

SOP 18

Standard Operating Procedure for Calibration of Graduated Neck-Type Metal Volumetric Field Standards Volumetric Transfer Method¹

1 Introduction

1.1 Purpose of Test

This procedure may 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, for example. The test measure prover should be evaluated for conformance to appropriate specifications if being used for legal weights and measures applications.

The procedure assumes that the water temperature is stable during the transfer from the standard to the unknown test measure. 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, or unstable environments.

Limiting factors: For a 5 gal test with a stainless steel standard and stainless steel unknown test measure, the temperature of the water between the standard and unknown must not change more than 0.5 °C during the calibration. If the unknown test measure is mild steel, 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. This is to ensure that the impact on measured values is less than the resolution and repeatability on a 5 gal test measure with 1 in³ graduations. If smaller graduations are present, error due to temperature variations must be evaluated further or SOP 19 is recommended.

1.2 Prerequisites

1.2.1 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.

1.2.2 Verify that valid calibration certificates are available for all of the standards used in the test.

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.

- 1.2.3 Verify that the slicker-plate type standard to be used has sufficiently small standard uncertainties for the intended level of calibration (i.e., less than 0.2 in³ with a 95 % confidence interval).
- 1.2.4 Verify the availability of an adequate supply of clean water (GLP 10).
- 1.2.5 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.
- 1.2.6 Verify that the laboratory facilities meet the following minimum conditions to enable meeting the expected uncertainty achievable with this procedure:

Table 1. Laboratory environmental conditions.

Procedure	Temperature	Relative Humidity
Volume transfer	18 °C to 27 °C, stable to ± 2.0 °C/h	35 % to 65 % ± 20 % maximum change / 4 h

1.3 Field tests

- 1.3.1 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.
- 1.3.2 The care required for field calibrations includes proper safety, a clean and air-free water supply, measurement control programs, and a stable temperature environment shaded from direct sunshine to allow the prover, field standard, and test liquid (water) to reach an equilibrium temperature with minimal evaporation. Environmental conditions must be selected to be within stated laboratory conditions during the measurements. All data and appropriate environmental conditions must be documented regardless of test location. SOP 19 is a more suitable procedure for non-laboratory conditions.
- 1.3.3 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

2 Methodology

2.1 Scope, Precision, Accuracy

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.

2.2 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 "wet-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.

2.3 Equipment

2.3.1 Calibrated slicker-plate standard made of stainless steel, with recent calibration certificate and demonstrated metrological traceability to the international system of units (SI), which may be to the SI through a National Metrology Institute such as NIST, and whose volume is equivalent to that of the vessel to be calibrated.

2.3.2 Calibrated thermometer, accurate to ± 0.1 °C, with recent calibration certificate and demonstrated metrological traceability to the international system of units (SI), which may be to the SI through a National Metrology Institute such as NIST.

2.3.3 Meniscus reading device. (See GMP 3).

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

2.3.5 Supply of clean water, preferably soft water (filtered if necessary).

2.4 Procedure

2.4.1 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. (See GMP 6).

- 2.4.2 Fill a hand-held vessel with water to its nominal level and pour contents during a 30 ± 5 s period then drain for a 10 s period after cessation of flow. Touch off any adhering drop from the neck. If a stationary test measure is being calibrated, the valve is opened, the measure is emptied, followed by a 30 s drain time after the cessation of flow. This constitutes the "wet-down" condition. Filling the vessel from the slicker-plate type standard following the instructions in steps 2.4.3 and 2.4.4 will ensure that both the standard and vessel are properly "wet-down".
- 2.4.3 Run 1 - Fill the slicker-plate standard with water, raised by surface tension, slightly higher than the rim. Use the slicker plate to strike off excess water, checking to see that no air bubbles are entrained in the water during the leveling process.
- 2.4.4 Open the valve at the base at the same time as removing the slicker plate from the top of the standar to transfer water from the standard to the wet-down vessel. Allow a 30 s drain period after cessation of flow.
- 2.4.5 Level the vessel (or suspend it by its handle, if appropriate) and read the neck scale. Record the reading.
- 2.4.6 Adjust the graduated scale of the vessel as described in 3.3. Seal the scale adjustment device.
- 2.4.7 Run 2 - Make a duplicate determination, which should agree with the former within ± 0.02 % of the volume (± 0.2 in³ for 5 gal test measure). The test measure or prover must be capable of repeating within 0.02 % of the test volume during calibration.

NOTE: If excess disagreement, check all vessels for cleanliness, leaks, or other damage, identifying and correcting any problems. Repeatability problems may be due to contamination or lack of cleanliness, or poor field conditions, such as when calibration is conducted in an unstable environment. Repeatability problems must be corrected before calibration can be completed.

3 Calculations

- 3.1 Because the water temperature is usually 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. If prover volumes, errors and/or corrections are reported, use calculations provided in SOP 19.

- 3.2 Within the accuracy requirements, no corrections arising from dissimilarities of the standard and vessel are necessary. If differences are suspected, 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: If the accuracy requirements necessitate a temperature correction, the temperature of the water must be measured in both the standard and the unknown and the calibration is made according to the procedure given in SOP 19.

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

$$\text{Prover volume} = V_{Nom} + C_s - \text{gauge reading} \quad \text{Eqn. 1}$$

where:

$$\begin{aligned} V_{Nom} &= \text{Nominal Volume (taking care to match units)} \\ C_s &= \text{Correction from the calibration report for the slicker-plate standard} \end{aligned}$$

4 Measurement Assurance

- 4.1 Duplicate the process with a suitable check standard. See SOP 17, SOP 20 and SOP 30. Plot the check standard volume and verify it is within established limits OR a t -test may be incorporated to check the observed value against an accepted value. The mean of the check standard observations is used to evaluate bias and drift over time. Check standard observations are used to calculate the standard deviation of the measurement process which contributes to the Type A uncertainty components.
- 4.2 If a standard deviation chart is also used for measurement assurance, the standard deviation of each combination of Run 1 and Run 2 is calculated and the pooled (or average) standard deviation and may be used to estimate the short-term variability in the measurement process. The short-term standard deviation may be used to incorporate an F-test (Observed vs. Accepted) into the measurement process and represents the variability in condition of test measures submitted for calibration.

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 , at the 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 report. 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 control chart for the check standard to reflect 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 should be used in the uncertainty calculations.
- 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, 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 data showing reproducibility, environmental variations over time, and bias or 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.

6 Report

6.1 Report results as described in SOP 1, Preparation of Calibration/Test Results, 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.