SOP SVP[1]

Standard Operating Procedure
For
Gravimetric Calibration of Dynamic Small Volume Provers[2]

1 Introduction

1.1 Purpose of Test

This procedure describes the calibration of the "to deliver" volume of dynamic small volume provers (SVP) that may be used as volumetric measuring standards. The procedure uses gravimetric calibration principles to minimize calibration uncertainties. Accordingly, the procedure is especially useful for high accuracy calibrations. The procedure does not incorporate measurement control steps to ensure the validity of the standards and the measurement process; additional precautions must be taken. The procedure makes use of an electronic balance and is suitable for all sizes of gravimetric calibrations only limited by the capacity and resolution of the balance. The procedure can be used for both upstream and downstream calibrations. Each direction will be treated as a separate calibration.

1.2 Prerequisites

1.2.1 Verify that valid calibration certificates are available for the standards used in the test.

1.2.2 Verify that the standards and instruments (e.g., thermometer, barometer, and hygrometer) to be used have sufficiently small standard uncertainties for the level of calibration. Reference standards should not be used for gravimetric calibration.

1.2.3 Verify that the balance used is in good operating condition with sufficiently small process standard deviation as verified by a valid control chart or preliminary experiments to ascertain its performance quality when a new balance is put into service. The accuracy of the balance and weighing procedures should be evaluated to minimize potential bias in the measurement process.

1.2.4 Verify that the operator is experienced in precision weighing techniques and has had specific training in SOP 2, SOP 4, SOP 29, GMP 10, and gravimetric calibrations such as those used in SOP 14.

1.2.5 Verify that an adequate supply of distilled or de-ionized water (see GLP 10) is available.

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[1] Certain commercial equipment, instruments, or materials are identified in this publication in order to specify the experimental procedure adequately. Such identification is not intended to imply recommendation or endorsement by the National Institute of Standards and Technology, nor is it intended to imply that the materials or equipment identified are necessarily the best available for the purpose.
[2] 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.

1.2.6 Verify that the laboratory facility is free of air currents and/or temperature fluctuations where the SVP will be set up for testing.

1.2.7 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
SVP/ Large Volume Gravimetric	20 °C to 23 °C, a set point ± 2 °C, maximum change 1.0 °C/h	40 % to 60 % ± 10 % maximum change / 4 h

1.2.8 All necessary maintenance to the test artifact 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. Replace the seals on the prover if there is any doubt as to their integrity. If “as found” data is required, that data should be obtained before any seal replacements.

1.2.9 The prover inlet and outlet must be blocked by the device owner before calibration by using a blind flange or double-block and bleed valve.

1.2.10 The device owner must provide all necessary manufacturers’ manuals as well as trained personnel to operate the device.

2 Methodology

2.1 Scope, Precision, Accuracy

The procedure is applicable for the calibration of any size of measuring container SVP that, when filled with water, will not overload the electronic balance used. Typical containers SVPs range in capacity from 15 gal to 120 gal. The precision of calibration will depend on strict adherence to the various steps of the procedure. The accuracy attainable will depend on the uncertainties of the standard weights, the air buoyancy, density of water, thermal expansion corrections that are made, and the repeatability of the SVP.

2.2 Summary

The mass of the water delivered by the SVP is determined by weighing the water on an electronic balance and comparing it to mass standards. Volume is derived from mass using the equations provided.

2.3 Equipment and Standards

2.3.1 Electronic balance having sufficient capacity to weigh the loaded vessel. The sensitivity of the balance will be a limiting factor in the accuracy of the measurement. The resolution and repeatability should be smaller than the accepted uncertainty of the calibration.

2.3.2 Calibrated mass standards with adequate accuracy and NIST traceable mass values. Ordinarily, weights of ASTM Class 2 or 3 specifications, or equivalents are required.

2.3.3 Thermometer, calibrated to ± 0.1 °C for measuring the temperature of the water, prover, and detector bar; ± 0.5 °C for measuring the temperature of the air and calculating the air density, traceable to NIST.

2.3.4 Barometer, calibrated to ± 1 mm Hg and a hygrometer, calibrated to ± 5 % for calculating the air density, traceable to NIST.

2.3.5 Pressure gauge for determining the prover pressure, with calibration traceable to NIST. (Include the uncertainty of the pressure gauge in the uncertainty calculations.)

2.3.6 Distilled or de-ionized water and methods to verify its purity (See GLP No. 10).

2.4 Procedure

2.4.1 Install SVP volumetric test kit per manufacturer's instructions. (Provided by device owner.)

2.4.2 Connect water supply to prover.

2.4.2.1 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. Set device valves as appropriate for the test to be conducted.

2.4.2.2 For Calibron (brand) SVPs, turn water supply on, and open appropriate valves to eliminate entrapped air according to manufacturer's instructions. After all air is bled off, close associated valves. Then open the appropriate valves and allow water to circulate until the temperature has stabilized.

2.4.2.3 For Daniels (brand) SVPs, establish valve position for control of upstream or downstream flow.

2.4.3 Install certified thermometers to record air temperature, water temperature, detector bar temperature (T_d), and prover temperature (T_p).

2.4.4 Install calibrated pressure gauge to record prover pressure (P_p).

2.4.5 Connect prover control cable to controller (from the volumetric test kit.)

2.4.6 Apply power to the prover and the water draw circuit. One of the valves will switch on immediately. Note which valve switches on, and mark it as the overboard or bypass valve. The other valve is the draw valve and will only be on while the flag is between the two volume switches.

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

Note: All references in the following weighing options to the empty receiving vessel assume that the empty receiving vessel has been placed in a "wet-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.

2.4.8 Weighings (Option A)

2.4.8.1 Zero the balance and record the balance indication as O_1. Place a standard mass, M_{s1}, on the balance platform (M_{s1} should be slightly larger than the mass of the empty receiving vessel.) Record the balance indication as O_2.

2.4.8.2 Place the empty receiving vessel on balance platform and record the balance indication as O_3. Caution: the receiving vessel must be dry on the outside for all weighings.

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

2.4.8.4 Zero the balance and record the balance indication as O_4.

2.4.8.5 Manipulate the system to cause the prover piston to move to the starting position and begin the water draw sequence. Record all pertinent test artifact temperatures and pressures during the water draw sequence. Immediately upon completion of the water draw sequence, place the filled receiving vessel on the balance and record the balance indication as O_6. Read and record the temperature of the water in the container immediately after weighing.

2.4.8.6 Remove the filled receiving vessel and 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_5.

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

2.4.8.8 Make five duplicate runs with 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 repeat within 0.02 %.

2.4.9 Weighings (Option B)

2.4.9.1 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.

2.4.9.2 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.

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

2.4.9.4 Place the empty receiving 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).

2.4.9.5 Manipulate the system to cause the prover piston to move to the starting position and begin the water draw sequence filling the receiving vessel. Read and record all pertinent test artifact temperatures and pressures during the water draw sequence. Place the lid on the receiving vessel to limit evaporation.

2.4.9.6 Immediately prior to weighing full vessel record balance reading as (d_1). This reading will be used to calculate drift while filling the vessel.

2.4.9.7 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.

2.4.9.8 Remove the filled receiving vessel and record the balance indication as (d_2). This reading will be used to calculate drift over the entire process.

2.4.9.9 Immediately after removing the filled receiving vessel from the balance, record the temperature of water in the vessel.

2.4.9.10 Make five duplicate runs with 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 repeat within 0.02 %.

3 Calculations

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

(Option A)

$$V_T = \left[O_{6(\,filled\,)} \frac{M_{s2}\left(1 - \dfrac{\rho_a}{\rho_{s2}}\right)}{\left(O_5 - O_4\right)} - O_{3(\,drained\,)} \frac{M_{s1}\left(1 - \dfrac{\rho_a}{\rho_{s1}}\right)}{\left(O_2 - O_1\right)} \right] \left(\frac{1}{\rho_w - \rho_a} \right) \qquad \text{Eqn. 1}$$

Table 2. Variables for volume equation

Variable	Description
M_s, M_{s1}, M_{s2}	mass of standards
ρ_s	density of M_S
ρ_w	density of water at the temperature of measurement
ρ_a	density of air at the conditions of calibration
V_t	Volume at the temperature of the test

(Option B)

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

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

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

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

$$V_{60} = \frac{Vw}{CCF} \qquad \text{Eqn. 2}$$

where

$$CPL_p = \frac{1}{\left[1 - \left((0.0000032 * P_p\right)\right]}$$

$$CTS_p = \left\{\left(1 + \left[\left(T_p - T_b\right) * G_a\right]\right) * \left(1 + \left[\left(T_d - T_b\right) * G_l\right]\right)\right\}$$

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

$$CCF = CPL_p * CTS_p * CPS_p$$

Table 4. Additional Variables for Volume Equations

Variable	Description
T_b	Reference (Base) temperature
T_d	Detector bar temperature (Temperature, detector)
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 expansion (switchbar)
G_a	Coefficient of flow tube area thermal expansion
CPL	Correction for the pressure on the liquid
CTS	Correction for the temperature on the standard prover
CPS	Correction for the pressure on the standard prover
CCF	Combined correction factor

3.3 Calculate water density using the equation provided in GLP 10.

3.4 Calculate the air density per SOP 2.

3.5 Calculate the uncertainty for the calibration using Appendix B:

4 Measurement Assurance

4.1 Record and plot standard deviations of each five run calibration to determine short-term variations of the measurement process.

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, 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 Standard deviation of the measurement process from control chart performance (See SOP 17 or 20.)

The value for s_p is obtained from the control chart data or estimated based on the range of duplicate measurements over time. This value will incorporate a repeatability factor related to the precision of the weighings.

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, and standard uncertainties associated with the density of the standards used or the lack of internal cleanliness.

6 References

6.1 Manual of Petroleum Measurement Standards; Chapter 11.2.3 - Water Calibration of Volumetric Provers; First Edition, August 1984.

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	Area Thermal Expansion Coefficient (Ga)	
Reference Temperature(Tb) (^{o}F)	60	Detector Thermal Expansion Coefficient (GI)	
Reference Temperature (^{o}C)	15.55555556	Modulus of Elasticity (flow tube) (E)	
Reference Pressure (psig)	0	Flow Tube Inside Diameter (inches) (ID)	
		Flow Tube Wall Thickness (inches) (WT)	

	Standards Used	
	Full Vessel	Empty Vessel
Total True Mass		

Observations

Fill Number	Fill # 1	Fill # 2	Fill # 3	Fill # 4	Fill # 5
Flow Rate	Fast	Fast	Fast	Slow	Slow
Run Time (sec)					
Zero Balance, O_1					
Balance Reading O_2 (Standards Empty)					
Balance Reading O_3 (Empty Vessel)					
Zero Balance, O_4					
Balance Reading O_5 (Standards Full)					
Balance Reading O_6 (Full Vessel)					
Air Temperature (^{o}C)					
Barometric Pressure (mmHg)					
Relative Humidity (% RH)					
Water Temperature (^{o}C)					
Detector Bar Temperature (T_d) (^{o}C)					
Prover Temperature (T_p) (^{o}C)					
Prover pressure (P_p) (psig)					

Example uncertainty components listed in order of effect on total uncertainty:

- Standard deviation of the measurement process, s_p
- Water temperature
- Reproducibility
- Mass calibration uncertainty
- Water density
- Prover temperature
- Water compressibility
- Detector temperature
- Prover pressure
- Area thermal expansion (Ga)
- Air temperature
- Detector thermal expansion (Gl)
- Barometric pressure
- Relative humidity
- Modulus of elasticity
- Flow tube diameter (ID)
- Tube wall thickness (WT)