SOP 19

Standard Operating Procedure for Calibration of Large Neck-Type Metal Provers (Volume Transfer Method)[1]

1 Introduction

1.1 Purpose of Test

This procedure is used to calibrate large graduated neck type metal provers (40 L (10 gal) and larger) that are used in verification of petroleum, biodiesel, ethanol, milk, and/or water meters. It may also be used for the calibration of test measures (20 L (5 gal)) when temperature corrections are needed.

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 and compliance with environmental disposal requirements.

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.

1.2.4 Verify the availability of an adequate supply of clean water (GLP 10) (Note: this is critical when calibrating food-grade provers.)

1.2.5 Verify that the operator has had specific training and is proficient in SOP 19, SOP 17, GMP 3, 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 make possible the expected uncertainty achievable with this procedure:

[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.

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 uncontrolled environments, such as out-of-doors. Calibrations conducted under field and laboratory conditions are not considered equivalent.

1.3.2 The care required for field calibrations includes proper safety, 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.

2 Methodology

2.1 Scope, Precision, Accuracy

This procedure is applicable for the calibration of any size metal prover within the limitations of the standards available. The precision attainable will depend on strict adherence to the procedure, the care in volumetric adjustments, and the number of transfers, in the case of multiple transfers. The accuracy will depend on the standards used.

2.2 Summary

Water is delivered from a volumetric standard to the prover being calibrated. Depending on the respective volumes, multiple transfers may be required. While these should be minimized, a maximum number of 15 transfers are permitted to ensure that final uncertainties and systematic errors are sufficiently small for the intended applications. The temperature cannot be considered to be constant during multiple transfers; hence, the temperature of the water for each transfer must be measured. Because of the large volumes, the difference in thermal expansion of the respective vessels must be considered.

2.3 Equipment

2.3.1 Calibrated volumetric standard with recent calibration certificate traceable to NIST.

2.3.2 Calibrated 1 gal flask (or other suitable size) to calibrate neck of prover and a funnel.

2.3.3 Meniscus reading device (See GMP 3).

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

2.3.5 Timing device (calibration is not required.)

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

2.3.7 Sturdy platform, with appropriate safety conditions, with sufficient height to hold standard and to permit transfer of water from it to the prover by gravity flow.

2.3.8 Clean pipe or tubing (hoses) to facilitate transfer of water from the laboratory standard to prover. Pipe and hose lengths should be minimized to reduce water retention errors. Care must be taken during wet-downs and runs to ensure complete drainage and consistent retention in all hoses or pipes.

2.4 Procedure

2.4.1 Cleanliness verification

Fill and drain both standard and prover to be calibrated and check for visual evidence of soiling and of improper drainage. If necessary, clean with detergent and water (see GMP 6).

2.4.2 Neck scale plate verification

2.4.2.1 Fill the prover with water from the standard. Check the prover level condition in the same way in which it will be used and adjust if necessary. Check the prover system for leaks. This is a wet-down run.

2.4.2.2 Bleed the liquid level down to a graduation near the bottom of the upper neck. "Rock" the prover to "bounce" the liquid level, momentarily, to ensure that it has reached an equilibrium level.

Read and record this setting. This is in preparation for verification of the neck scale plate.

2.4.2.3 Recheck the scale reading, then add water from calibrated standards equal to $^{1}/_{4}$ or $^{1}/_{5}$ of the graduated neck volume and record the scale reading.

2.4.2.4 Repeat 2.4.2.3 by successive additions until water is near the top of the scale. Record scale readings after each addition. The closer the water is to the top of the neck, the harder it may be to "bounce" the liquid in the gauge.

2.4.2.5 A plot of scale readings with respect to volume added should be linear and will be a gross check of the validity of this calibration.

2.4.2.6 Calculate and check accuracy of the neck scale for each interval. The error should be less than 0.5 % of the graduated neck volume or $^{1}/_{4}$ of a graduation (whichever is smaller). If more than this, the scale should be replaced. Otherwise issue the NSCV and instructions to user.

2.4.2.7 The neck scale calibration value is calculated as follows:

$$NSCV = \frac{V_w}{(sr_f - sr_i)}$$

Table 2. Variables for neck scale verification value equation

NSCV	Neck scale calibration value
Vw	Total volume of water added to neck
sr_f	Scale reading, final
sr_i	Scale reading, initial

2.4.3 Body Calibration

2.4.3.1 Fill prover with water and level it. Drain water, then wait 30 s after cessation of full flow, before closing drain valve. This establishes a "wet-down" condition for provers with no bottom zero. If a bottom zero is present, follow the guidance provided in SOP 21 for LPG provers as follows: When the liquid reaches the top of the lower gage glass, close the valve and allow the water to drain from the interior of the prover into the lower neck for 30 s. Then bleed slowly with the bleed valve (4) until the bottom of the liquid meniscus reaches the zero graduation. (This step should be started during the 30 s drain period but should not be completed before the end of the drain period.) Alternatively, the prover may be completely drained with a 30 s drain time and then refilled with a funnel and small volume of water to set the zero mark (which will add to the prover calibration uncertainty due to variable retention characteristics).

2.4.3.2 Run 1. Fill the standard and measure and record the temperature.

2.4.3.2.1 Measure and record the temperature of the water, t_i, then adjust the standard prover to its reference mark or record the neck reading, and then discharge into the unknown prover. Wait 30 s after cessation of full flow to attain specified drainage, then close the delivery valve.

2.4.3.2.2 Repeat step 2.4.3.2.1. as many times as necessary (note the 15-drop limit) to fill the unknown prover to its nominal level. Level the prover if necessary and record the neck reading. Measure the temperature of the water in the prover, t_w, and record.

2.4.3.2.3 Perform the calculations described in section 3 to find the prover volume.

2.4.3.4 Adjust the scale as needed. If adjusted, record the adjusted prover gauge reading for determining the "as left" value for Run 1.

2.4.3.5 Run 2 - Repeat the process described in 2.4.3.2.

2.4.3.6 The test measure or prover must be capable of repeating to 0.02 % of the test volume during calibration. Repeatability problems may be due to a leak in the valves or seals of the prover, contamination or lack of cleanliness, air bubbles, inconsistent retention in delivery hoses, or poor field conditions such as when a calibration is conducted in an unstable environment. If excessive disagreement is found, clean the prover and take other corrective actions as necessary, then recalibrate until consecutive duplicate determinations agree within 0.02 % of the nominal volume. Repeatability problems must be corrected before the calibration can be completed.

2.4.3.7 Seal equipment as specified in laboratory policy.

3 Calculations

The following calculations assume that the standard was calibrated using a reference temperature of 60 °F *(15.56* °C*) and that you are calibrating a field standard to a* *reference temperature of 60* °F *(15.56* °C*). Equations for situations where different reference temperatures are involved will follow.*

3.1 Single Delivery

3.1.1 Calculate V_{X60}, the volume of the unknown prover at 60 °F, using the following equation:

$$V_{X60} = \frac{\rho_1 \left\{ \left(V_{S60} + \Delta_1 \right) \left[1 + \alpha \left(t_1 - 60\ ^\circ\mathrm{F} \right) \right] \right\}}{\rho_x \left[1 + \beta \left(t_x - 60\ ^\circ\mathrm{F} \right) \right]} \qquad \text{Eqn. 1}$$

3.2 Multiple Deliveries

3.2.1 Calculate V_{X60}, the volume of the unknown prover at 60 °F, using the following equation:

$$V_{X60} = \frac{\rho_1 \left\{ \left(V_{S60} + \Delta_1 \right) \left[1 + \alpha \left(t_1 - 60^\circ\mathrm{F} \right) \right] \right\} + \rho_2 \left\{ \left(V_{S60} + \Delta_2 \right) \left[1 + \alpha \left(t_2 - 60^\circ\mathrm{F} \right) \right] \right\} + \ldots + \rho_N \left\{ \left(V_{S60} + \Delta_N \right) \left[1 + \alpha \left(t_N - 60^\circ\mathrm{F} \right) \right] \right\}}{\rho_x \left[1 + \beta \left(t_x - 60^\circ\mathrm{F} \right) \right]} \qquad \text{Eqn. 2}$$

Table 3. Variables for V_{X60} equations

Symbols Used in Equations	
V_{X60}	volume of the unknown vessel at 60 °F
V_{S60}	volume of the standard vessel at 60 °F
$\rho_1, \rho_2, \ldots, \rho_N$	density of the water in the standard prover where ρ_1 is the density of the water for the first delivery, ρ_2 is the density of the water for the second delivery, and so on until all N deliveries are completed
$\Delta_1, \Delta_2, \ldots, \Delta_N$	volume difference between water level and the reference mark on the standard where the subscripts 1, 2,...,N, represent each delivery as above. If the water level is below the reference line, Δ is negative. If the water level is above the reference line, Δ is positive. If the water level is at the reference line, Δ is zero NOTE: units must match volume units for the standard
$t_1, t_2, \ldots, t_N$	temperature of water for each delivery with the subscripts as above
α	coefficient of cubical expansion for the standard in units / ºF
ß	coefficient of cubical expansion for the prover in units / ºF
t_x	temperature of the water in the filled unknown vessel in units ºF
ρ_x	density of the water in the unknown vessel in g/cm^3
Note: Values for the density of water at the respective temperatures may be found in Table 9.8 (in NISTIR 6969) or it may be calculated from the equation given in GLP 10.	

3.3 Prover Error/Correction or Deviation From Nominal

The total calculated volume of the prover at its reference temperature should be reported on the calibration report.

The prover volume for an open neck prover equals the V_{x60} value minus the gauge reading that is the difference from the nominal volume (with matched units).

$$Prover\ volume = V_{X\,60} - gauge\ reading \qquad \text{Eqn. 3}$$

$$Prover\ error = Prover\ volume - V_{Nom} \qquad \text{Eqn. 4}$$

$$Prover\ error = V_{X\,60} - gauge\ reading - V_{Nom} \qquad \text{Eqn. 5}$$

where:

V_{Nom} = Nominal Volume (taking care to match units)

V_{X60} is the calculated volume of water that should be observed in the prover. A positive prover error means that the prover is larger than nominal. A negative prover error means that the prover is smaller than nominal.

Example 1: If V_{X60} is 100.02 gal and gauge reading is 0.02 gal (above nominal);
then the prover volume at nominal is 100.00 gal;
and the prover error and correction are 0; and
no adjustment is needed.

Example 2: If V_{X60} is 100.02 gal and gauge reading is -0.02 gal (below nomival);
then the prover volume at nominal is 100.04 gal;
the prover error is + 0.04 gal; and
to adjust the prover, set the gauge to read 0.02 gal (the volume level will show a gauge reading of 0.02 gal, which is 4.62 in^3 or about 5 in^3, above nominal.)

3.4 Alternative Reference Temperatures

3.4.1 Reference temperatures other than 60 ºF (15.56 ºC) may occasionally be used. Common reference temperatures for other liquids follow:

Commodity	Reference Temperature
Frozen food labeled by volume (e.g., fruit juice)	-18 ºC (0 ºF)
Beer	3.9 ºC (39.1 ºF)
Food that must be kept refrigerated (e.g., milk)	4.4 ºC (40 ºF)
Distilled spirits or petroleum	15.56 ºC (60 ºF)
Petroleum (International Reference)	15 ºC (59 ºF)
Wine	20 ºC (68 ºF)
Unrefrigerated liquids (e.g., sold unchilled, like soft drinks)	20 ºC (68 ºF)
Petroleum (Hawaii)	26.67 ºC (80 ºF)

Equations for calculations when using alternative reference temperatures follow:

3.5 Single Delivery

3.5.1 Calculate V_{Xtref}, the volume of the unknown prover at its designated reference temperature (°F), using the following equation:

$$V_{Xtref} = \frac{\rho_1 \left\{ \left(V_{S\,tref} + \Delta_1 \right) \left[1 + \alpha \left(t_1 - \mathrm{t}_{\mathrm{ref\ S}} \right) \right] \right\}}{\rho_x \left[1 + \beta \left(t_x - \mathrm{t}_{\mathrm{ref\ X}} \right) \right]} \qquad \text{Eqn. 6}$$

3.6 Multiple Deliveries

3.5.1 Calculate V_{Xtref}, the volume of the unknown prover at its designated reference temperature, using the following equation:

$$V_{XtrefX} = \frac{\rho_1 \left\{ \left(V_{StrefS} + \Delta_1 \right) \left[1 + \alpha_1 \left(t_1 - t_{\mathrm{refS}} \right) \right] \right\} + \rho_2 \left\{ \left(V_{StrefS} + \Delta_2 \right) \left[1 + \alpha_2 \left(t_2 - t_{ref\,S} \right) \right] \right\} + \ldots + \rho_N \left\{ \left(V_{StrefS} + \Delta_N \right) \left[1 + \alpha_N \left(t_N - t_{ref\,S} \right) \right] \right\}}{\rho_x \left[1 + \beta \left(t_x - t_{ref\mathrm{X}} \right) \right]} \qquad \text{Eqn. 7}$$

Table 3A. Variables for V_{XtrefX} equations

Symbols Used in Equations	
V_{XtrefX}	volume of the unknown vessel, V_X at its designated reference temperature, $t_{ref\,X}$
V_{StrefS}	volume of the standard vessel, V_S at its designated reference temperature, $t_{ref\,S}$
$\rho_1, \rho_2, \ldots, \rho_N$	density of the water in the standard where ρ_1 is the density of the water for the first delivery, ρ_2 is the density of the water for the second delivery, and so on until all N deliveries are completed
$\Delta_1, \Delta_2, \ldots, \Delta_N$	volume difference between water level and the reference mark on the standard where the subscripts 1, 2,..., N, represent each delivery as above. If the water level is below the reference line, Δ is negative. If the water level is above the reference line, Δ is positive. If the water level is at the reference line, Δ is zero NOTE: units must match volume units for the standard
$t_1, t_2, \ldots, t_N$	temperature of water for each delivery with the subscripts as above
α	coefficient of cubical expansion for the standard in its designated units
ß	coefficient of cubical expansion for the prover in its designated units
t_x	temperature of the water in the filled unknown vessel in designated units
ρ_x	density of the water in the prover in g/cm^3
Note: Values for the density of water at the respective temperatures may be found in Table 9.8 (in NISTIR 6969) or it may be calculated from the equation given in GLP 10. Note: The cubical coefficient of the material used must match the unit assigned to the temperature measurement.	

4 Measurement Assurance

4.1. If a check standard is used (See SOP 20, SOP 30), repeat the process for the unknown artifact on the check standard, without adjustments.

4.2 Plot the check standard volume and verify it is within established limits. Alternatively a *t*-test may be incorporated to check the observed value against an accepted value.

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

4.4 Check standard values are used to calculate the standard deviation of the measurement process.

4.5 When a check standard is not used, a range chart may be used to monitor repeatability and estimate the standard deviation of the measurement process using the equation (see SOP 20 for details):

$$s_p = \frac{\overline{R}}{d_2^*} \qquad \text{Eqn. 11}$$

5 Assignment of Uncertainties

5.1 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*. See SOP 29 for the complete standard operating procedure for calculating the uncertainty when multiple deliveries or multiple standards are used.

5.3 Neck calibration uncertainty should be estimated based on the uncertainty of standards used, errors observed during calibration and the repeatability of the neck calibration.

5.4 The standard deviation of the measurement process from control chart performance (See SOP 20 and SOP 30).

5.4.1 The value for s_p is obtained from the control chart data of the check standard, or may be estimated using the range from the control chart, using large volume transfer procedures. Fifteen is the maximum recommended number of deliveries from a laboratory standard to a prover under test to minimize calibration uncertainties to the levels identified previously.

5.5 Other standard uncertainties usually included at this calibration level may 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, water density equation, uncertainties due to water viscosity, round robin data showing reproducibility, environmental variations over time, and bias or drift of the standard.

5.5.1 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, whether gravity drain is used during calibration or whether the volume of water is eliminated by pumping, the cleanliness of the prover and calibration medium, prover retention characteristics related to inside surface, contamination or corrosion, and total drain times, and possible air entrapment in the water, and connecting pipes. Systematic errors may be observed between laboratory calibration practices where a gravity drain is used and field use where the pumping system is used.

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 Total prover volume, uncertainty, reference temperature, material, thermal coefficient of expansion (assumed or measured), construction, any identifying markings, tolerances (if appropriate), laboratory temperature, water temperature, barometric pressure, relative humidity, out-of-tolerance conditions, and the total drain time from opening of the valve, including the 30 s drain after cessation of flow.

Additional References:

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, (2006).