SOP 26

Standard Operating Procedure For Gravimetric Calibration of Dynamic Volumetric Systems used as Standards[1]

1 Introduction

1.1 Purpose of Test

This procedure describes the gravimetric calibration of volumetric systems that may be used as volumetric measuring standards. (Gravimetric calibration of fixed volume graduated-neck or slicker-plate type standards should be completed using SOP 14). This procedure uses gravimetric calibration principles with mass values and pure water sources to obtain calibration uncertainties that are generally less than those obtained during volume transfer procedures of similar standards. Alternative test liquids are not considered in this procedure. Accordingly, the procedure is especially useful for high accuracy calibrations. The procedure uses replicate testing to incorporate measurement assurance steps to ensure the validity of the measurement process; additional analyses must be performed to assess for bias (e.g., gravimetric calibration of other similar sized standards using the same methods, or interlaboratory comparisons with similar standards or provers). The procedure makes use of an electronic balance and is suitable for all sizes of gravimetric calibrations as limited by the capacity and resolution of the balance, available water source, mass standards, and handling capacity of the laboratory. Detailed measurement ranges, standards, equipment, and uncertainties for this SOP are generally compiled in a separate document in the laboratory.

This procedure can be used for single direction and bi-directional calibration of small volume provers, for calibration of volumes delivered through a master meter, for fixed volume delivery from alternative volumetric measuring systems, or other alternative test methods and standards where the gravimetric calibration provides a useful calibration result and measurement uncertainty. All steps in the procedure and calculations must be thoroughly reviewed and evaluated for appropriateness for the specific standards under test to ensure accurate measurement results are reported to the user.

1.2 Conformity Assessment

Standards that are calibrated for use in legal weights and measures applications should be evaluated for conformance to the appropriate specifications and tolerances that apply. Where compliance is required by law, conformity evaluations should be conducted prior to performing calibrations. See Section 6.2 for reporting results.

[1] Non-SI units are predominately in common use in State legal metrology laboratories, and/or the petroleum industry for many volumetric measurements, therefore non-SI units are used to reflect the practical needs of the laboratories performing these measurements when appropriate.

1.3 Prerequisites

1.3.1 Verify that valid calibration certificates with appropriate values and uncertainties are available for all standards, instruments and sensors used in this procedure. Calibration values must have demonstrated metrological traceability to the International System of Units (SI), which may be to the SI, through a National Metrology Institute such as NIST.

1.3.2 Verify that all standards and instruments (e.g., mass standards, thermometer, barometer, and hygrometer) have sufficiently small standard uncertainties for the level of calibration. Reference standards that need special care and protection should not be used for gravimetric calibration where there is risk for contamination or damage from water.

1.3.3 Verify that the balance has sufficient capacity and is in good operating condition with sufficiently small process standard deviation. Process standard deviations need to be verified by a valid control chart or preliminary experiments to ascertain performance quality. The accuracy of the balance and weighing procedures should also be evaluated to minimize potential bias in the measurement process.

1.3.4 Verify that the operator is experienced and has demonstrated proficiency in precision weighing techniques and volumetric measurements and has had specific training in NISTIR 6969 SOP 2, SOP 4, and NISTIR 7383 SOP 19, SOP 29, GMP 10, and especially in gravimetric calibrations such as those used in SOP 14.

1.3.5 Verify that an adequate quality and supply of distilled or de-ionized water (see GLP 10) is available. Do not use tap or other impure water sources for this procedure! The laboratory must have a continuous flow of water available and may thermally equilibrate water in a large volumetric prover or a water tank provided purity is ensured. Note: Calibration of some systems may require other liquids due to viscosity or lubricity requirements and alternative procedures should be considered. Use of calibration liquids other than water is not considered in this SOP.

1.3.6 Verify that the laboratory facility is free of air currents and/or temperature fluctuations where the mass measurements will be performed. The volumetric proving system must also be calibrated in a thermally stable environment. Where the volume and weighing areas are not the same, movement of standards and transfer vessels must not negatively affect environmental stability. Stable temperatures, and thermal equilibration of water, are especially important to minimize possible convection currents on precision balances. Humidity control is essential for minimizing possible evaporation and/or condensation during the measurement process.

1.3.7 Verify that the laboratory facilities meet the following minimum conditions to meet the expected uncertainty possible with this procedure:

Table 1. Laboratory environmental conditions.

Procedure	Temperature	Relative Humidity
Gravimetric	18 °C to 23 °C Stable to ± 1 °C / 1 h during the calibration.	40 % to 60 % Stable to ± 10 % / 4 h

1.3.8 All necessary maintenance to the unknown volumetric standard must be completed by the device owner prior to starting the gravimetric calibration. It is advisable to perform a static leak test prior to performing the test on systems that allow such testing. Replace any seals that are present on the prover if there is any doubt as to their integrity. If "as found" data is required, it must be obtained before any seal replacements. A second calibration will be required after any maintenance is completed to obtain an "as left" calibration value. Note: "As found" data is essential for setting, monitoring, or adjusting calibration intervals.

1.3.9 Any standard inlet(s) and outlet(s) that are not used in the calibration process must be blocked by the device owner before calibration by using a blind flange, double-block and bleed valve, or other appropriate hardware. All extra valves, piping must be evaluated to ensure that air is not entrapped in the system.

1.3.10 The device owner must provide all necessary manufacturers' manuals and hardware (e.g., including pipe fittings, valves, solenoids, wiring, etc).. as well as trained personnel to operate the device during the calibration to ensure that the device is calibrated in the same way it will be used.

2 Methodology

2.1 Scope, Precision, Accuracy

The procedure is applicable for the calibration of any size of volumetric measuring system that, when filled with water, will not overload the electronic balance used. Typical volumetric proving systems ranging in capacity from 5 gal to 120 gal may be calibrated. Continuous systems that measure these quantities that are controllable in the laboratory may be considered as well (e.g., a metering system delivering 40 gal per min to 100 gal per min may be possible). Flow rates that will be assessed need to be determined between the laboratory and the standard owner prior to calibration and should consider the practical range of use, the laboratory capability, and any documentary or legal requirements of applicable specifications and tolerances.

The precision of this calibration depends on strict adherence to the various steps of the procedure. The accuracy attainable will depend on the uncertainties of the standard weights, the uncertainties associated with the air buoyancy corrections, the accuracy of temperature measurements, the purity and density of the water, and the thermal expansion corrections that are made. Another important factor to consider is the repeatability of the procedure and the repeatability of the standard under test.

2.2 Summary

The mass of the water delivered by the volumetric proving system is determined by weighing the water on an electronic balance and comparing it to mass standards. Volume is derived from the mass of the mass standards, the density of pure water, appropriate air buoyancy corrections, and thermal and pressure expansion corrections, using the equations provided in Section 3.

2.3 Standards, Equipment, and Calibration Notes

2.3.1 An electronic balance having sufficient capacity to weigh the loaded vessel is required. The repeatability of the balance, measurement process, and standard under test are limiting factors in the accuracy of the measurement. The resolution of the balance and repeatability of the measurement process must be smaller than the expected uncertainty of the calibration. Note: Standard deviations obtained from precision mass calibrations do not reflect the process repeatability of this procedure; therefore, repeatability must be assessed using this procedure.

2.3.2 Mass standards of sufficient quantity and capacity are required. Ordinarily, standards of ASTM Class 2 or 3 or OIML Class F_1 or F_2 weight specifications are required. Working standards of other classes are generally not designed to maintain adequate stability needed; however, corrections obtained within a few days of the volume calibration may be used if the uncertainty is sufficiently small. Mass standards are selected so that they are slightly larger than the volume of water and container or transfer vessel to be weighed. Linearity errors or additional uncertainties may need to be considered when the mass standards are slightly less than the volume or container being measured. When summations of masses are used, the summation mass is used, and the "effective density" must be calculated, taking care not to use "average" density values.

2.3.3 Filter paper and padding for protection of mass standards when placed on the balance pan, stacked, or placed on the calibration item may be used. When all such materials are used, care must be taken to determine when and how the mass of these items are incorporated into the process (as tare weight, or as tared/zeroed off the balance readings).

2.3.4 A transfer vessel is required that is made of some inert and stable material with a volumetric capacity larger than the volume to be measured and a port large enough to fill the vessel on the top surface. The transfer vessel should be as small as possible to minimize its influence on the balance, but approximately 25 % larger than the volume to be measured to prevent water loss due to splashing out of the top fill port. The transfer vessel may need to be suspended with a hoist or mounted to a pallet to be moved with a forklift or pallet jack.

2.3.5 Weight handling equipment is required for this procedure that has adequate capacity for handling the filled and empty transfer vessel. It must have the ability to be moved slowly and gently to minimize water sloshing and to gently set the empty or filled vessel on the balance pan.

2.3.6 Thermometers, with resolution and uncertainty less than 0.1 °C to determine water and volumetric proving system temperatures. Submersible thermometer probes and cables must be long enough to measure the temperature of the water near the center of the transfer vessel. Therefore, liquid-in-glass thermometers are not adequate.

2.3.7 Barometer with resolution and uncertainty less than 135 Pa (1 mmHg to determine barometric pressure (absolute).

2.3.8 Hygrometer with resolution and uncertainty less than 10 % to determine relative humidity.

2.3.9 Pressure gauge(s) with resolution and uncertainty less than 1 psig for determining the volumetric proving system pressure (where applicable).

2.3.10 If a proving system does not have operational temperature sensors and pressure gauges installed, the initial inspection should reveal that the proving system is not adequately prepared for calibration. If the calibration relies on the accuracy of the temperature sensors and pressure gauges installed on the prover, they must be installed prior to the volume calibration and have current calibration reports from an accredited calibration laboratory that are readily available for inspection. When laboratory pressure and temperature sensors are used during the calibration all laboratory sensors must all have current calibration reports.

2.3.11 Mechanical problems and sources of errors that may be encountered during a calibration and affect the ability of the system to repeat may include many possibilities, several of which are listed below. Mechanical problems with the volumetric standard must be corrected before calibration. Quantification of errors that cannot be eliminated must be evaluated and included in the uncertainty budget table.

- Proper operation of the system;
- Air in the system;
- Instability of the temperature of water or ambient conditions;
- Pressure fluctuations in the system;
- Error in reading the test measure(s);
- Incorrect drain time for reading test measure(s);
- Contamination of the systems;

- Damaged measures/system;
- Error in reading temperature or pressure;
- Displacer problems (when present);
- Malfunction of switches (when present);
- Damage, worn, or badly fitted seals;
- Damage, scoring, or pitting on the inside of the proving system;
- Damaged or misaligned detector switches (when present);
- Light interference in optical switches (when present);
- Malfunction of solenoid valve (when present);
- Leaks in prover/system or bypass valves.

2.3.12 Errors that may be encountered in weighing steps of the calibration may include any of the items listed below. Quantification of errors that cannot be eliminated must be evaluated and included in the uncertainty budget table.

- Balance drift;
- Off center loading of the balance;
- Non-linearity of the balance and improper selection of the mass standards to calibrate the weighing instrument in the range of use;
- Air currents or convection currents when weighing is performed in a large mass laboratory or when the temperature of the water, system, or laboratory are not adequately equilibrated;
- Care in placing items on the weighing instrument;
- Inadequate water quality.

2.4 Procedure

2.4.1 Inspect the volumetric proving system to verify that it and associated piping are drained, clean, and safe for placement in the laboratory. For large volumetric proving systems, ensure that there is adequate space in the laboratory to safely position the system near the pure water source and that a drain is accessible and that the proximity of the mass calibration area is convenient. Direct the driver to position to system appropriately, taking appropriate precautions to prevent injury to anyone or damage to the laboratory and/or equipment and standards. Allow adequate time for the system under test to equilibrate with the laboratory environment. The time required will be dependent on the difference between inside and outside temperature and relative humidity.

2.4.2 Initial inspection. Record the data about the system being calibrated, taking care to record all applicable serial numbers, coefficients of cubical expansion, reference temperatures, flow rates, presence or lack of the calibration status of all auxiliary standards associated with the standard (such as pressure gauges and temperature sensors), and means for sealing the system after calibration.

2.4.3 Install, or have the owner install, volumetric proving system hardware per manufacturer's instructions or have the owner provide this service. (Hardware must be provided by device owner).

2.4.4 Connect the water supply to the volumetric proving system.

2.4.4.1 In general, ensure that all air is bled from the system, suitable water supply is provided, and the device temperature is in equilibrium with that of the water supply. Several runs are generally required prior to the calibration to ensure that all air is out of the system and that all components are in thermal equilibrium with the water. Set device valves as appropriate for the test to be conducted.

2.4.4.2 Follow the operator's guidance (and per manufacturer's instructions) to become familiar with and to establish correct valve positions for each step of the calibration when applicable.

2.4.5 Install and/or use calibrated thermometers with evidence of current metrological traceability to record air temperature, water temperature, detector bar temperature (T_d) (where present), volumetric proving system temperatures (T_p).

2.4.6 Install and/or use calibrated pressure gauge(s) to record prover pressure(s) (P_p) when present and appropriate.

2.4.7 Analyze the piping and valves to determine if the configuration needs to be changed to deliver water into the transfer vessel.

2.4.8 Record all the prover-specific variables on the data entry form.

Note: All references in the following weighing options to the empty transfer vessel assume that the empty transfer vessel has been placed in a "wetted-down" condition. This wet down condition is achieved when the vessel has been filled with water and then drained using the same process that will be used throughout this calibration. Several runs may be needed on the volumetric proving system to ensure proper wetting of all lines and drains before the measurement data is recorded.

2.4.9 Weighings (Option A)

2.4.9.1 Zero the balance. Place a standard mass, M_{s1}, on the balance platform. M_{s1} should be slightly larger than the mass of the empty transfer vessel. Record the balance indication as O_1.

2.4.9.2 Zero the balance and then place the empty transfer vessel on balance platform and record the balance indication as O_2. Caution: the transfer vessel must be dry on the outside for all weighings.

2.4.9.3 Record air temperature, barometric pressure and relative humidity at the time of these measurements. Due to timing of this type of calibration, environmental conditions must be recorded for both the empty and full transfer vessel.

2.4.9.4 Zero the balance and then place a standard mass, M_{s2}, on the balance platform. M_{s2} should be slightly larger than the mass of the filled vessel. Record the balance indication as O_3. (This observation may be made before or after the weighing of the filled transfer vessel, depending on laboratory configurations and convenience).

2.4.9.5 Fill the transfer vessel through the volumetric proving system. Do not fill the transfer vessel while it is on a laboratory balance. Ensure that all the water from the volumetric standard or registered on a meter is captured in the transfer vessel. Verify that the water temperature in the system is uniform and stable throughout the filling process (± 0.2 °C). Read and record all pertinent volumetric standard temperatures and pressures during the calibration sequence. Place the lid on the transfer vessel to limit evaporation.

2.4.9.6 Zero the balance and then place the filled transfer vessel on the balance and record the balance indication as O_4.

2.4.9.7 Read and record the temperature of the water in the transfer vessel immediately after weighing. It is critical to measure the temperature after weighing to ensure that water is not removed from the transfer vessel prior to obtaining the mass value.

2.4.9.8 Record the air temperature, barometric pressure and relative humidity at the time of these measurements.

2.4.9.9 Make at least five replicate runs with at least two runs at a flow rate differing from the others by at least 25 % The measured result (V_{60}) of each of these calibration runs must agree within 0.02 % of the average volume, or the limits on the standard deviation or range charts (whichever is smaller) which may be evaluated through the use of an F-test, of all volume calibration runs (i.e., the maximum result minus the minimum result must be less than 0.02 % of the average measured volume).

2.4.9.10 Calculate the volume using Equation 1 in Section 3.1.1., (Option A).

2.4.10 Weighings (Option B)

2.4.10.1 Place mass standards that approximate the mass of the empty transfer vessel (with a lid) and adequate filter paper or other clean, lint-free padding material (to protect the standards being used) on the electronic balance. With these items on the balance, zero the balance. Note: The padding materials must be included on all measurements or their mass treated as tare.

2.4.10.2 Place additional mass standards approximating the nominal mass of the water volume to be measured on the balance. Take care to place the weights on filter paper or other appropriate protective padding. Record this balance indication as O_1.

2.4.10.3 Remove all mass standards. Record the air temperature, barometric pressure, and relative humidity readings.

2.4.10.4 Place the empty transfer vessel on the balance, with the padding material and zero the balance indication. Remove the transfer vessel, lid, and padding material and record the base empty vessel reading (B).

2.4.10.5 Fill the transfer vessel from the volumetric proving system. Read and record all pertinent volumetric standard temperatures and pressures during the calibration sequence. Place the lid on the transfer vessel to limit evaporation.

2.4.10.6 Immediately prior to weighing full vessel record balance reading as (d_1). This reading will be used to calculate any balance drift that occurred while the transfer vessel was being filled.

2.4.10.7 Place the filled vessel and all padding material on the balance. If needed, add known mass standards as tare weights to bring the water mass up to the mass of the standards used for O_1. This balance reading is recorded as O_2. Record the mass of all tare weights.

2.4.10.8 Remove the filled transfer vessel and record the balance indication as (d_2). This reading is used to calculate balance drift over the entire calibration process.

2.4.10.9 Immediately after removing the filled transfer vessel from the balance, record the temperature of water in the vessel.

2.4.10.10 Make at least five replicate runs with at least two runs at a flow rate differing from the others by at least 25 % when the system allows for varying flow rates. The range measured result (V_{60}) of each of these calibration runs must agree within 0.02 % of the volume (i.e., the maximum result minus the minimum result must be less than 0.02 % of the volume being measured) or be less than the limits on the standard deviation or range charts (whichever is smaller).

2.4.10.11 Calculate the volume using Equations 2 and 3 in Section 3.1.1., (Option B).

3 Calculations

3.1 Compute the volume at the temperature of the measurement, V_t, for each determination using the equation:

3.1.1 Option A.

$$V_t = \left[\frac{O_4}{O_3} M_{s2} \left(1 - \frac{\rho_a}{\rho_{s2}} \right) - \frac{O_2}{O_1} M_{s1} \left(1 - \frac{\rho_a}{\rho_{s1}} \right) \right] \left(\frac{1}{\rho_w - \rho_a} \right) \qquad \text{Eqn. (1)}$$

Table 2. Variables for volume equation.

Variable	Description
M_s, M_{s1}, M_{s2}	mass of standards M_{s1}, is the mass of the standards used with the empty transfer vessel M_{s2}, is the mass of the standards used with the transfer vessel after filling with delivered water
ρ_s	density of M_S standards
ρ_w	density of water at the temperature of measurement
ρ_a	density of air at the conditions of calibration – may be different for empty and filled weighing of the transfer vessel
V_t	Volume at the temperature of the test for the unknown prover/system

3.1.2 Option B.

$$V_t = \left\{ \left[O_{2c} \left(\frac{M_s}{O_1} \right) \left(1 - \frac{\rho_a}{\rho_s} \right) \right] - M_t \left(1 - \frac{\rho_a}{\rho_t} \right) \right\} \left(\frac{1}{\rho_w - \rho_a} \right) \qquad \text{Eqn. (2)}$$

$$O_{2c} = O_2 + \left\{ \left[\frac{(d_1 + d_2)}{2} \right] - B \right\} \qquad \text{Eqn. (3)}$$

Table 3. Variables for volume equations.

Variable	Description
O_1	Observation 1, balance reading for mass standard
O_2	Observation 2, balance reading for water delivered from prover
O_{2c}	Drift-compensated O_2 reading
d_1	Drift while filling the vessel
d_2	Drift over entire process
B	Base balance reading drift is based on
M_S	Mass of mass standards (true mass, vacuum mass)
M_t	Mass of tare weights
ρ_a	Air density
ρ_s	Density of M_S
ρ_t	Density of M_t
ρ_w	Water density (calculated using GLP 10 equations)

3.2 Compute V_{60}, the volume at 60 °F, for each run, using the equations noted below:

$$V_{60} = \frac{V_t}{CCF}$$

where,

$$CCF = CPL_p \times CTS_{p(1\text{ or }2)} \times CPS_p$$

$$CPL_p = \frac{1}{\left[1-\left(0.0000032 \times P_p\right)\right]} \qquad \text{Eqn. (4)}$$

$$CTS_{p1} = \left(1+G_a\left(T_p - T_b\right)\right)\left(1+G_l\left(T_d - T_b\right)\right)$$

$$CTS_{p2} = \left(1+G_c\left(T_p - T_b\right)\right)$$

$$CPS_p = 1+\left(\frac{\left(P_p \times ID\right)}{E \times WT}\right)$$

Table 4. Additional Variables for Volume Equations.

Variable	Description
T_b	Reference (Base) temperature
T_d	Detector bar temperature (Temperature, detector) – where present
T_p	Prover temperature (Temperature, prover)
P_p	Prover pressure (Pressure, prover) in psig
E	Modulus of elasticity (flow tube)
WT	Wall Thickness of flow tube
ID	Inside Diameter of flow tube
G_l	Coefficient of **linear** thermal expansion (switch bar) – where present (either linear and area values are used in combination or the cubical value is used)
G_a	Coefficient of flow tube **area** thermal expansion (either linear and area values are used in combination or the cubical value is used)
G_c	Coefficient of **cubical** thermal expansion (either linear and area values are used in combination or the cubical value is used)
CPL	Correction for the pressure on the liquid
CTS	Correction for the temperature on the standard prover CTS_{p1}, based on **linear and area** coefficients of expansion or CTS_{p2}, based on **cubical** coefficients of expansion
CPS	Correction for the pressure on the standard prover
CCF	Combined correction factor

3.3 Calculate water density for air-saturated water, using the equations provided in GLP 10.

3.4 Calculate the air density per NISTIR 6969, Selected Mass Calibration Procedures, SOP 2, Option B.

3.5 Calculate the within process standard deviation, s_w, for the replicate runs and determine the applicable degrees of freedom (number of replicates minus one; if five runs are made, there are four degrees of freedom). The repeatability data is plotted on a standard deviation chart.

3.6 An F-test may be incorporated to compare observed standard deviations from the current calibration with the accepted standard deviations of the process for similar type proving systems used as standards. Where used, calculate the F statistic to compare the observed within process standard deviation, s_w, to the accepted (pooled) within process standard deviation for the measurement process. (See NISTIR 6969, Sections 8.4 and 8.9.2, for more information on pooling standard deviations and F-tests).

$$F = \frac{s^2_{w\,\text{Observed}}}{s^2_{w\,\text{Accepted}}} \qquad \text{Eqn. (5)}$$

The calculated F statistic must be less than the F value obtained from an F table at 95 % confidence level (Table 9.12, NISTIR 6969) to be acceptable. The F value is obtained from the F table for numerator degrees of freedom and denominator degrees of freedom equal to the number of degrees of freedom in the pooled within process standard deviation. If the data fails the F-test and the source of the error cannot be determined conclusively and corrected, the measurement must be repeated.

3.7 Calculate and report the mean volume of the volumetric standard at its applicable reference temperature and reference pressure.

If adjustments were made during replicate runs, report the "as found" volume or the mean of "as found" volumes and the "as left" volume or mean of "as left" volumes, as applicable, at the appropriate reference temperature. (I.e., do not calculate a mean value by combining "as found" and "as left" values when adjustments are made). If the standard has bi-directional volumes, report the mean volume for each direction at its applicable reference temperature and reference pressure.

3.8 Calculate the uncertainty for the calibration using Section 5.

4 Measurement Assurance

4.1 Record and plot the values of the five runs and calculate the observed standard deviation to determine short-term repeatability of the measurement process, s_w following SOP 20. The range of the five runs must repeat to within 0.02 % of the volume, or the standard deviation must be less than applicable control/action limits on standard deviation charts, or the observed standard deviation must pass the F-test statistics (as applicable).

For standards that may be and are adjusted, do not combine an "as found" value with an "as left" value for the replicate runs when entered a standard deviation chart; use the adjusted value from those runs with the as left value from remaining runs, all at the applicable reference temperature, when entering values in a standard deviation or range chart. A minimum of 12 replicate measurements are required to establish initial process limits and 25 to 30 points are required for reporting valid uncertainty values.

4.2 Perform applicable calibrations of check standards where available.

5 Assignment of Uncertainties

The limits of expanded uncertainty, U, include estimates of the standard uncertainty of the mass standards used, u_s, plus the uncertainty of measurement, s_p, and the additional items noted below and in the uncertainty budget table, Table 4, at approximately a 95 % level of confidence. See SOP 29 for the complete standard operating procedure for calculating the uncertainty.

5.1 The standard uncertainty for the mass standards, u_s, is obtained from the calibration certificates for the standards used. The combined standard uncertainty, u_c, is used and not the expanded uncertainty, *U*, therefore the reported uncertainty for the standard is usually divided by the coverage factor *k*.

5.2 The standard deviation of the measurement process is obtained from replicate measurements and standard deviation chart performance data. See SOP 17 or 20.

The value for s_p is obtained from the standard deviation chart data and results for the current run must be compared to the standard deviations observed from replicate measurements over time. The standard deviation of the process determined from this approach incorporates a repeatability factor related to the precision of all weighings and need not be duplicated.

5.3 Other standard uncertainties usually included for this type of calibration level include uncertainties associated with water temperature measurements, thermometer accuracy, calculation of air density, pressure measurements and standard uncertainties associated with the density of the standards used or the lack of internal cleanliness.

5.4 An example uncertainty budget table is shown in Table 4. Additional components may need to be considered depending on the technology being calibrated.

Table 5. Example uncertainty budget table.

Uncertainty Component Description	Symbol	Source	Typical Distribution
Mass Standards (filled; empty)	u_s	Calibration certificate(s)	Expanded divided by k
Mass Density	$u_{\rho s}$	Assumed reference densities, or OIML R111	Rectangular
Standard deviation of the process	s_p	Standard deviation chart	Normal, 1s
Prover Temperature	u_{tp}	Ability to measure and possibility of gradients	Rectangular
Water Temperature	u_{tw}	Ability to measure and possibility of gradients	Rectangular
Water Density Equation	$u_{\rho w}$	GLP 10	Rectangular
Water Compressibility	u_K	Kell, CIPM Reference	Rectangular
Cubical Coefficient of Thermal Expansion (CCE)	u_{Gc}	5 % to 10 % of the CCE (EURAMET CG-19)	Rectangular
Area Coefficient of Thermal Expansion	u_{Ga}	5 % to 10 % of the CCE (EURAMET CG-19)	Rectangular
Linear Coefficient of Thermal Expansion	u_{Gl}	5 % to 10 % of the CCE (EURAMET CG-19)	Rectangular
Modulus of Elasticity (E)	u_E	Estimate 1 % of the E	Rectangular
Prover Pressure	u_{psig}	Calibration certificate	Rectangular
Flow Tube Internal Diameter (ID)	u_{ID}	Estimate by one division in last decimal place	Rectangular
Tube Wall Thickness (WT)	u_{WT}	Estimate by one division in last decimal place	Rectangular
Air Temperature	u_{ta}	NISTIR 6969, SOP 2, Calibration certificate	Rectangular
Barometric pressure (absolute)	u_p	NISTIR 6969, SOP 2, Calibration certificate	Rectangular
Relative Humidity	u_{RH}	NISTIR 6969, SOP 2 Calibration certificate	Rectangular
Air Density Equation	$u_{\rho a\ eqn}$	NISTIR 6969, SOP 2, Calibration certificate	Rectangular
Viscosity	u_V	NIST SP 250-72	Rectangular

5.5 Uncertainty Evaluation

Where applicable, uncertainties for volume calibrations that are assessed for conformity must meet decision rule criteria in the applicable documentary standards.

6 Calibration Certificates

6.1 Report results as described in NISTIR 6969, SOP 1, Preparation of Calibration Certificates, with the addition of the following:

6.1.1 Volume, reference temperature, uncertainty, material, thermal coefficient of expansion (assumed or measured), construction, any identifying markings, tolerances (if appropriate), laboratory temperature, water temperature(s) at time of test, barometric pressure, relative humidity, and any out–of-tolerance conditions. Volume may need to be reported as "up-stream" and "down-stream" volumes for bi-directional provers.

6.1.2 The status of items that are not calibrated by the laboratory but that may be used to perform measurements must be noted on the calibration report (e.g., temperature sensors, pressure gauges, or full flow rates that may be used in the field but that are not calibrated by the laboratory). A statement such as the following must be included on calibration reports: "Only items presented on this calibration report were tested under the conditions shown". Any sensors or gauges that were not calibrated by the laboratory should be clearly identified on the report (e.g., "Calibration certificates for the system included temperature sensors and pressure gauges that were provided to the laboratory for evaluation prior to calibration and were found to be acceptable.")

6.2 Conformity Assessment

Evaluate compliance to applicable tolerances as needed or required by the customer or by legal metrology requirements. Compliance assessments must note the applicable documentary standard and which portions of the standard were or were not evaluated. The uncertainty for volume calibrations must be less than the tolerances published in the applicable documentary standards. For volume calibrations where the unknown standard can be adjusted, it is standard practice to adjust the standard or leave the scale plate in a position close enough to its nominal volume to ensure that the absolute value of the measurement result plus the uncertainty is less than the applicable tolerance. Where the unknown standard cannot be adjusted, and a portion of the uncertainty band from the error exceeds tolerance limits, it is not appropriate to state compliance with the tolerances unless additional decision rules are communicated with and agreed to by the end user. Correction values (measurement results) may need to be used by the end user in such cases.

Where no specific documentary standards apply, or a laboratory does not perform an assessment, a notice should be included on the calibration certificate to the effect that a legal metrology assessment was not performed. (For example, "Conformity assessment was not completed as a part of this calibration. Accurate use in field testing requires assessment of repeatability in field applications as well as proper training of the operator, maintenance of the proving system, integrity of all seals and sensors, and any other auxiliary mechanical or electronic components. The entire system has not been assessed for suitability as a field standard for weights and measures applications.")

7 References

Chapter 4, Manual of Petroleum Measurement Standards, and the following Sections as applicable:

Section 1 – Introduction
Section 2 – Conventional Pipe Provers
Section 3 – Small Volume Provers
Section 4 – Tank Provers
Section 5 – Master-Meter Provers
Section 6 – Pulse Interpolation
Section 7 – Field Standard Test Measures
Section 8 – Operation of Proving Systems

NIST Handbook 105-7: Specifications and Tolerances for Reference Standards and Field Standard Weights and Measures, 7. Specifications and Tolerances for Dynamic Small Volume Provers, 1996.

Bean, V. E., Espina, P. I., Wright, J. D., Houser, J. F., Sheckels, S. D., and Johnson, A. N., NIST Calibration Services for Liquid Volume, NIST Special Publication 250-72, National Institute of Standards and Technology, Gaithersburg, MD, (2009).

EURAMET Calibration Guide 19, Guidelines on the Determination of Uncertainty in Gravimetric Volume Calibration, (Version 2.1, 03/2012).

Appendix A

Gravimetric Calibration Data Sheet

Company Name: ____ Test No: __________________

Address: ____ Purchase Order No: __________________

City, State, Zip: ____ Date Scheduled: __________________

Representative: ____ Date Received: __________________

Phone: __________________ Fax: ________________ Date Tested: __________________

Email: ____ Date Returned: __________________

Company URL: ____ Next Appointment: __________________

General Description ____

Manufacturer: ____

Model Number: _______________ Serial Number: ___________

Condition: ____

Compressibility factor for water	3.2000E-06	Cubical Thermal Expansion Coefficient (G_c)	
Reference Temperature (*Tb*) (ºF)		Area Thermal Expansion Coefficient (G_a)	
Reference Temperature (ºC)		Detector Thermal Expansion Coefficient (G_l)	
Reference Pressure (psig)		Modulus of Elasticity (flow tube) (*E*)	
		Flow Tube Inside Diameter (inches) (*ID*)	
		Flow Tube Wall Thickness (inches) (*WT*)	

Mass standard(s) data:

Identification (ID) (Note ID and whether for Filled or Empty Transfer Vessel)	Nominal Mass	Mass Correction[A]	Expanded Unc: From Cal. Cert.	Unc: *k* factor	Density g/cm^3
S					
S					
S					
S					
S					
S					
S					
S					

[A] Mass Correction = *Mass* values are required for this procedure (i.e., Conventional Mass must *not* be used).

Observations (Option A):

Fill Number	Fill # 1	Fill # 2	Fill # 3	Fill # 4	Fill # 5
Flow Rate	Fast	Fast	Fast	Slow	Slow
Run Time (sec)					
Balance Reading O_1 (Standards for Empty Transfer Vessel)					
Balance Reading O_2 (Empty Transfer Vessel)					
Balance Reading O_3 (Standards for Filled Transfer Vessel)					
Balance Reading O_4 (Filled Transfer Vessel)					
Air Temperature (ºC)					
Barometric Pressure (mmHg)					
Relative Humidity (% RH)					
Water Temperature (ºC) – record all values					
Detector Bar (where present) Temperature (T_d) (ºC)					
Prover Temperature (T_p) (ºC)					
Prover pressure (P_p) (psig)					

Observations (Option B):

Fill Number	Fill # 1	Fill # 2	Fill # 3	Fill # 4	Fill # 5
Flow Rate	Fast	Fast	Fast	Slow	Slow
Run Time (sec)					
Balance Reading O_1 (standards)					
Balance Reading Empty Transfer Vessel Tared and Removed (B))					
Balance Reading Immediately Before O_2 Reading (d_1)					
Balance Reading O_2 (water)					
Balance Reading Full Transfer Vessel Removed (d_2)					
Validity Check (Compares d_1-B, and d_2-d_1)					
Drift Compensated O_2 (O_{2c})					
Air Temperature (°C)					
Barometric Pressure (mmHg)					
Relative Humidity (% RH)					
Water Temperature (ºC) – record all values					
Water Temperature (°F)					
Detector Bar (where present) Temperature (Td) (°C)					
Detector Bar (where present) Temperature (Td) (°F)					
Prover Temperature (Tp) (°C)					
Prover Temperature (Tp) (°F)					
Prover pressure (Pp) (psig)					

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