SOP 18

Standard Operating Procedure for Calibration of Graduated Neck-Type Metal Volumetric Field Standards Volumetric Transfer Method[1]

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 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 change between the standard and unknown must be less than 0.5 ºC during the calibration. If the unknown test measure is mild steel, the change in temperature between standard and unknown must be less than 0.2 ºC 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 in^3 graduations. If smaller graduations are present, error due to temperature variations must be evaluated further.

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 the standards used in the test.

1.2.3 Verify that the standards to be used have sufficiently small standard uncertainties for the intended level of calibration

[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.

1.2.4 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, maximum change 2.0 °C/h	40 % to 60 % ± 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 should be selected to be as close to laboratory conditions as possible. All data and appropriate environmental conditions must be documented regardless of test location. SOP 19 is a more suitable procedure when poor conditions are noted.

1.3.3 An increased number of check standard verifications are required to ensure continued suitability of calibration values generated in field conditions.

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 will depend 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 volu-

metric vessels which can influence their drainage characteristics. The accuracy will depend 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 gauge scale 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 traceable to NIST, and whose volume equals that of the vessel to be calibrated.

2.3.2 Calibrated thermometer, accurate to ± 0.1 °C, with recent calibration certificate traceable to NIST.

2.3.3 Meniscus reading device. (See GMP 3).

2.3.4 Timing device (Calibration is not required.)

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 detergent and water, as necessary, and rinse thoroughly. (See GMP 6).

2.4.2 Fill 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. This constitutes the "wet-down" condition. Filling the vessel from the 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 slicker-plate standard with water, raised by surface tension, slightly higher than the rim. Use 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 valve at base and transfer water from the standard to the wet-down vessel. Allow a 30 s drain period after cessation of flow.

2.4.5 Level vessel (or suspend it by its handle, if appropriate) and read scale on its neck. Record reading.

2.4.6 Adjust the 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 in^3 for 5 gal test measure). If excess disagreement, check all vessels for cleanliness and repeat duplicate determinations until satisfactory agreement between consecutive runs is obtained.

2.4.8 The test measure or prover must be capable of repeating within 0.02 % of the test volume during calibration. 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, no temperature corrections are made. When conditions are not reasonably close to 60 ºF 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.

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 Determination of the test vessel volume:

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

where:

V_{Nom} = Nominal Volume (taking care to match units)
C_s = Correction on standard

4 Measurement Assurance

4.2 Duplicate the process with a suitable check standard or have a suitable range of check standards for the laboratory. See SOP 17 and SOP 30.

4.3 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.

4.4 The mean of the check standard observations is used to evaluate bias and drift over time.

4.5 Check standard observations are used to calculate the standard deviation of the measurement process.

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.

5.2 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*.

5.3 Standard deviation of the measurement process, s_p, is obtained from control chart performance (See SOP 17 or 20, and SOP 30).

5.4 Other standard uncertainties usually included at this calibration level include uncertainties associated with the ability to read the meniscus, only part of which is included in the process variability, the cubical coefficient of expansion for the prover under test, use of proper temperature corrections, the accuracy of temperature measurements, round robin data showing reproducibility, environmental variations over time, and bias or drift of the standard.

5.5 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

6.2 Report results as described in SOP 1, Preparation of Calibration/Test Results, with the addition of the following:

6.2.1 Volume, uncertainty, reference temperature, material, thermal coefficient of expansion (assumed or measured), construction, any identifying markings, tolerances (if appropriate), laboratory temperature, water temperature(s), barometric pressure, relative humidity, and out–of-tolerance conditions.