

SOP 18

Standard Operating Procedure for Calibration of Graduated Neck-Type Metal Volumetric Field Standards Using a Slicker-Plate Type¹

1 Introduction

1.1 Purpose of Test

This procedure may only be used to calibrate small non-pressurized, graduated neck-type, metal field standards such as the 5 gal (or 20 L) standards used by weights and measures officials to test liquid dispensing equipment, gasoline pumps within a narrow range of laboratory and water temperatures (e.g., laboratory and water temperatures between 50 °F and 70 °F) and for test measure graduations that are at least 1 in³. Outside these limitations, SOP 19 is required.

1.2 Conformity Assessment

The test measure or prover being calibrated should be evaluated for conformance to appropriate specifications and tolerances (using the checklist provided in NIST Handbook 105-3, Specifications and Tolerances for Graduated Neck Type Volumetric Field Standards, 2010) if being used for legal weights and measures applications. (Alternatively, if requested by the customer, evaluation against OIML R 120 (2010), Standard capacity measures for testing measuring systems for liquids other than water, may be referenced). Where compliance is required by law, conformity evaluations should be conducted prior to performing calibrations. See Section 6.2 for reporting results.

1.3 Limiting factors:

This procedure uses a slicker-plate type standard to fill an unknown test measure of equal nominal volume in one delivery. It requires water temperature stability during the transfer from the standard to the unknown test measure within the limits shown in Table 1. SOP 19 is a more appropriate procedure when temperature corrections are needed due to lack of water equilibration, temperature differences between the standard and unknown provers, performance of the calibration outside the stated temperature range, and for unstable environments. Even water that is temperature-equilibrated with the laboratory environment may not provide adequate measurement results depending on test measure resolutions.

If the limits in Table 1 are exceeded, or materials other than stainless steel and mild steel are used, use SOP 19. These limits are to ensure that the impact of errors resulting from temperature differences on measured values is less than the

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 have been used to reflect the practical needs of the laboratories performing these measurements as appropriate. Most laboratory standards for this calibration procedure are 5 gal “slicker-plate” type standards. Very few laboratories have 20 L “slicker-plate” type standards.

resolution and repeatability on a 20 L or 5 gal test measure with 1 in³ graduations. For graduations less than or equal to 0.5 in³, errors due to temperature variations, including the ability to measure temperature and ensure stable temperatures in both the standard and test measure, require the use of SOP 19.

Table 1. Temperature limitations between 50 °F and 70 °F for 1 in³ graduations.

Graduation Size in³	Standard Material	Unknown Material	Temperature limit between standards (°C)
1	Stainless steel	Stainless steel	< 0.5 °C
1	Stainless steel	Mild steel	< 0.2 °C

1.4 Prerequisites

- 1.4.1 Verify that the unknown prover has been properly cleaned and vented with all petroleum products removed prior to submission for calibration to ensure laboratory safety.
- 1.4.2 Verify that current calibration certificates with measurement values and uncertainties are available for all of the standards used in the test. All calibration values must have demonstrated metrological traceability to the international system of units (SI). Metrological traceability may be to the SI through a National Metrology Institute such as NIST.
- 1.4.3 Verify that the slicker-plate type standard has sufficiently small standard uncertainties for the intended level of calibration (i.e., less than 0.2 in³ with an approximate 95 % level of confidence).
- 1.4.4 Verify the availability of an adequate supply of clean, preferably soft water (filtered and thermally equilibrated as appropriate) (GLP 10). Water does not need to be distilled or deionized for use in this procedure. The equations used in GLP 10 for the calculation of water density (air saturated) may be used without a significant impact on the measurement results.
- 1.4.5 Verify that the operator has had specific training and is proficient in SOP 18, GMP 3, SOP 17 and is familiar with the operating characteristics and conditioning of the standards used.
- 1.4.6 Verify that the laboratory facilities meet the following minimum conditions to enable meeting the expected uncertainty achievable with this procedure:

Table 2. Laboratory environmental and equilibrated water conditions.

Procedure	Temperature	Relative Humidity
Volume transfer	10 °C to 21 °C (50 °F to 70 °F) Stable to ± 1.0 °C / 1 h	35 % to 65 % Stable to ± 20 % / 4 h

1.5 Field tests

- 1.5.1 A “field” calibration is considered one in which a calibration is conducted in an uncontrolled environment, such as out-of-doors. Calibrations conducted under field and laboratory conditions are not considered equivalent, and uncertainties must reflect the conditions of the calibration.
- 1.5.2 SOP 19 is a more suitable procedure for non-laboratory conditions. The care required for field calibrations includes proper safety, a clean and bubble-free water supply, measurement control programs, and a stable temperature environment shaded from direct sunshine to allow the prover, field standard, and clean test liquid (water) to reach an equilibrium temperature with minimal evaporation. Environmental conditions must be selected to be within stated laboratory conditions as shown in Table 2, during the measurements. All data and appropriate environmental conditions must be documented regardless of test location.
- 1.5.3 An increased number of check standard verifications are required to ensure continued suitability of calibration values generated in field conditions as well as to verify the validity of any standards taken out of a secure laboratory environment once the standard(s) are returned to the laboratory.

2 Methodology

2.1 Scope, Precision, Accuracy

- 2.2 This procedure is applicable for the calibration of a small test measure within the limitations of the standards available. The precision attainable depends on the care used in the various volumetric adjustments and readings, in the strict observance of drainage times, and the internal cleanliness of the various volumetric vessels which can influence their drainage characteristics. The accuracy depends on the uncertainties of the calibrations of the standards used.

2.3 Summary

Water is delivered from the standard to the vessel under calibration. Because the “to deliver” volume of the latter is calibrated, the delivery must be into a “wetted-down” vessel. The wet-down also ensures consistent retention in the slicker-plate type standard. The gauge scale of the test vessel is adjusted to a correct reading, as necessary, and then sealed. Hand held test measures require a 30 s (± 5 s) pour

followed by a 10 s drain, with the measure held at a 10 degree to 15 degree angle from vertical, during calibration and use. Provers are emptied by gravity drain, followed by a 30 s final drain after the cessation of the main flow.

2.4 Standards and Equipment

2.4.1 Calibrated slicker-plate type standard made of stainless steel.

2.4.2 Calibrated thermometer, with a resolution and uncertainty less than 0.1 °C.

2.4.3 Meniscus reading device. See GMP 3.

2.4.4 Timing device (calibration is not required; uncertainty of the measurement only needs to be less than 5 s for a 30 s pour or drain time).

2.5 Procedure

2.5.1 Cleanliness Verification - Fill and drain both standard and vessel to be calibrated and check for any soiling that would affect drainage, as evidenced by clinging droplets, greasy films, and the like. Clean either or both with non-foaming detergent and water, as necessary, and rinse thoroughly. Note: Many laboratories have a policy regarding cleanliness of submitted volumetric standards to minimize water contamination with flammable petroleum products. See GMP 6.

2.5.2 Fill the standard with water and then transfer the water into the unknown prover establishing a wet down of the standard. Wait 30 s after the cessation of the main flow on the standard before closing the drain valve.

2.5.3 Wet down the unknown test measure or prover to its nominal level and empty it following the applicable procedure. Hand held test measures require a 30 s (± 5 s) pour followed by a 10 s drain, with the measure held at a 10 degree to 15 degree angle from vertical, touching off any adhering drop from the neck, during calibration and use. Provers are emptied by gravity drain, followed by a 30 s final drain after the cessation of flow.

Note: Filling the vessel from the slicker-plate type standard following the instructions in steps 2.5.1, 2.5.2, and 2.5.3 will ensure that both the standard and vessel are properly “wetted-down”.

2.5.4 Run 1. Fill the slicker-plate standard with water by placing the fill tube or hose inside the standard near the bottom to minimize air bubbles. Fill the standard to slightly higher than the rim as the water level is raised by surface tension. Ensure that the reference standard is level to ensure consistent drain and retention characteristics. Measure and record the temperature in the standard. Use the slicker plate to strike off excess water, checking to see that no air bubbles are entrained in the water during this process.

- 2.5.5 Position the unknown vessel beneath the slicker-plate standard drain valve or piping to be sure no water is lost. Open the valve at the base of the standard at the same time as removing the slicker plate from the top of the standard to transfer water from the standard to the wet-down vessel. Allow a 30 s drain period after cessation of the main flow on the standard. Close the valve on the slicker-plate standard and then move the unknown vessel from beneath the drain valve or piping to prevent additional water transfer.
- 2.5.6 Level the test measure or prover and verify that the neck of the unknown test measure is vertical, or perpendicular to a level horizontal plane, by placing a level on the side of the neck as specified in NIST Handbook 105-3 (or suspend by its handle, if appropriate). Read and record the gauge reading. Measure and record the temperature of water in the unknown measure. If the temperature change between the standard and unknown test measure exceeds the allowable limits shown in Table 1, use SOP 19.
- 2.5.7 Adjust the graduated scale of the vessel as described in 3.3 if needed. Seal the scale adjustment device.
- 2.5.8 Run 2. Make a duplicate determination.
- 2.5.9 Replicate runs of the test measure or prover must agree within ± 0.02 % of the nominal volume during calibration at the reference temperature (± 0.2 in³ for 5 gal test measure), pass the F-test, or be within the limits on the standard deviation or range charts (as applicable). Agreement of 0.02 % is calculated as the difference between two consecutive runs, divided by the nominal volume, and then multiplied by 100.

Note: If excess disagreement between the runs is observed, check all vessels for cleanliness, leaks, or other damage, identify and correct any problems. Agreement problems may be due to temperature changes, contamination or lack of cleanliness, or poor field conditions, such as when calibration is conducted in an unstable environment. Agreement problems must be corrected before calibration can be completed.

3 Calculations

- 3.1 When the water temperature is reasonably close to 60 °F, the coefficients of expansion of the standard and the test vessel are sufficiently close together, and the deliveries and readings are made over a short period of time, temperature corrections are not made in this procedure. When conditions are not reasonably close to 60 °F and temperature corrections are needed, use SOP 19. Calculations provided in SOP 19 may be required for reporting volumetric results.
- 3.2 Within the accuracy requirements of Table 1, no temperature corrections arising from dissimilarities of the standard and vessel are used. If differences are suspected or observed, use SOP 19.

- 3.3 The reading of Run 1 is used to adjust the scale of the vessel, if necessary, to the correct reading, which is set at the calibrated volume of the slicker-plate standard at 60 °F. Record the adjusted value as the “as left” value. Run 2 will validate the setting. Alternatively, the average of Run 1 and Run 2 may be used with the adjustment made after Run 2. In that case, a validation run should be conducted to ensure correct setting of the gage plate.

Note: When the accuracy requirements necessitate a temperature correction, the temperature of the water that has been recorded in both the standard and the unknown should be used to perform calculations according to the procedure given in SOP 19.

- 3.4 Determine and report the volume of the test vessel as follows:

$$\text{Prover volume} = V_S - \text{gauge reading} \quad \text{Eqn. (1)}$$

where: V_S = Volume of the slicker-plate standard at the appropriate reference temperature (usually 60 °F).

- 3.1 Calculate the within process standard deviation, s_w , for the replicate runs. The degrees of freedom for this SOP is one. This value is plotted on the standard deviation chart and may be used to incorporate an F-test as well.
- 3.2 If the F-test is 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).

$$F = \frac{s_{w\text{Observed}}^2}{s_{w\text{Accepted}}^2} \quad \text{Eqn. (1)}$$

Where used, 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 one degrees of freedom for the numerator and the degrees of freedom for the denominator is equal to the number of degrees of freedom in the pooled within process standard deviation. If the repeatability evaluation fails and the source of the error cannot be determined conclusively, the measurement must be repeated (or possibly, the test measure must be rejected).

- 3.3 Calculate and report the mean volume of the volumetric standard at its applicable reference temperature.

If adjustments were made during replicate runs, report the “as found” volume or the mean of “as found” volumes and also 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).

4 Measurement Assurance

4.1 Duplicate the process with a suitable check standard. See SOP 17 or SOP 30. Plot the check standard volume and verify that it is within established limits. The mean of the check standard observations may be used to evaluate bias and drift over time when a reference value for the check standard is available. Check standard observations are used to calculate the standard deviation of the measurement process which contributes to the Type A uncertainty components.

4.1.1 A t-test may be incorporated to check the observed value against an accepted value.

$$t = \frac{(S_c - \bar{S}_c)}{s_p} \quad \text{Eqn. (2)}$$

The t-statistic is evaluated using Eqn. 2 with a 95 % confidence level. All values must be entered in the control chart, even if failing this statistic to ensure the variability obtained for the process is truly representative of the process and not unduly reduced over time. The observed value of the check standard is compared to the accepted mean value of the check standard and divided by the standard deviation for the check standard observations over time. The limits for the t-test are based on applicable warning and action limits on the control chart. The applicable limits are shown in SOP 20 and based on the number of replicates.

4.2 A standard deviation chart is also used for measurement assurance through the evaluation of replicate measurements. The standard deviation of each combination of Run 1 and Run 2 is calculated and the pooled (or average) standard deviation over time may be used to estimate the short-term variability in the measurement process. The short-term standard deviation may be used with an F-test (observed vs. accepted) to assess the measurement process. A standard deviation chart for unknown provers represents the variability in condition of test measures submitted for calibration as well as the short-term repeatability of the measurement process but does not monitor the stability of the reference standard.

For unknown standards that are adjusted, do not combine an “as found” value with an “as left” value for the two runs recorded in the chart; use the adjusted value from Run 1 and the value from Run 2, both at the applicable reference temperature, when entering values in a standard deviation or range chart.

5 Assignment of Uncertainties

The limits of expanded uncertainty, U , include estimates of the standard uncertainty of the laboratory volumetric standards used, u_s , plus the standard deviation of the process, s_p , and the additional items noted below and in the uncertainty budget table, Table 3, at an approximate 95 %

level of confidence. See SOP 29 for the complete standard operating procedure for calculating the uncertainty.

- 5.1 The standard uncertainty for the standard, u_s , is obtained from the calibration certificate. The combined standard uncertainty, u_c , is used and not the expanded uncertainty, U , therefore the reported uncertainty for the standard will usually need to be divided by the coverage factor k .
- 5.2 The standard deviation of the measurement process, s_p , is obtained from a control chart for the check standard so that it reflects performance over time (See SOP 17 or 20, and SOP 30). The larger of the value from the standard deviation over time for a check standard from Section 4.1 or from the standard deviation chart taken from Section 4.2 is used in the uncertainty calculations. Where multiple staff members perform this calibration and obtain values for the check standard, the standard deviation for a check standard may represent variability and systematic errors associated with the meniscus reading. When a standard deviation chart is used alone, the repeatability represents the process only and does not represent potential systematic differences in meniscus readings. In a laboratory with only one staff member performing calibration and entering data for a check standard, additional uncertainties for meniscus readings are needed per Section 5.3 and GMP 3.
- 5.3 Other standard uncertainties usually included at this calibration level primarily include 1) uncertainties associated with the ability to read the meniscus, only part of which is included in the process variability due to parallax and visual capabilities (See GMP 3), and 2) uncorrected temperature corrections related to the cubical coefficient of expansion for the prover under test, use of proper temperature corrections, and the accuracy of temperature measurements. Additional factors that might be included are: round robin or proficiency testing data showing bias, environmental variations over time, and drift of the standard as noted in control charts.
- 5.4 To properly evaluate uncertainties and user requirements (tolerances), assessment of additional user uncertainties may be required by laboratory staff. Through proper use of documented laboratory and field procedures, additional uncertainty factors may be minimized to a level that does not contribute significantly to the previously described factors. Additional standard uncertainties in the calibration of field standards and their use in meter verification may include: how the prover level is established, how delivery and drain times are determined, the use of a proper “wet-down” prior to calibration or use, the cleanliness of the prover and calibration medium, prover retention characteristics related to inside surface, contamination or corrosion, total drain times, and possible air entrapment in the water.
- 5.5 Example components to be considered for an uncertainty budget table are shown in Table 3.

Table 3. Example uncertainty budget table.

Uncertainty Component Description	Symbol	Source	Typical Distribution
Uncertainty of the standard (5.1)	u_s	Calibration certificate	Expanded divided by coverage factor
Accepted standard deviation of the process (5.2)	s_p	Control chart, standard deviation chart	Normal
Ability to read the Meniscus in S (5.3)	u_m	None if using a slicker-plate type standard	Triangular
Ability to read the Meniscus in X (5.3)	u_m	GMP 3	Triangular
Uncorrected temperature errors (5.3)	u_{te}	Calculated based on limits and SOP 19 equations	Rectangular
Uncertainty of bias or drift of standards (5.3)	u_b	From control chart	Rectangular
Water temperature (S)	u_{ts}	Consider accuracy, resolution, and gradients	Rectangular
Water temperature (X)	u_{tx}	Consider accuracy, resolution, and gradients	Rectangular
Cubical Coefficient of Expansion (CCE) on S	u_{CCE}	5 % to 10 % of the CCE (EURAMET CG-21)	Rectangular
Cubical Coefficient of Expansion (CCE) on X	u_{CCE}	5 % to 10 % of the CCE (EURAMET CG-21)	Rectangular
Uncertainty of drain time (insignificant if closely following the procedure)	u_d	From experimental data	Rectangular

5.6 Uncertainty Evaluation

Where applicable, uncertainties for volume calibrations that are assessed for conformity must be less than the tolerances specified in Handbook 105-3 and used for decision rules.

6 Calibration Certificate

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

“To Contain” or “To Deliver” 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.

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.

Appendix A Volume Transfer Data Sheet

Laboratory data and conditions:

Vessel Owner		Operator	
Vessel Identification		Date	
Material		Air Temperature	
Cubical Coefficient of Expansion		Relative Humidity	
Standard deviation of the process, s_p		Reference temperature of unknown prover	
s_p from Control Chart or Standard Deviation Chart			
Degrees of Freedom			

Volume standard(s) data:

Identification (ID) (Note ID of Standards)	Nominal	Volume/Correction	Expanded Unc: From cal. report	Unc: k factor	Cubical Coefficient of Expansion
S					
S					
S					
S					

Observations:

Run	Standard Material (MS/SS)	Unknown Material (MS/SS)	Water Temp in Standard (°C)	Unknown Gauge Reading (in ³)	Water Temp in Unknown (°C)	Adjustment Made?	Temperature Difference Acceptable for SOP 18?
1						___ Yes ___ No	___ Yes ___ No
2						___ Yes ___ No	___ Yes ___ No
3						___ Yes ___ No	___ Yes ___ No

Temperature Difference Evaluation:

If Standard is stainless steel (SS) and Unknown is stainless steel, the temperature of the water between the standard and unknown must not change more than 0.5 °C during the calibration.

If the standard is stainless steel and the unknown test measure is mild steel (MS), the change in water temperature between standard and unknown must be less than 0.2 °C during the calibration.

If these limits are exceeded, use SOP 19 calculations to determine the volume of the unknown volumetric measure.

This page is intentionally blank.