SOP 21

**Standard Operating Procedure for
Calibration of LPG Provers[1]**

1   Introduction

1.1   Purpose of Test

This procedure may be used to calibrate a volume standard used to test systems designed to measure and deliver liquefied petroleum gas (LPG) in the liquid state by definite volume, whether installed in a permanent location or mounted on a vehicle. A schematic diagram of such a prover is shown in Figure 1, together with numbers, e.g., 1, 2, 3, and 4 to clarify the various operations described in the procedure. The parts labeled A, B, and C are hose connections used in meter testing (versus prover calibrations). The prover being calibrated should be evaluated for conformance to appropriate specifications (using the checklist provided in NIST Handbook 105-4, Specifications and Tolerances for Liquefied Petroleum Gas and Anhydrous Ammonia Liquid Volumetric Provers, 2010) if being used for legal weights and measures applications. You should also review the schematic provided in Handbook 105-4.

1.2   Prerequisites

1.2.1   Verify the unknown prover has been properly cleaned and vented with all petroleum products removed prior to submission for calibration to ensure laboratory safety. The prover and/or pressure gauge may be visually inspected to determine that residual products are not present. Smell is not necessarily an adequate indicator of cleanliness.

NOTES: Many laboratories have a policy regarding cleanliness of submitted volumetric standards to minimize water contamination with flammable petroleum products. Be sure to follow laboratory safety policies for dealing with pressurized vessels.

1.2.2   Verify that valid 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 (for example, NIST.)

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[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. The majority of LPG provers in use are 20 gal, 25 gal, and 100 gal nominal sizes. The volume of LPG provers is established at 60 ºF and 100 psig.

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

1.2.4 Verify the availability of an adequate supply of clean, preferably soft water (filtered and thermally equilibrated as appropriate) water (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.

1.2.5 Verify that the operator has had specific training in SOP 19, SOP 20, SOP 21, and GMP 3 and is familiar with the operating characteristics and conditioning of the standards and unknown test items being used and calibrated.

1.2.6 Cylinder of nitrogen or compressed air, or a regulated air source of at least 200 psig, and a proper pressure regulator.

1.2.7 Verify that the laboratory facilities meet the following minimum conditions to make possible 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 / 1 h	35 % to 65 % Stable to ± 20 % / 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.

1.3.2 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 during the measurements. 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 LPG provers with capacities of 100 L to 500 L (20 gal to 100 gal) or larger when appropriate. Provers of 20 gal, 25 gal, and 100 gal (with gal and in^3 units) are encountered most frequently, hence the procedure is written with that in mind. The changes necessary for testing provers of other capacities will be obvious and are not described in this document. The agreement of duplicate measurements made within a short period of time on a given 100 gal LPG prover must be within 5 in^3 (0.02 gal). For any nominal volume, replicate values must agree within 0.02 % of the volume. Where the demonstrated standard deviation of the process is less than 0.02 % of the volume, replicate values must agree to within the limits on the standard deviation or range charts. The accuracy will depend on the uncertainty in the volume of the standard, on the care exercised in making the various measurements and temperature readings, and on correct application of the corresponding corrections.

   2.2 Summary

      The procedure is a modification of one described by M.W. Jensen in NBS Handbook 99, "Examination of Liquefied Petroleum Gas Liquid- Measuring Devices." The LPG prover is calibrated with a known volume of water delivered into it from a standard prover of calibrated volume. Depending on the respective volumes, multiple transfers may be required. While multiple transfers should be minimized, a maximum number of 15 transfers are permitted to ensure that final calibration uncertainties are sufficiently small to meet user applications. The temperature of the calibration medium (water) cannot be considered to be constant during 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. The LPG prover is pressurized and the liquid level is measured at each of several values of applied pressure. The calibration thus defines the capacity of the prover over its expected range of operational pressures.

   2.3   Standards and Equipment

      2.3.1   Calibrated standard prover of minimum volume of 5 gal for 20 gal and 25 gal LPG provers is acceptable. A 10 gal standard is acceptable for calibrating a 100 gal LPG prover, but a standard that is of the same volume as the LPG prover is preferable. NOTE: standard provers used for calibration may need to have an alternative calibration value based on restricted flow delivery as the opening for many LPG provers may not be adequate to receive the full flow delivery from the reference standard.

2.3.2   A funnel for transferring water from calibrated flasks into the unknown prover.

2.3.3   Calibrated flasks of suitable sizes to calibrate the neck of prover.

2.3.4   Thermometers (2) with resolution and uncertainty less than 0.1 °C.

2.3.5   Meniscus reading device (See GMP 3).

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

2.3.7   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 the pipe or tubing (hoses) to facilitate transfer of water from the laboratory standard to prover. Nearly all LPG provers require reducers to be used between normal laboratory piping and the top hole on the prover. Pipe and hose lengths must 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. Arrange all hoses so that there are no loops or low spots that can hold water.

2.3.9   Compressed nitrogen cylinder or air, suitable regulator, and an appropriate pressure gauge. The calibration relies on the accuracy of the pressure gauge on the prover. It assumes that systematic errors in the prover pressure gauge will be present in field applications as well, thus calibration of the laboratory pressure gauge is *not* essential. However, the prover must be recalibrated if pressure gauges are changed in the field. When a laboratory pressure gauge is used during the calibration, the unknown pressure gauge and laboratory gauge must both have current calibration reports.

2.4   Procedure

2.4.1   Preliminary Operations

2.4.1.1 Install and level the standard(s) on a raised platform with appropriate security and safety ensured for the prover(s) and operator(s). Provide pipe or tubing for delivery of water by most direct route to prover.

2.4.1.2 Position and level the unknown prover where it can be reached from the elevated standard by the shortest feasible delivery system.

2.4.1.3 It is recommended that all valves, hoses, and piping be removed from ports below the nominal volume line to eliminate errors that may result from leakage or expansion while the prover is under pressure. These potential errors typically only affect the calibration but not during normal use. Plug one port with a fitting and valve of suitable pressure rating for draining the prover. Plug all other ports with sealed plugs of suitable pressure ratings.

2.4.1.4 Remove the plug and relief valve (1) from the top, and extend the pipe into the hole. This may require the use of a reducer and a short length of hose (about 1 inch in diameter). If this is a tight fit, open the vapor return line valve (2) to provide an air bleed.

Use the prover inlet line (3) as a gravity drain. If necessary, remove the fitting on the end and connect a hose or pipe to make the necessary drain line.

Warning: ensure that a check valve is not plumbed into the prover inlet line. If it is, remove the check valve, otherwise the prover will need to be drained via the plug in the bottom of the lower neck.

2.4.2 Cleanliness Check

Both the standard and the unknown prover must be internally clean. This should be verified by checking that water drains properly from them. If necessary, either or both should be cleaned with water and non-foaming detergent (see GMP No. 6) to attain good drainage characteristics. Additional effort may be required to eliminate scaling and contamination build-up from the inside of LPG provers.

2.4.3 Neck scale plate verification (This is generally only conducted for new or damaged measures or those that have not been calibrated by the laboratory in the past.)

2.4.3.1 Fill the unknown 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 by placing a precision spirit or electronic digital level vertically on the neck on at least two locations, 90 degrees apart around the circumference of the neck and adjust the orientation of the standard until the neck is as close to vertical (plumb or perpendicular to the horizontal plane) as possible. Verify and adjust any mounted levels that are on the prover to agree (when present and when possible). Check the prover system for leaks. This is a wet-down run.

2.4.3.2 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 to be used as the initial scale reading sr_i. This is in preparation for calibration of the neck scale.

2.4.3.3 Remove the fill hose or pipe from the top and insert a funnel.

2.4.3.4 Recheck the scale reading, then add water in a quantity equal to approximately ¼ or $^1/_5$ of the graduated neck volume from a suitable standard; record the scale reading.

2.4.3.5 Repeat 2.4.3.4 by successive additions until water is near the top of the scale (the neck capacity is usually about 5 gal on a 100 gal LPG prover). Record scale readings after each addition. The last reading will be the final scale reading, sr_f. The closer the water is to the top of the neck, the harder it may be to "bounce" the liquid in the gauge.

A plot of scale readings with respect to the total volume of water that is added V_w should be linear and will be a gross check of the validity of this calibration.

2.4.3.6 Calculate and assess the accuracy of the neck scale for each interval. The maximum capacity tolerance between the nominal volume line and any other line on the scale shall be less than two (2) major scale divisions as listed in Handbook 105-4. If more than this, the scale should be replaced. Alternatively, a Neck Scale Correction Value (NSCV) may be issued with instructions to the user if it is anticipated that this correction value will be used.

2.4.3.7 Neck scale errors less than the limits provided in NIST Handbook 105-4 should be included in the uncertainty estimate associated with the prover calibration. Uncertainties associated with the neck scale calibration are primarily related to the setting of the meniscus on the flask used (with each drop), the reading of the meniscus on the unknown prover (at each volume reading), and the calibration uncertainty of the flask. These uncertainties must be considered when evaluating the acceptability of the neck scale.

2.4.3.8 The neck scale correction value is calculated as follows:

$$NSCV = \frac{V_w}{\left(sr_f - sr_i\right)} \qquad \text{Eqn. 1}$$

Table 2.   Variables for neck scale correction value equation.

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

2.4.4   Body Calibration

2.4.4.1 Drain the unknown LPG prover through its inlet valve and the liquid bleeder valve. When the liquid reaches the top of the lower gauge glass, close the inlet valve and allow the water to drain from the interior of the prover into the lower neck for 30 s, while controlling the water flow and level with the bleeder valve until the liquid meniscus reaches the zero graduation. The liquid level should be exactly at the zero graduation and the bleeder valve closed simultaneously at the 30 s drain time. (Draining with the bleeder valve close to the zero mark should be started during the 30 s drain period but should not be completed before the end of the drain period.)

Alternatively, though not recommended, the prover may be completely drained with a 30 s drain time and then refilled with a funnel that has been wet down, and adding a small volume of water to set the zero mark. Errors from this process will result due to the additional drain and retention time and other factors associated with the process and should be considered in the uncertainty assessment.

2.4.4.2 Run 1. Transfer the volume from the standard in the usual manner, and record the standard and prover temperature readings. If multiple transfers are required, record temperature of the standard at the time of each transfer, but that of the prover only after the final transfer. Check the prover level by placing a precision spirit or electronic digital level vertically on the neck on at least two locations, 90 degrees apart around the circumference of the neck and adjust the orientation of the standard until the neck is as close to vertical (plumb or perpendicular to the horizontal plane) as possible. Verify and adjust any mounted levels that are on the prover to agree (when present and when possible). "Rock" the prover to "bounce" the liquid in the upper gauge glass before reading. Record the final scale reading after the nominal volume has been transferred into the unknown LPG prover with no pressure applied.

Note regarding temperature measurements: A digital temperature sensing device with a long cable can allow insertion of the probe into the standard and the unknown to enable direct liquid temperature measurements at the bottom, middle, and top of the provers. If the prover thermometer wells are used, ensure that the prover has equalized with the temperature of the water.

2.4.4.3 Run 2. Drain the LPG prover as described in 2.4.4.1 and make another test run. Record the temperatures of the standard(s) and the unknown and final scale reading. Calculate the prover error at 0 psig (no pressure applied) for each run using the appropriate equations in Section 3; these values are used to evaluate repeatability of the test only. The error at 0 psig for the test runs must agree within 0.02 % of the prover volume or approximately one-half the prover tolerance (i.e., 5 in^3 on a 100 gal LPG prover). If the two runs fail to agree within 0.02 % of the prover volume, or the limits on the standard deviation or range charts (whichever is smaller), identify and correct problems with the prover or set-up. Then, continue until replicates agree within these limits, taking care to ensure that poor cleanliness, prover condition, contamination, bubbles in hoses, leaking valves or seals are not contributing to poor repeatability. Lack of measurement agreement may also be due to poor field conditions, such as when calibration is conducted in unstable environments. Poor agreement must be corrected before calibration can be completed.

2.4.4.4 While the LPG prover remains full from Run 2, replace the relief valve and plug in the top of the prover using suitable pipe joint compound or tape.

2.4.5 Prover Adjustments

2.4.5.1 The internal pressure and hence the volume of the prover may vary during use. Accordingly, a pressure correction must be made using the data of steps 2.4.6.

To minimize the amount of correction needed when the prover is in use, the prover should be adjusted to indicate its nominal capacity when 100 psig is applied. (An internal pressure of 100 psig is suggested as being convenient.) If the actual volume of the prover is not near a convenient whole gallon value and cannot easily be adjusted to a whole gallon value, a prover correction value can be computed (see 3.3) and added to the pressure correction values to obtain a set of combined prover and pressure correction values to be computed. The pressure correction is computed in 3.3. The pressure gauge that is mounted to the LPG

prover may be used for setting calibration pressures, but, if following NIST Handbook 105-4, it is required to be calibrated. Since the correction values assigned to the prover during calibration will match those during use, the prover corrections obtained using a mounted pressure gauge are generally acceptable. Calibration status of the pressure gauge mounted on the prover should be assessed and included on the calibration report.

2.4.5.2   Use a cylinder of nitrogen or compressed air and a proper pressure regulator with an integrated pressure gauge.

Connect the cylinder regulator output to the vapor return fitting (2) near the top of the neck. This may require fashioning a connection with steel pipe nipples or other appropriate materials and the existing fittings.

Caution: Ensure that all piping and fittings are rated for the pressures to which they will be exposed.

Make sure all valves are closed except the vapor return valve. Verify that the final scale reading has not changed since it was recorded (if it has changed it may signal a leak in one of the valves or fittings), and then slowly introduce pressure until the installed prover gauge reads 100 psig. Lightly tap the gauge to ensure that the gauge needle is not sticking. "Bounce" the liquid in the neck, then read and record the liquid level at this applied pressure.

Return pressure to 0 psig and reapply pressure as above. This reading at 100 psig should agree with the above recorded reading within 0.02 % of the nominal volume of the prover. Leaking seals or valves may cause problems with repeatability of the gauge readings under pressure.

2.4.5.3   With the pressure in the prover at 100 psig, adjust the upper scale to read the nominal volume. This is accomplished by adjusting the upper scale so that the water level reading is:

$$Desired\ scale\ reading = \frac{V_{60}}{1.00032} - V_{NOM} \qquad\qquad Eqn.\ 2$$

Take care to use like units in the calculation. The calculation for V_{X60} is given in 3.1, Eqn. 5 or Eqn. 6.

Note: The correction factor 1.00032 corrects for the compressibility of the water at 60 °F and 100 psig. If the upper scale is not adjustable, see 2.4.5.4.

2.4.5.4 For provers with only an adjustable lower scale (or one in which the upper scale is not adjustable), a prover correction, L_C, may be calculated at 100 psig as follows:

$$L_c = \frac{V_{60}}{1.00032} - V_{NOM} - sr_u \qquad \text{Eqn. 3}$$

where:

L_C	Prover correction at 100 psig
V_{X60}	Volume of unknown prover at 60 °F
V_{NOM}	Nominal volume of prover
sr_u	Upper scale reading at 100 psig
1.00032	Correction factor for the compressibility of water at 100 psig

Take care to use like units in the calculation.

If the prover correction is negative, move the bottom scale down to increase the prover volume. If the prover correction is positive, move the bottom scale up to decrease the prover volume. The distance h that the bottom scale is to be moved is:

$$h = \frac{4 \left| L_C \right|}{\pi \, d^2} \qquad \text{Eqn. 4}$$

where:

h	Distance in inches the bottom scale is to be moved, up or down
L_C	Prover correction at 100 psig in cubic inches
d	Inside diameter of the lower neck of the prover in inches (as noted on identification plate)

2.4.6 Pressure Correction

2.4.6.1 Return pressure to 0 psig. Record the reading. Slowly introduce pressure until the installed prover gauge reads 50 psig. Lightly tap the gauge to ensure that the gauge needle is not sticking. "Bounce" the liquid in the neck, then read and record the liquid level at this applied pressure.

2.4.6.2 Repeat step 2.4.6.1 at 100, 150, and 200 psig. Other pressure points in between those listed may be tested if so desired. (The water level should decrease 10 in^3 to 15 in^3 for each 50 psig increase in pressure on a 100 gal prover, although this varies depending on the geometry and age of the LPG prover. Water level changes significantly greater than indicated may be due to leaking seals and/or valves.) Erratic pressure readings may also be due to air entrapment based on prover design; repeated pressurizing of the prover should eliminate entrapped air. Air entrapment problems due to design may need to be investigated and corrected.

2.4.6.3 Repeat step 2.4.6.1 as the pressure is bled down to 150, 100, 50, and 0 psig (atmospheric pressure). The readings must agree with those previously obtained within approximately 0.02 % of the nominal prover volume. If the data are not linear with respect to pressure, repeat the series of measurements above to verify the nonlinearity of the readings. Leaking seals or valves may cause problems with repeatability of the gauge readings under pressure. The cause of poor agreement must be identified and corrected before continuing the calibration.

2.4.7 Final Operations

2.4.7.1 Seal the bottom and top scales as specified by laboratory policy and as appropriate.

2.4.7.2 Drain prover, then remove plug (5) at the lower neck to facilitate drainage below the lower gauge. If time permits, let the prover drain overnight.

2.4.7.3 With the nitrogen cylinder or compressed air connected, blow nitrogen or air through the prover to remove remaining moisture. Be sure to blow out the drain line and any other portions of the system that may have become contaminated with water.

2.4.7.4 If water has entered the pump-off system, pour several gallons of alcohol into the prover and pump the alcohol through the system to remove the water to prevent it from freezing in the pump when LP gas is used.

3 Calculations

3.1 Single Delivery

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

$$V_{X60} = \frac{\rho_1 \left\{ \left(V_{S60} + \Delta_1 \right) \left[1 + \alpha \left(t_1 - 60 \, ^\circ F \right) \right] \right\}}{\rho_x \left[1 + \beta \left(t_x - 60 \, ^\circ F \right) \right]} \quad \text{Eqn. 5}$$

3.2 Multiple Deliveries

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

$$V_{X60} = \frac{\rho_1 \left\{ \left(V_{S60} + \Delta_1 \right) \left[1 + \alpha \left(t_1 - 60 ^\circ F \right) \right] \right\} + \rho_2 \left\{ \left(V_{S60} + \Delta_2 \right) \left[1 + \alpha \left(t_2 - 60 ^\circ F \right) \right] \right\} + \ldots + \rho_N \left\{ \left(V_{S60} + \Delta_N \right) \left[1 + \alpha \left(t_N - 60 ^\circ F \right) \right] \right\}}{\rho_x \left[1 + \beta \left(t_x - 60 ^\circ F \right) \right]} \quad \text{Eqn. 6}$$

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. For slicker-plate type standards, the Δ is zero.
$t_1, t_2, \ldots, t_N$	temperature of water for each delivery with the subscripts as above
α	coefficient of cubical expansion for the standard in units / ºF
β	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 calculated using the equations given in GLP 10 for air-saturated water.	

3.3 Pressure Corrections

Compute the pressure correction, P$_{corr}$, at each pressure that the prover was read, after any adjustments, by correcting for the compressibility of the water.

The equation is:

$$P_{corr} = Scale\ reading\ @\ 100\ psig - scale\ reading\ @\ X\ psig + \left(water\ volume\ correction\ value\right)\left(\frac{100\ psig - X\ psig}{100}\right)$$

Eqn. 7

where the water compressibility factor due to compressibility at 60 °F and 100 psig is 0.00032 gal/gal.

Nominal Prover Volume	Water volume correction value due to compressibility at 60 °F and 100 psig	
	in^3	gal
20 gal	1.5	0.0064
25 gal	1.8	0.0080
100 gal	7.4	0.032

The compressed water volume correction value is provided in both cubic inches and gallons so the proper unit can be selected depending upon the unit used for the scale readings.

Plot the pressure corrections. If the corrections versus the pressure are linear, make a straight line best fit of the data and interpolate to obtain the pressure corrections for any desired pressure. If the data is nonlinear, then perform a straight line interpolation between adjacent pressure readings to obtain pressure corrections at any desired intermediate pressures. Alternatively, a best fit curve can be drawn for the nonlinear data and the pressure corrections interpolated from the graph for intermediate pressures.

3.4   Prover Volume

LPG provers are generally adjusted to the nominal value using 100 psig as the reference pressure and a 60 °F reference temperature. A gauge reading on a prover that indicates a nominal volume at 0 psig will be lower when pressurized to 100 psig due to both the compressibility of water under pressure and the expansion of the prover. The expansion of the prover is consistent whether there is water in the prover or LPG. However, a correction must be made for the compressibility of water as noted in 3.3. Thus, to adjust a 100 gal prover to its nominal volume at 100 psig, the gauge reading should be set to the V_{X60} value minus 7.4 in^3. (The water level on the prover's upper scale will be 7.4 in^3 less than the calculated V_{X60} value when pressurized to 100 psig.) Adjusting the scale plate while the prover is under pressure at 100 psig to indicate the corrected water level will correct for the compressibility of water and set the correct prover volume.

Another possible way to handle the compressibility of water is to add the volume of water (e.g., 7.4 in^3 for a 100 gal prover) to the volume of water in the prover so that it can be set to the V_{X60} value at 100 psig.

The calculated mean volume of the prover at its reference temperature and pressures at 0 psig, 50 psig, 100 psig, and 150 psig and 200 psig, are reported on the calibration certificate.

4   Measurement Assurance

4.1   Duplicate the process with a suitable check standard or have a suitable range of check standards for the laboratory. 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 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 is used as the estimate of variability in the measurement process. Note: the pooled or average standard deviation over time will reflect varying conditions of LPG provers that are submitted to the laboratory but do not reflect potential meniscus reading errors (See GMP 3.)

For unknown standards that are adjusted, do not combine an "as found" value with an "as left" value for the two runs entered into 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.

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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, and the additional items noted below and in the uncertainty budget table, Table 4, at the 95 % level of confidence. See SOP 29 for the complete standard operating procedure for calculating the uncertainty.

5.1.1   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*.

Note: See SOP 29 for the complete standard operating procedure for calculating the uncertainty when multiple deliveries or multiple standards

are used to correctly calculate correlated uncertainties. 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.

5.2   The standard deviation of the measurement process s_p, is taken from a control chart for an LPG check standard (not from charts for a refined fuel check standard) or standard deviation charts for LPG calibrations (See SOP 17, SOP 20 and SOP 30). Check standards are not normally available.

5.3   Neck calibration uncertainty should be estimated based on the uncertainty of standards used, errors observed during calibration, ability to read the meniscus of all standards involved (see GMP 3), and the repeatability of the neck calibration.

5.4   Other standard uncertainties usually included at this calibration level are 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) uncertainties associated with temperature corrections that include values for the cubical coefficient of expansion for the prover under test, the accuracy and gradients associated with temperature measurements in the provers. Additional factors that might be included are: data showing reproducibility, environmental variations over time, and bias or drift of the standard as noted in control charts. Factors that are usually insignificant are uncertainties associated with viscosity of the water as a calibration medium and uncertainties associated with the compressibility of water.

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, 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. Systematic errors may be observed between laboratory calibration practices where a gravity drain is used and field use where the pumping system is used.

5.6   Example components to be considered for an uncertainty budget table are shown in Table 4. Multiple values of some items may need to be considered (e.g., multiple drops from the standard, multiple meniscus readings, and multiple temperature readings.)

Table 4. Example Uncertainty Budget Table.

Uncertainty Component Description	Symbol	Source	Typical Distribution
Uncertainty of the standard (5.1)	u_s	Calibration report; may be multiplied or added based on dependencies	Rectangular or Normal divided by coverage factor
Accepted standard deviation of the process (5.2)	s_p	Control chart, standard deviation chart	Normal
Uncertainty or uncorrected error associated with a neck calibration (5.3)	u_n	From experimental data	Rectangular
Ability to read the Meniscus in S (5.4)	u_m	None if using a slicker-plate type standard; GMP 3	Triangular
Ability to read the Meniscus in X (5.4)	u_m	GMP 3	Triangular
Water temperature (S) (5.4)	u_{ts}	Consider accuracy, resolution, and gradients	Rectangular
Water temperature (X) (5.4)	u_{tx}	Consider accuracy, resolution, and gradients	Rectangular
Cubical Coefficient of Expansion on S (5.4)	u_{CCE}	5 % to 10 % (EURAMET CG-21)	Rectangular
Cubical Coefficient of Expansion on X (5.4)	u_{CCE}	5 % to 10 % (EURAMET CG-21)	Rectangular
Uncertainty associated with the pressure gauge	u_{psig}	Handbook 105-4, 5 psig increments; use 2.5 psig	Rectangular
Uncertainty of bias or drift of standards (5.2)	u_b	From control chart	Rectangular
Uncertainty of drain time	u_d	From experimental data	Normal

6   Calibration Certificate

    6.1   Report results as described in SOP No. 1, Preparation of Calibration Certificates, with the addition of the following:

        For LPG provers, the mean prover volume and uncertainty, reference temperature, material, coefficient of expansion (assumed or measured), 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.

        The report should also include temperature and pressure correction tables or chart, along with a note regarding possible differences in retention characteristics between water, the calibration medium, and LPG products.

The SI unit of volume is m^3, so a conversion factor is to be included on the report in the notes section when other volumes are used.

Figure 1. LPG Prover Schematic.
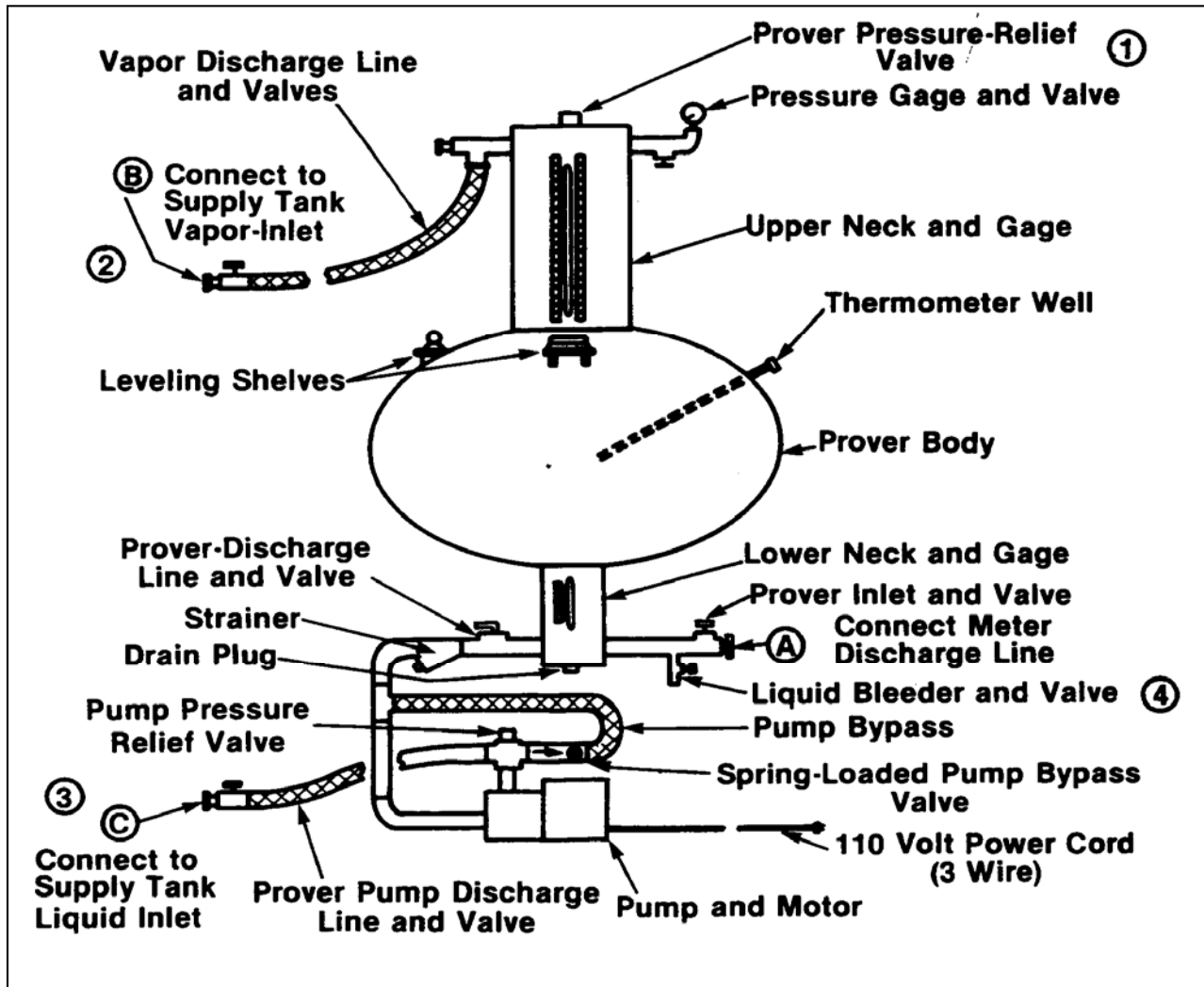
Appendix A
Volume Transfer Data Sheet for LPG Provers

Laboratory data and conditions:

Vessel Owner		Operator	
Vessel ID		Date	
Nominal Volume		Air Temperature	
Material		Relative Humidity	
Cubical Coefficient of Expansion		Standard deviation of the process, s_p from the Standard Deviation Chart	
Reference temperature of unknown prover		Degrees of Freedom	
Unknown prover graduations		Applicable specifications and tolerances	

Volume standard(s) data:

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

Run 1: Measurement Observations

DROP #	Reported Volume (gal)	Material (MS/SS/BS/SL)	Water Temp (°C) (Must be ≥ 0.5 °C and < 40 °C)	Gauge Delta (in³)
1				

Material for the Unknown:		MS, SS, TP, AL, PVLC
Final Gauge Reading at 0 psig:		in^3
Final Water Temperature:		°C

Run 2: Measurement Observations

DROP #	Reported Volume (gal)	Material (MS/SS/BS/SL)	Water Temp (°C) (Must be ≥ 0.5 °C and < 40 °C)	Gauge Delta (in³)
1				

Material for the Unknown:		MS, SS, TP, AL, PVLC
Final Gauge Reading at 0 psig:		in^3
Final Water Temperature:		°C

Run 2: Pressure Observations

Gauge Reading at 100 psig		in^3			
Gauge Reading at 0 psig:		in^3	Acceptable pressure repeatability? Y/N		
Repeated Gauge Reading at 100 psig		in^3			
Increasing Pressure Readings, 0 psig		Decreasing Pressure Readings, 150 psig			in^3
Increasing Pressure Readings, 50 psig		Decreasing Pressure Readings, 100 psig			in^3
Increasing Pressure Readings, 100 psig		Decreasing Pressure Readings, 50 psig			in^3
Increasing Pressure Readings, 150 psig		Decreasing Pressure Readings, 0 psig			in^3
Increasing Pressure Readings, 200 psig					in^3

Appendix B
Example Temperature Correction Table*

CCE	Mild Steel $1.86 \times 10^{-5}/°F$		Stainless Steel $2.65 \times 10^{-5}/°F$	
Temperature °F	in^3	gal	in^3	gal
Example for a <u>100</u> gallon prover and 60 °F reference temperature.				
-20	-34	-0.149	-49	-0.212
-15	-32	-0.139	-46	-0.199
-10	-30	-0.130	-43	-0.186
-5	-28	-0.121	-40	-0.172
0	-26	-0.112	-37	-0.159
5	-24	-0.102	-34	-0.146
10	-21	-0.093	-31	-0.133
15	-19	-0.084	-28	-0.119
20	-17	-0.074	-24	-0.106
25	-15	-0.065	-21	-0.093
30	-13	-0.056	-18	-0.079
35	-11	-0.047	-15	-0.066
40	-9	-0.037	-12	-0.053
45	-6	-0.028	-9	-0.040
50	-4	-0.019	-6	-0.026
55	-2	-0.009	-3	-0.013
60	0	0.000	0	0.000
65	2	0.009	3	0.013
70	4	0.019	6	0.026
75	6	0.028	9	0.040
80	9	0.037	12	0.053
85	11	0.046	15	0.066
90	13	0.056	18	0.080
95	15	0.065	21	0.093
100	17	0.074	24	0.106
105	19	0.084	28	0.119
110	21	0.093	31	0.133
115	24	0.102	34	0.146
120	26	0.112	37	0.159
CCE = coefficient of cubical expansion				

*Provide only the applicable coefficient of cubical expansion for the prover under test.