

## SOP 21

### Standard Operating Procedure for Calibration of LPG Provers<sup>1</sup>

#### 1 Introduction

##### 1.1 Purpose

This procedure is used to calibrate a volumetric field standard that is 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, with the various system components numbered to clarify the various operations described in the procedure. Upon completion of this procedure the prover volume is reported to provide the correct volume at the reference temperature of 60 °F and reference pressure of 100 psig. Detailed measurement ranges, standards, equipment, and uncertainties for this SOP are generally compiled in a separate document in the laboratory.

##### 1.2 Conformity Assessment

Standards that are calibrated for use in legal weights and measures applications should be evaluated for conformance to the appropriate specifications and tolerances that apply. LPG provers being calibrated should be evaluated for conformance using the checklist provided in NIST Handbook 105-4, Specifications and Tolerances for Liquefied Petroleum Gas and Anhydrous Ammonia Liquid Volumetric Provers, 2016. Where compliance is required by law, conformity evaluations should be conducted prior to performing calibrations. See Section 6.2 for reporting results.

##### 1.3 Prerequisites

1.3.1 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 must be visually inspected to determine that residual products are not present. Smell is not necessarily an adequate indicator of cleanliness.

1.3.2 Note: 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 working with petroleum products and pressurized vessels.

1.3.3 Verify that valid current calibration certificates with measurement values and uncertainties are available for all the standards used in the calibration.

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<sup>1</sup> 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 LPG provers in use are 20 gal, 25 gal, and 100 gal nominal sizes. The volume of LPG provers is established at reference conditions of 60 °F and 100 psig.

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 such as NIST.

- 1.3.4 Verify that the standards to be used have sufficiently small standard uncertainties for the intended level of the calibration.
- 1.3.5 Verify the availability of an adequate supply of clean water complying with GLP 10. Conditioning the water to achieve equilibration with the laboratory environment, to achieve minimal air entrainment and filtering to ensure minimal suspended solids is recommended. 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 because the density values are used as ratios.
- 1.3.6 Verify that the operator has had specific training in SOP 19, SOP 20, SOP 21, SOP 31, and GMP 3 and is familiar with the operating characteristics and conditioning of the standards and unknown test items being used and calibrated.
- 1.3.7 Ensure that a full cylinder of nitrogen or compressed air, or a regulated air source of at least 200 psig, and a proper pressure regulator, connection hose, pressure control valves and an appropriately sized pressure relief valve set to relieve at no more than 300 psig are available.
- 1.3.8 Ensure that proper safety procedures are followed for any installation requiring that the operator work from an elevated surface; that any fall restraints, handrails or other safety related items have been properly inspected and are used as required by laboratory policy.
- 1.3.9 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

- 1.3.10 The care required for calibrations conducted outside of a permanent laboratory includes proper safety, a clean and bubble-free water supply, measurement control programs, minimal air movement and a stable temperature environment shaded from direct sunshine to allow the prover, field standard, and clean calibration 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 calibration location.

## 2 Methodology

### 2.1 Scope, Precision, Accuracy

This procedure is applicable for the calibration of LPG field standard 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<sup>3</sup> units) are encountered most frequently, thus the procedure is written with that in mind. The changes necessary for calibrating 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 LPG field standard prover must agree within 0.02 % of the volume. Where the demonstrated standard deviation of the measurement process is less than 0.02 % of the volume, replicate values must agree within the limits on the standard deviation chart. The calibration 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 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 constant during transfers; thus, 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. Upon completion of this procedure the prover volume is reported to provide the correct volume,  $V_{ref}$ , at the reference temperature of 60 °F and reference pressure of 100 psig.

### 2.3 Standards and Equipment

The following are required to properly perform the calibration of a Field Standard LPG prover:

- 2.3.1 A calibrated standard prover having a minimum volume of 5 gal is acceptable for 20 gal and 25 gal LPG provers. 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 of many LPG provers may not be adequate to receive the full flow delivery from the reference standard. The calibrated volume of all standards used in this procedure must be determined using the same outlet piping as is used for LPG prover calibrations.

- 2.3.2 A funnel to aid in transferring water from calibrated flasks into the unknown prover.
- 2.3.3 Calibrated standards, typically glassware, of suitable sizes to calibrate the neck of prover.
- 2.3.4 Thermometers (2) with resolution and uncertainty better than 0.1 °C.

Note regarding temperature measurements: A digital temperature sensing device having a long cable will 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.

- 2.3.5 Meniscus reading devices (See GMP 3).
- 2.3.6 Timing device (calibration is not required; the 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 and complying with laboratory policies, with sufficient height to hold standard and to permit transfer of water from it to the prover by gravity flow.
- 2.3.8 Clean smoothbore pipe or tubing (hoses) to facilitate transfer of water from the laboratory standard to the LPG 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 calibration 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 Cylinder of compressed nitrogen or air with suitable regulator, as noted with prerequisites, and an appropriate pressure gauge.

#### 2.3.10 Pressure Gauges

The accuracy of the prover calibration relies on the accuracy of the pressure gauge used to measure the internal prover pressure. When the gauge mounted on the prover is used for the measurement, it is assumed that

systematic errors in the prover pressure gauge will be present in field application and use, thus calibration of the laboratory pressure gauge is not essential. However, the prover must be recalibrated if the pressure gauge is changed or repaired in the field.

When a laboratory pressure gauge is used to measure the internal prover pressure during the calibration, the laboratory gauge and the prover gauge must both have current calibration certificates.

2.3.11 Pipe plugs to seal unused prover ports, and piping and valves needed to facilitate gravity draining the prover in a time as similar as possible to the time taken to empty the prover using the LPG system pump.

2.3.12 Materials to seal all piping threads to permit pressurization to 200 psig. If thread tape is used in place of paste thread sealant, extra care must be exercised to ensure that no thread tape debris enters the prover under test.

## 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 the most direct route to the prover. Position and level the unknown prover where it can be reached from the elevated standard by the shortest feasible delivery system. Replace all valves and piping connected to the prover body below the nominal volume line with plugs and laboratory owned valves and fittings to eliminate errors that will result from leakage or expansion when the prover is pressurized to 200 psig or more during the calibration. These potential errors typically affect only the prover calibration process and have no effect during normal use because the prover zero indication is established under LPG system pressure. (If there is no significant change in system pressure once the zero setting is established during a meter test the change in system volume is minimal). Install in one lower port a fitting and valve for draining the prover. Plug all other ports with sealed plugs of suitable pressure ratings.

2.4.1.2 Remove the plug and/or relief valve (1) from the top of the LPG prover, and extend the drain pipe of the standard prover 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 (connected to 12c) to provide an air bleed.

2.4.1.3 If the prover inlet hose (11) is to be used as a gravity drain, it must be disconnected from the bottom of the prover, any check valves (12a) installed during normal operations removed and a valve installed between the prover body and the hose to ensure isolation of the hose from the pressurized prover. Failure to insert this

isolation valve will result in errors during the pressurized portion of the prover calibration. These errors will NOT be offset during use!

#### 2.4.2 Cleanliness Check

Both the standard and the unknown prover must be internally clean. This should be verified by checking that water properly drains 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. It is a good practice to remove the bottom drain plug (13) from the bottom of the prover lower neck to allow removal of any debris that may have accumulated in the lower neck of the prover.

#### 2.4.3 Neck scale plate calibration

Neck scale plate calibrations are generally conducted only for new or damaged volumetric measures, those that have not been calibrated by the laboratory in the past, or those for which the calibration data is not available. See SOP 31 for the neck scale plate calibration procedure.

#### 2.4.4 Body Calibration

2.4.4.1 Drain the unknown LPG prover through the drain valve installed during prover preparations and the connected hose. When the liquid reaches the top of the lower gauge glass, close the drain valve and adjust the liquid meniscus level to the lower neck zero graduation. Record the elapsed time required to drain the prover to the top of the neck,  $t_i$ . Continue adjusting the liquid level until completion of the final drain time of 30 s. The liquid level should be exactly at the zero graduation and the drain valve closed simultaneously at the end of the 30 s drain time. (Final adjustment of the water level close to the zero mark should be started during the 30 s final drain period but should not be completed before the end of the 30 s drain period).

Alternatively, though not recommended because of resulting errors, the prover may be completely drained, given a 30 s final drain time and then refilled to the lower zero graduation with a funnel that has been wet down by adding the small volume of water needed to bring the level to the zero graduation. Errors will result from this process due to the extended time between closing the valve and setting the zero level and other factors associated with the process and should be considered in the uncertainty assessment. These errors can be minimized if the prover is refilled to the lower neck zero graduation within 30 s of cessation of main flow.

2.4.4.2 Once the zero indication has been properly established, transfer the nominal volume from the standard(s) into the LPG prover in the usual manner, recording the associated delta (offset of the water

level in the standard from nominal) and water temperature reading in the standard. If multiple transfers are required, record the water temperature of the standard at the time of each transfer, but that of the LPG prover only after the final transfer. Verify that the LPG prover retained its level condition or adjust it to the level condition as noted earlier. "Rock" the prover to "bounce" the liquid in the upper sight gauge before reading. Record the final scale indication and prover temperature after the nominal volume has been transferred into the unknown LPG prover with 0 psig pressure applied, *Run I<sub>ATM</sub>*.

2.4.4.3 Reinstall the plug and relief valve in the top of the prover using an appropriate sealant to create a leak free installation.

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

2.4.4.4 Pressurize the prover to 200 psig, hold that pressure for several minutes, then return the prover pressure to 0 psig and verify that the prover indication has not changed from that previously recorded as *Run I<sub>ATM</sub>*. If the prover indication returns to the recorded value, pressurize the prover to 100 psig and record the prover indication and temperature, *Run I<sub>100</sub>*.

Note: The prover indication at 0 psig must return to the value recorded as *Run I<sub>ATM</sub>*. If it does not, corrective action must be taken to correct any causes, i.e., leaks, excessive air entrained in the water. Once corrective actions are completed repeat 2.4.1.1 through 2.4.4.3

2.4.4.5 Calculate the effective volume of water delivered into the prover using Eqn. 3 or Eqn. 4 as appropriate. Record that value, *Run I V<sub>ref</sub>*.

2.4.4.6 Calculate the prover volume for Run 1 using Eqn. 6 or Eqn. 7, as appropriate. Record the calculated prover volume, *Run I<sub>AsFound</sub>*. The calculated prover volume must be within  $\pm 0.2$  % of the prover nominal volume.

2.4.4.7 If the indication is not within  $\pm 0.2$  % of the prover nominal volume the prover must be adjusted (whenever possible) to indicate the  $V_{ref}$  value as described in 2.4.6 Prover Adjustment to Nominal.

2.4.4.8 After completing the prover adjustment process, repeat 2.4.4.1 through 2.4.4.6 as needed until the "As Left" prover volume is as near to the prover nominal as possible. Record the final prover volume, *Run I<sub>Final</sub>*.

2.4.4.9 Repeat steps 2.4.4.1 through 2.4.4.6 for Run 2 recording *Run 2<sub>ATM</sub>*, and *Run 2<sub>100</sub>*.

- 2.4.4.10 Calculate the effective volume of water delivered into the prover using Eqn. 3 or Eqn. 4 as appropriate. Record that value as *Run 2 V<sub>ref</sub>*.
- 2.4.4.11 Calculate the prover volume for Run 2 using Eqn. 6 or Eqn. 7. Record the calculated prover volume for *Run 2<sub>Final</sub>*. The calculated prover volume must be within  $\pm 0.2$  % of the prover nominal volume.
- 2.4.4.12 The calculated prover volume, *Run 2<sub>Final</sub>*, must agree with the *Run 1<sub>Final</sub>* value within  $\pm 0.02$  % of the prover nominal volume or pass the F-test as shown in Section 3 (use the more stringent limits for assessment of repeatability).
- 2.4.4.13 If the prover volumes do not repeat within 0.02 % of prover nominal volume, or pass the F-test (whichever limit is more stringent), corrective action must be initiated and the calibration runs repeated until two consecutive calibration runs repeat within 0.02 % of prover nominal volume or pass the F-test indicating that the accepted laboratory process limits are met.

#### 2.4.5 Pressure Correction Curve Determination

- 2.4.5.1 The internal pressure and thus the volume of the prover may vary during use. Accordingly, a pressure correction must be made, using the Pressure Correction Calculations given in section 3.3.
- 2.4.5.2 The calibration status of the pressure gauge mounted on the prover MUST be assessed and documented on the calibration certificate.

It is a best practice to permanently mark the mounted pressure gauge with a unique identifier that can be included as the gauge identification on the prover calibration certificate.

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

- 2.4.5.3 Ensure that all valves are closed except the vapor return valve, if still installed and being used to apply pressure to the prover.
- 2.4.5.4 Verify that the final scale indication has not changed since it was recorded at the end of Run 2 (if it has changed it may signal a leak in one of the valves or fittings), and then bleed off pressure until the installed prover gauge reads 0 psig.
- 2.4.5.5 Apply pressure to the prover until the installed pressure gauge indicates 20 psig.

Note: As pressure is applied, the density of the water increases thus decreasing the water level inside the prover; the gas in the prover heats up and then cools; the prover shape begins to change due to

pressure deformation; all resulting in a drifting pressure setting as the system comes to equilibrium. Continue adjusting the pressure until a stable pressure indication is obtained while lightly tapping the gauge to ensure that the gauge needle is not sticking. (Rotating the handle of a screwdriver against the side of the pressure gauge is a good alternative to tapping on the gauge).

2.4.5.6 "Bounce" the liquid in the neck, then read and record the liquid level at this applied pressure,  $SR_{20i}$ .

2.4.5.7 Continue increasing the pressure in the prover, setting a stable pressure at 50 psig, 100 psig, 150 psig and 200 psig, recording the prover indication at each pressure,  $SR_{50i}$ ,  $SR_{100i}$ ,  $SR_{150i}$ , and  $SR_{200i}$ .

Note: The scale indication at 100 psig should be the same as was observed for Run  $2_{100}$ .

2.4.5.8 Slowly decrease the prover pressure, setting a stable pressure at 150 psig, 100 psig, 50 psig and 20 psig, recording the prover indication at each pressure,  $SR_{150d}$ ,  $SR_{100d}$ ,  $SR_{50d}$ , and  $SR_{20d}$ .

Note: As the pressure is released the components of the system 'relax' and the gas temperature cools, the pressure drifts as the system equilibrates with each pressure change.

2.4.5.9 The prover indication at each corresponding decreasing pressure must repeat the increasing pressure indication within 0.02 % of nominal prover volume.

2.4.5.10 If any decreasing pressure indication fails to repeat the corresponding increasing pressure indication within 0.02 % of nominal prover volume the cause for this failure must be determined and the calibration performed again until repeatability is obtained.

2.4.5.11 A plot of the scale indications versus the applied pressure should be relatively linear. If significant non-linearity is detected it may indicate either trapped air or significant deformation of the prover body with changes in pressure.

If the issue is determined to be trapped air, the cause of this air entrapment must be corrected and the prover calibration repeated.

If the problem is determined to be significant deformation of the prover body with changes in pressure, the prover must be taken out of service and a hydrostatic pressure test scheduled as it may indicate failure of the materials from which the prover is made. This will likely require replacement of the prover.

## 2.4.6 Prover Adjustment to Nominal

2.4.6.1 With the pressure in the prover at 100 psig, the prover volume, calculated using the appropriate equation in Section 3, should differ from nominal no more than 0.2 % of nominal prover volume.

2.4.6.2 Adjust the LPG prover upper scale to read the calculated Run 1  $V_{ref}$  value.

2.4.6.3 If the LPG prover scale plate has a 0 indication at nominal, adjust the scale plate to indicate Run 1,  $V_{ref} - \text{Nominal}$ , converting units as needed.

2.4.6.4 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:

$$L_c = \text{Nominal} - V_{ref} + sr_u \quad \text{Eqn. (1)}$$

where:

**Table 2. Variables used in Eqn. 1.**

Variable	Description
$L_c$	Prover correction at 100 psig
Nominal	LPG prover nominal volume
$V_{ref}$	Volume of water in the unknown prover at 60 °F and 100 psig
$sr_u$	Upper scale indication at 100 psig
Note: Take care to use matching units in the calculation.	

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

$$h = \frac{4 |L_c|}{\pi d^2} \quad \text{Eqn. (2)}$$

where:

**Table 3. Variables used in Eqn. 2.**

Variable	Description
$H$	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)

2.4.6.6 Once the prover is correctly adjusted, repeat the Body Calibration process to verify that the adjustment successfully corrected any indication errors.

## 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 the prover, then remove the plug (13) at the lower neck to facilitate complete drainage of the prover. If time permits, let the prover drain overnight. If it is not possible to remove the plug, other means, such as siphoning or vacuuming, must be used to remove all water from the bottom of the prover.

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. Flushing the prover with 1 or 2 gallons of ethanol will aid in complete removal of the 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. This will not be an issue if the recommendations of removing all user connections from the prover body described in Preliminary Operations have been followed.

## 3 Calculations

### 3.1 Single Delivery

Calculate the effective volume of water delivered to the unknown prover at 60 °F and 100 psig,  $V_{ref}$ , using Eqn. 3:

$$V_{ref} = \frac{1}{Pf} \left( \frac{\rho_1 \{ (V_{s60} + \Delta_1) [1 + \alpha (t_1 - 60 \text{ °F})] \}}{\rho_x [1 + \beta (t_x - 60 \text{ °F})]} \right) \quad \text{Eqn. (3)}$$

### 3.2 Multiple Deliveries

Calculate the effective volume of water delivered to the unknown prover at 60 °F and 100 psig,  $V_{ref}$ , using Eqn. 4:

$$V_{ref} = \frac{1}{Pf} \left( \frac{\rho_1 \{ (V_{s60} + \Delta_1) [1 + \alpha (t_1 - 60 \text{ °F})] \} + \rho_2 \{ (V_{s60} + \Delta_2) [1 + \alpha (t_2 - 60 \text{ °F})] \} + \dots + \rho_n \{ (V_{s60} + \Delta_n) [1 + \alpha (t_n - 60 \text{ °F})] \}}{\rho_x [1 + \beta (t_x - 60 \text{ °F})]} \right) \quad \text{Eqn. (4)}$$

These equations correct the volume of water in the unknown prover for the effects of temperature on the density of the water, the thermal expansion and contraction of the standard and unknown provers, and the compressibility of water at 100 psig.

**Table 4. Variables for  $V_{ref}$  Equations 3 and 4.**

Variable	Description
$V_{ref}$	Effective volume of water delivered to the unknown prover corrected to 60 °F and 100 psig.
$V_{S60}$	Volume of the standard vessel at 60 °F and atmospheric pressure (If the standard has been calibrated at a different reference temperature, i.e., 20 °C (68 °F) for glassware, that temperature must be used in the calculation of the temperature correction in Eqn. 3 and Eqn. 4, taking care to make coefficients of expansion units).
$P$	Internal LPG prover pressure at a specific data point; psig
$P_f$	Pressure factor to be used at: 0 psig: 1.000 000 20 psig: 1.000 064 50 psig: 1.000 161 100 psig: 1.000 322 150 psig: 1.000 483 200 psig: 1.000 644 These factors are the ratio between the water density at 60 °F and 0 psig and that at 60 °F and the given pressure, using the equations in GLP 10.
$\rho_1, \rho_2, \dots, \rho_N$	Density of the water in the standard prover where $\rho_1$ is the density of the water for the first delivery, $\rho_2$ is the density of the water for the second delivery, and so on until all $n$ deliveries are completed
$\Delta_1, \Delta_2, \dots, \Delta_n$	Volume difference between the 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, delta ( $\Delta$ ) is negative. If the water level is above the reference line, $\Delta$ is positive. If the water level is at the reference line, $\Delta$ is zero  Note: units must match volume units for the standard. For slicker-plate type standards, the $\Delta$ is zero.
$t_{S1}, t_{S2}, \dots, t_{Sn}$	Temperature of water for each delivery with the subscripts as above
$\alpha$	Cubical coefficient of expansion (also CCE) for the standard: /°F
$\beta$	Cubical coefficient of expansion (also CCE) for the LPG prover: /°F
$t_x$	Temperature of the water in the filled unknown vessel: °F
$\rho_x$	Density of the water in the unknown vessel: g/cm <sup>3</sup> using GLP 10.

### 3.3 Pressure Correction Calculations

3.3.1 Compute the pressure correction,  $P_{corr}$ , at each pressure that the prover was read by correcting for the compressibility of the water using Eqn. 5:

$$P_{corr} = sr_{100} - sr_x + \left[ (1.000322 \times P_N - P_N) \left( \frac{100 \text{ psig} - x \text{ psig}}{100} \right) \right] \quad \text{Eqn. (5)}$$

where the water compressibility factor at 60 °F and 100 psig is 1.000322. Caution: Be certain to match the pressure correction units to those of the prover scale plate indications.

**Table 5. Variables used in Eqn. 5.**

Variable	Description
$P_{corr}$	Correction to scale indication resulting from prover pressure: x psig
$sr_{100}$	Prover Scale Plate reading at 100 psig
$sr_x$	Prover Scale Plate reading at x psig
$P_n$	Nominal volume of prover

3.3.2 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.

3.3.3 Eqn. 5 can also be used to generate a pressure correction table for the prover across the range of 20 psig to 200 psig.

### 3.4 Prover Volume

3.4.1 LPG provers are generally adjusted to the nominal value using 100 psig as the reference pressure and a 60 °F reference temperature. Because the calibration medium, water, compresses thereby decreasing in volume by 0.00032 gal/gal at 100 psig, the prover indication must be adjusted to indicate the decreased water volume to correctly indicate the volume of LPG at 100 psig. The value calculated as  $V_{ref}$  correctly represents the volume of water delivered at atmospheric pressure after pressurizing to 100 psig.

3.4.2 For an LPG prover having a scale plate that directly indicates the total volume delivered the prover volume is calculated as:

$$\text{Prover volume} = V_{ref} - sr_{100} + \text{Nominal} \quad \text{Eqn. (6)}$$

3.4.3 For a prover having a '0' graduation marking at the nominal volume delivered, with a scale plate that indicates a volume above and below that graduation as a positive or negative value, the prover volume is calculated as:

$$\text{Prover volume} = V_{ref} - sr_{100} \quad \text{Eqn. (7)}$$

For either type scale plate the prover should be adjusted so that the prover indicates the effective volume of water delivered:  $V_{ref}$ .

- 3.5 Calculate the within process standard deviation,  $s_w$ , for the replicate runs. For two runs, the degrees of freedom will be one.
- 3.6 Calculate the F statistic to compare the observed within process standard deviation,  $s_w$ , to the accepted (pooled) within process standard deviation for the measurement process. (See NISTIR 6969, Sections 8.4 and 8.9.2, for more information on pooling standard deviations and F-tests).

$$F = \frac{s_w^2 \text{ Observed}}{s_w^2 \text{ Accepted}} \quad \text{Eqn. (8)}$$

The calculated F statistic must be less than the F value obtained from an F table at 95 % confidence level (Table 9.12, NISTIR 6969) to be acceptable. The F value is obtained from the F table for one degrees of freedom for the numerator and the degrees of freedom for the denominator is equal to the number of degrees of freedom in the pooled within process standard deviation. If the data fails the F-test and the source of the error cannot be determined conclusively and corrected, the measurement must be repeated.

- 3.7 Calculate and report the mean volume of the volumetric standard at 60 °C and 100 psig.

If adjustments were made during replicate runs, report the “as found” volume or the mean of “as found” volumes and the “as left” volume or mean of “as left” volumes, as applicable, at 60 °F and 100 psig. (I.e., do not calculate a mean value by combining “as found” and “as left” values when adjustments are made).

#### 4 Measurement Assurance

- 4.1 Due to the cost of obtaining an LPG prover for use as a check standard it will be unlikely that a laboratory will have one in place. Instead a standard deviation chart is used to monitor the within process standard deviation.

- 4.2 A standard deviation chart is used for measurement assurance through the evaluation of replicate measurements. The standard deviation of each combination of Run 1 and Run 2 is calculated and the pooled standard deviation over time may be used to estimate the short-term variability in the measurement process. A standard deviation chart for unknown provers represents the variability in condition of test measures submitted for calibration as well as the short-term repeatability of the measurement process but does not monitor the stability of the reference standard or represent the variability or potential systematic errors associated with meniscus readings. The range of the calibration runs must repeat to within 0.02 % of the volume, or the standard deviation must be less than applicable control/action limits on standard deviation charts, or the observed standard deviation must pass the F-test statistics (as applicable).

For unknown standards that are adjusted, do not combine an “as found” value with an “as left” value for the two runs recorded in the chart; use the adjusted value from Run 1 and the value from Run 2, both at the applicable reference temperature and pressure, when entering values in a standard deviation or range chart.

## 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,  $s_p$ , 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 The standard uncertainty for the standard,  $u_s$ , is obtained from the calibration certificate. 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 NISTIR 6969, 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 standard deviation chart for LPG provers (and not from charts for a refined fuel check standard) (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 (See SOP 31).
- 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 the following table. 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 6. Example uncertainty budget table.**

Uncertainty Component Description	Symbol	Source	Typical Distribution
Uncertainty of the standard (5.1)	$u_s$	Calibration certificate; may be multiplied or added based on dependencies	Expanded divided by coverage factor
Accepted standard deviation of the process (5.2)	$s_p$	Standard deviation chart (or control chart)	Normal
Uncertainty or uncorrected error associated with a neck calibration (5.3)	$u_n$	SOP 31	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 (CCE) on S (5.4)	$u_{CCE}$	5 % to 10 % of the CCE (EURAMET CG-21)	Rectangular
Cubical Coefficient of Expansion (CCE) on X (5.4)	$u_{CCE}$	5 % to 10 % of the CCE (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, drift, or variability of standards (5.2)	$u_b$	From calibration history of reference standards	Rectangular
Uncertainty of drain time (insignificant if closely following the procedure)	$u_d$	From experimental data	Normal

## 5.7 Uncertainty Evaluation

Where applicable, uncertainties for volume calibrations that are assessed for conformity must meet decision rule criteria in the applicable documentary standards.

## 6 Calibration Certificate

### 6.1 Report results as described in NISTIR 6969, SOP No. 1, Preparation of Calibration Certificates, with the addition of the following:

For LPG provers, calculate and report the mean final prover volume and its associated uncertainty, reference temperature, reference pressure, 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, identification and calibration status of the mounted pressure gauge and if the gauge is uncalibrated a Note stating the requirement that the prover be recalibrated if the gauge is repaired or replaced.

The certificate 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.

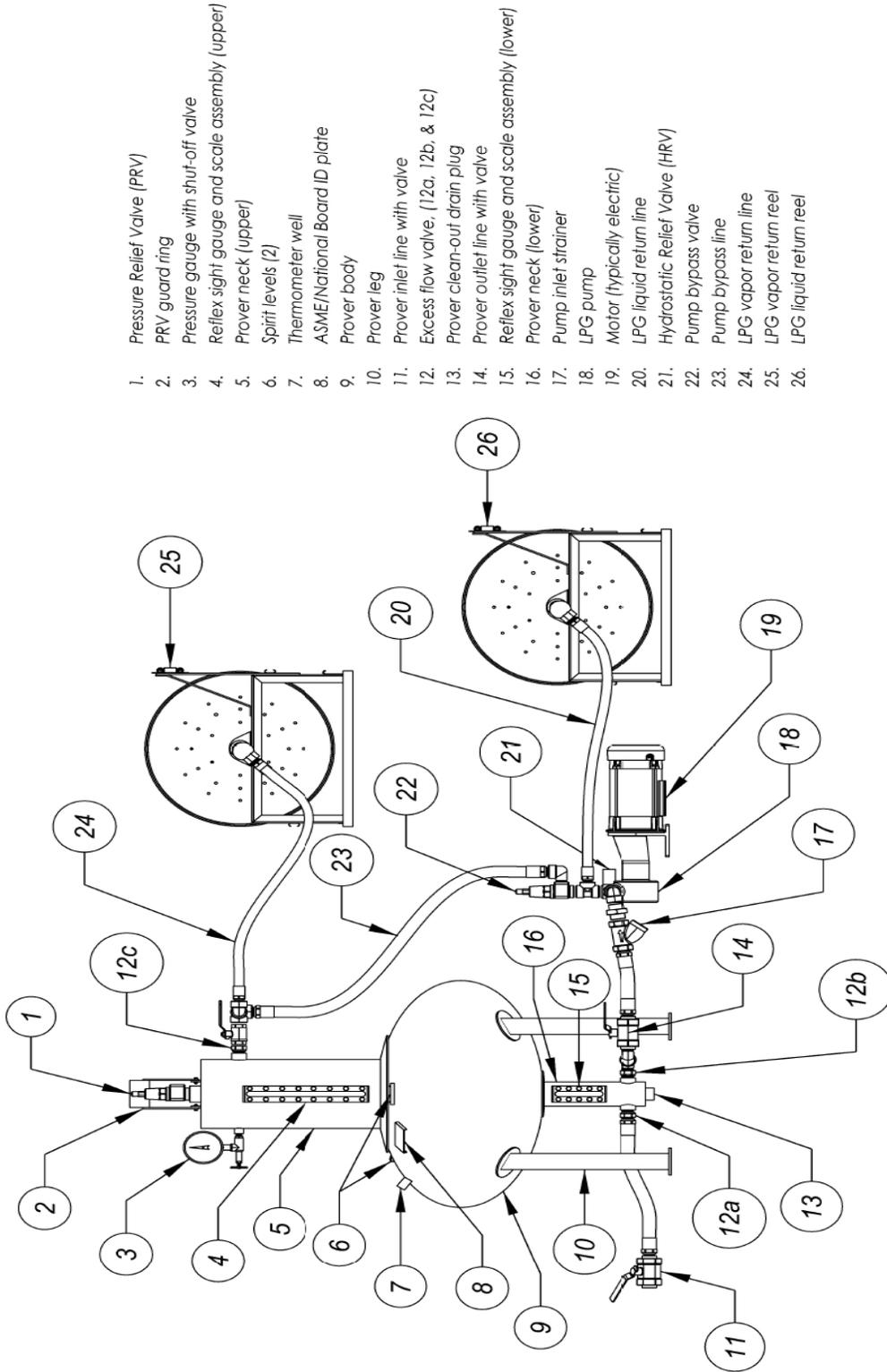
The SI unit of volume is m<sup>3</sup>, so a conversion factor must be included on the report in the notes section when other units are used.

## 6.2 Conformity Assessment

Evaluate compliance to applicable tolerances as needed or required by the customer or by legal metrology requirements. Compliance assessments must note the applicable documentary standard and which portions of the standard were or were not evaluated. The uncertainty for volume calibrations must be less than the tolerances published in the applicable documentary standards. For volume calibrations where the unknown standard can be adjusted, it is standard practice to adjust the standard or leave the scale plate in a position close enough to its nominal volume to ensure that the absolute value of the measurement result plus the uncertainty is less than the applicable tolerance. Where the unknown standard cannot be adjusted, and a portion of the uncertainty band from the error exceeds tolerance limits, it is not appropriate to state compliance with the tolerances unless additional decision rules are communicated with and agreed to by the end user. Correction values (measurement results) may need to be used by the end user in such cases.

**Table 7: Data recorded.**

Variable	Type Data	Step
$t_i$	Initial drain time	2.4.4.1
$Run\ 1_{ATM}$	Scale indication	2.4.4.2
$Run\ 1_{100}$	Scale Indication	2.4.4.4
$Run\ 1\ V_{ref}$	Calculated value Eqn. 3 or 4	2.4.4.5
$Run\ 1_{AsFound}$	Calculated value Eqn. 6 or 7	2.4.4.6
$Run\ 1_{Final}$	Calculated value Eqn. 6 or 7	2.4.4.8
$Run\ 2_{ATM}$	Scale indication	2.4.4.2
$Run\ 2_{100}$	Scale Indication	2.4.4.4
$Run\ 2\ V_{ref}$	Calculated value Eqn. 3 or 4	2.4.4.10
$Run\ 2_{Final}$	Calculated value Eqn. 6	2.4.4.11
$Pass/Fail$	$Run\ 1_{Final} - Run\ 2_{Final} < 0.02\ \% \text{ Nominal}$	2.4.4.12
	Pressure Gauge Identifier	2.4.5.2
$sr_{20i}$	Scale Reading at 20 psig increasing	2.4.5.6
$sr_{50i}$	Scale Reading at 50 psig increasing	2.4.5.7
$sr_{100i}$	Scale Reading at 100 psig increasing	2.4.5.7
$sr_{150i}$	Scale Reading at 150 psig increasing	2.4.5.7
$sr_{200i}$	Scale Reading at 200 psig increasing	2.4.5.7
$sr_{150d}$	Scale Reading at 150 psig decreasing	2.4.5.8
$sr_{100d}$	Scale Reading at 100 psig decreasing	2.4.5.8
$sr_{50d}$	Scale Reading at 50 psig decreasing	2.4.5.8
$sr_{20d}$	Scale Reading at 20 psig decreasing	2.4.5.8
$Pass/Fail$	Repeat all pressure point indications	2.4.5.10



1. Pressure Relief Valve (PRV)
2. PRV guard ring
3. Pressure gauge with shut-off valve
4. Reflex sight gauge and scale assembly (upper)
5. Prover neck (upper)
6. Spirit levels (2)
7. Thermometer well
8. ASME/National Board ID plate
9. Prover body
10. Prover leg
11. Prover inlet line with valve
12. Excess flow valve, (12a, 12b, & 12c)
13. Prover clean-out drain plug
14. Prover outlet line with valve
15. Reflex sight gauge and scale assembly (lower)
16. Prover neck (lower)
17. Pump inlet strainer
18. LPG pump
19. Motor (typically electric)
20. LPG liquid return line
21. Hydrostatic Relief Valve (HRV)
22. Pump bypass valve
23. Pump bypass line
24. LPG vapor return line
25. LPG vapor return reel
26. LPG liquid return reel

**Figure 1. Representative LPG Prover Schematic**

## Appendix A: Data Recording Sheet

LPG Prover Calibration

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### Laboratory Information

Metrologist		Date	
Location		Temperature	
Date		Relative Humidity	
Vessel Identification		Barometric Pressure	
$s_p$ of Process		Degrees of Freedom	

### Unknown Prover Information

Owner		Manufacturer	
Address		Serial	
		Material	
		Cubical Coefficient of Expansion ( $\beta$ )	
Phone		Reference Temperature	
Unknown prover graduations		Applicable Specifications and Tolerances	
Initial Drain time, $t_i$ , (s) (Time to drain full prover to Zero)		Maximum Permissible Error (MPE)	
Pressure Gauge Identification		Is Pressure Gauge Calibrated?	
Upper Neck Inside Diameter		Lower Neck Inside Diameter	

### Volume standard(s) data:

Identification (ID) (Note ID of Standards)	Reference Temp.	Nominal Volume	Volume Correction	Expanded Unc: From Cal. Cert.	Unc: $k$ factor	Cubical Coefficient of Expansion ( $\alpha$ )
$S_1$						
$S_2$						
$S_3$						
$S_4$						

### Run 1: Data for volumes delivered from the standards:

DROP #	Reported Volume (gal)	Material (MS/SS/)	Water Temperature ( $^{\circ}$ C) (Must be $\geq 0.5$ $^{\circ}$ C and $< 40$ $^{\circ}$ C)	Offset from Nominal $\Delta$ (in <sup>3</sup> )
1				
2				
3				
4				
5				

### Observations Calibration Data

Run 1 Variable	Indication (Left scale) Units ( )	Indication (Right scale) Units ( )	Prover Water Temperature ( $^{\circ}$ C)
<i>Run 1<sub>ATM</sub></i>			
<i>Run 1<sub>100</sub></i>			
<i>Run 1<sub>V<sub>ref</sub></sub></i>			
<i>Run 1<sub>AsFound</sub></i>			
<i>Run 1<sub>Final</sub></i>			
Within MPE?	Pass / Fail	Pass / Fail	

## Appendix A: Data Recording Sheet

LPG Prover Calibration

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### Run 2: Data for volumes delivered from the standards:

DROP #	Reported Volume (gal)	Material (MS/SS/)	Water Temperature (°C) (Must be $\geq 0.5$ °C and $< 40$ °C)	Offset from Nominal $\Delta$ (in <sup>3</sup> )
1				
2				
3				
4				
5				

### Observations Calibration Data

Run 2 Variable	Indication (Left scale) Units ( )	Indication (Right scale) Units ( )	Prover Water Temperature (°C)
<i>Run 2<sub>ATM</sub></i>			
<i>Run 2<sub>100</sub></i>			
<i>Run 2 V<sub>ref</sub></i>			
<i>Run 2<sub>AsFound</sub></i>			
Within MPE?	Pass / Fail	Pass / Fail	

### Observations Pressure Calibration Data

Pressure Setting	Indication (Left scale) Unit ( )	Indication (Right scale) Unit ( )	Pressure Setting	Indication (Left scale) Unit ( )	Indication (Right scale) Unit ( )
<i>SR<sub>20i</sub></i>			<i>SR<sub>150d</sub></i>		
<i>SR<sub>50i</sub></i>			<i>SR<sub>100d</sub></i>		
<i>SR<sub>100i</sub></i>			<i>SR<sub>50d</sub></i>		
<i>SR<sub>150i</sub></i>			<i>SR<sub>20d</sub></i>		
<i>SR<sub>200i</sub></i>					

### Repeatability Evaluation (Run 1 to Run 2)

Repeatability error < 0.02 % Nominal	Pass	Fail
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### Prover Volume (Average of Run 1<sub>Final</sub> and Run 2<sub>Final</sub>)

	Left scale Units ( )	Right scale Units ( )
Prover Volume at 60 °F and 100 psig		
Uncertainty		
<i>k</i>		

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## Appendix B

Example Temperature Correction Table<sup>A</sup>

Example for a 100 gallon prover and 60 °F reference temperature.				
CCE	Mild Steel $1.86 \times 10^{-5} / ^\circ\text{F}$		Stainless Steel $2.65 \times 10^{-5} / ^\circ\text{F}$	
Temperature °F	in <sup>3</sup>	gal	in <sup>3</sup>	gal
-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

<sup>A</sup> Provide only the applicable coefficient of cubical expansion for the prover under test.