du Bureau National de Metrologie, Volume 2001-2, No. 120.

Bair, M. and Rombouts, C., *Typical Measurement Uncertainty in Gas Flow Measured by GFS2102 Gravimetric Flow Standard*, Technical Note 6050TN09, DH Instruments, May 31, 2006.





The dynamic gravimetric gas flow standard developed by Fluke Inc.

A picture of two dynamic gravimetric systems in the Laboratoire National d'Essai circa 2001.

Uncertainty of the Fluke dynamic gravimetric flow standard for a flow of 50 sccm

Input Variable (x_i)	Notes	x_i Value	$\frac{u(x_i)/x_i}{[\%]}$	<i>S</i> _i [-]	Contrib. [%]
Tank mass change $\left(m_{ extsf{C,f}}-m_{ extsf{C,i}} ight)$	a	1 g	0.055	1	75
Inventory mass change	b	$1 \times 10^{-5} \text{ g}$	0.001	1	0
Slope calculation and flow stability	с	0.006 g	0.010	1	2
Collection time $(t_{\rm f} - t_{\rm i})$	d	1000 s	0.001	-1	0
Leaks	e	1 Pa/min	0	1	0
Type A (s)	f	0.015 sccm	0.03	1	22
Expanded uncertainty (95 %)	7	$I(\dot{m})/\dot{m} = 0.128\%$			

a) Includes balance resolution, zero, linearity, buoyancy corrections.

b) Based on making no inventory correction, 0.5 K / h room temperature change, nitrogen.

c) Assumes flow is kept stable, $u(m)/u(t) \ge 2.5$, and N is large.

d) Rectangular probability distribution applied to 23 Hz sampling at start and stop.

e) Measured by pressure decay test.

f) Measured during calibration of best existing device.

Other Primary Gas Flow Standards

For large enough flows, critical flow venturis, multi-path ultrasonic flow meters,...

Working Standards for Gas Flow

- Working standards have advantages in size, cost, complexity, speed, and ease of operation compared to primary standards.
- At NIST: Laminar flow meters < 1.5 L/min, critical flow venturis for > 1.5 L/min, turbines in natural gas.
- There are many gas flow meter types used as working standards (*e.g.*, laminar, turbine, Coriolis, positive displacement), but we will focus on critical flow venturi (critical nozzle) working standards.

Wright, J. D., Kayl, J.-P., Johnson, A. N., and Kline, G. M., *Gas Flowmeter Calibrations with the Working Gas Flow Standard*, NIST Special Publication 250-80, National Institute of Standards and Technology, Gaithersburg, MD, January 25, 2008.

Critical Flow Venturi (Nozzle)

Measurement Principle

- Accelerate gas to sonic velocity at the throat of a restriction
- Advantages
 - Simple, no moving parts to wear
 - Well understood physical model
 - Highly reproducible, stable calibration
 - Excellent working standards and transfer standards

Disadvantages

- Significant pressure drop
- Moderate turn-down ratio
- Flows > 1.5 L/min
- Rarely applied to liquid flows

Characteristics

- Uncertainty: ±0.1% of reading
- Rangeability: 5:1 (for 700 kPa source pressure, 100 kPa outlet)







Photo courtesy of Flow Systems, Inc.



CFV Principle of Operation





Baseline Mass Flow Model

- Solution of the Navier-Stokes Equations
- Choked Flow Conditions (*M*=1 @ CFV throat)



We can account for real gas effects by using a properties database (REFPROP) to calculate C_R^* instead of C_i^*

$$\dot{m}_{\rm th} = \underbrace{\frac{C_{\rm R}^* P_0 A \sqrt{\mathcal{M}}}{\sqrt{R_{\rm U} T_0}}}_{\sqrt{R_{\rm U} T_0}}$$

Calibration against a flow reference can account for 1-dimensional flow effects, isentropic flow, and throat diameter errors (or at least most of them):



Working Standards for Gas Flow

1. Calibrate CFV against a primary standard



2. Use CFV as a flow reference to calibrate other flow meters



NIST Working Gas Flow Standard (WGFS), 0.1 %, k = 2

Nozzle-to-Nozzle Test System



Carter, M., Johansen, W., Britton, C., Performance of a Gas Flow Meter Calibration System Utilizing Critical Flow Venturi Standards, FLOMEKO, Taipei, Taiwan, 2010.

Nozzles in Parallel



Multi-nozzle flow manifold of 162 nozzles with throat diameter of 7.9 mm to achieve air flows up to 18 kg/s.

Stevens, R. L., *Development and Calibration of the Boeing 18 kg/sec Airflow Calibration Transfer Standard*, International Symposium on Fluid Flow Measurement, Arlington, Virginia, USA, pp. 80–96, 1986.



A nozzle manifold used at NIST as a working gas flow standard. Most of the nozzles have throat diameter of 5.2 mm, but three have diameters of 4.8 mm, 4.5 mm, and 3.2 mm to achieve smaller flow increments.

Johnson, A. N., Li, C. H., Wright, J. D. Kline, G. M., Crowley, C. J., *Critical Flow Venturi Manifold Improves Gas Flow Calibrations*, International Symposium for Fluid Flow Measurement, Colorado Springs, Colorado, USA, 2012.
Questions?

UV-C Disinfection and Re-Use of N95 Masks



Masks placed in a UVConcepts disinfection chamber for 1 J/cm2 UV-C 254 nm exposure (10 x)

- A collaborator is using Human Coronavirus OC43 as a surrogate for COVID 19.
- "Focus Forming Assays" show 5-log inactivation from the UV-C exposure.
- Other mask tests are performed on uncontaminated masks, UV-C treated and untreated.



Flow resistance measurements for whole masks



Mask glued to 4" PVC pipe

Critical nozzle (flow reference)

Flow conditioning plate



Differential pressure taps

NIST flow resistance results for 3M Model 1860





NIOSH Testing



Fig 1A. Static Advanced Headform Fig 1B. Instron 5943 Tensile Tester Fig 1C. TSI 8130 Filter Tester

Respirator Model, Decon Method, # of cycles	Treated Sample #	Flow Rate (Lpm)	Initial Filter Resistance (mmH₂O)	Initial Percent Leakage (%)	Maximum Percent Leakage (%)	Filter Efficiency (%)
3M 1860, controls	Control 1	85	9.1	0.390	0.654	99.35
	Control 2	85	9.0	1.42	2.20	97.80
3M 1860, UV-C (1000 mJ/cm²), 10 cycles	1	85	9.4	0.762	1.03	98.97
	2	85	10.0	0.311	0.465	99.54
Min Fil Eff: 98.97%	3	85	9.4	0.326	0.529	99.47
	4	85	9.7	0.168	0.388	99.61
Max Fil Eff: 99.61%	5	85	10.0	0.944	0.944	99.06



https://www.youtube.com/watch?v=cfHPQVDy1

https://www.youtube.com/watch?v=piC