

Designation: D 6897 - 03a

Standard Test Method for Vapor Pressure of Liquefied Petroleum Gases (LPG) (Expansion Method)¹

This standard is issued under the fixed designation D 6897; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope*

- 1.1 This test method covers the use of automatic vapor pressure instruments to determine the vapor pressure of lique-fied petroleum gas products at a temperature of 37.8°C, vapor to liquid ratio of 0.5:1, and pressures from 200 to 1550 kPa on a sample volume of 3.33 mL.
- 1.2 This test method is applicable to the determination of vapor pressures of liquefied petroleum gas products at temperatures from 37.8 to 70°C, vapor to liquid ratios of 0.1:1 to 4:1, and pressures up to 3500 kPa; however, the precision of the test method (see Section 15) has only been determined for a vapor to liquid ratio of 0.5:1, at a temperature of 37.8°C, and a pressure range from 300 to 1500 kPa.

Note 1—This test method is not intended to determine the true vapor pressure of LPG samples, but rather determine and report the vapor pressure of LPG at the 37.8° C temperature and 0.5:1 vapor to liquid ratio as the Test Method D 1267 method.

Note 2—This test method is not a true vapor pressure method and will not measure the full contribution from any dissolved gases such as nitrogen or helium if they are present. The contribution of light gases to the measured vapor pressure is highly dependent on the test temperature, type of gas, and V/L ratio of the test. A task group has been formed to determine or quantify the effect that the contribution of light gases have on the measured vapor pressure of LPG sample types as a function of the test temperature, type of gas, and V/L ratio of the test.

- 1.3 The values stated in SI units are to be regarded as standard.
- 1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For specific warning statements, see Appendix X2.

2. Referenced Documents

- 2.1 ASTM Standards: ²
- D 1265 Practice for Sampling Liquefied Petroleum (LP) Gases (Manual Method)
- D 1267 Test Method for Gage Vapor Pressure of Liquefied Petroleum (LP) Gases (LP-Gas Method)
- D 2892 Test Method for Distillation of Crude Petroleum (15-Theoretical Plate Column)
- D 3700 Practice for Obtaining LPG Samples Using a Floating Piston Cylinder
- D 5191 Test Method for Vapor Pressure of Petroleum Products (Mini Method)
- D 6299 Practice for Applying Statistical Quality Assurance Techniques to Evaluate Analytical Measurement System Performance
- 2.2 Energy Institute Standards:³
- IP 181 Sampling Petroleum Gases

3. Terminology

- 3.1 Definitions:
- 3.1.1 liquefied petroleum gases (LPG), n—narrow boiling range hydrocarbon mixtures, consisting mainly of propane or propylene, or both (**Warning**—Extremely flammable. Harmful if inhaled), butanes and butylenes, or both; in which the concentration of hydrocarbon compounds with boiling point greater than 0°C is less than 5 % by liquid volume, and whose vapor pressure at 37.8°C (100°F) is not greater than 1550 kPa.
 - 3.2 Definitions of Terms Specific to This Standard:
- 3.2.1 total pressure (P_{tot}), n—the absolute pressure (relative to vacuum) exerted by the specimen at the specified temperature and vapor-liquid ratio.

 $^{^{\}rm 1}$ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.08 on Volatility.

Current edition approved Dec. 1, 2003. Published January 2004. Originally approved in 2003. Last previous edition approved in 2003 as D 6897–03.

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

³ Available from Energy Institute, 61 New Cavendish St., London, WIG 7AR, U.K.

- 3.2.2 *true vapor pressure*, *n*—the physical property of a given liquid which specifies the maximum pressure at which a vapor phase can coexist with the liquid phase at a given equilibrium temperature condition.
- 3.2.3 *vapor pressure of LPG*, *n*—the total pressure corrected relative to normal barometric pressure.
- 3.2.4 *vapor to liquid ratio*, *n*—the ratio of the volume of the vapor in equilibrium to the fill volume of liquid.

4. Summary of Test Method

- 4.1 Employing a measuring chamber with a built-in piston, the chamber is rinsed three times with a portion of sample, which is then discarded. A sample of defined volume is drawn from a pressurized sampling system into the temperature-controlled chamber at 5°C by moving the piston to the filling position. After sealing the chamber, the volume is expanded by moving the piston until the final volume produces the desired vapor to liquid ratio of 0.5:1. The temperature of the measuring chamber is then regulated to the test temperature of interest, such as 37.8°C.
- 4.2 The observed total pressure at equilibrium is corrected relative to 101.3 kPa and reported as the LPG vapor pressure at the selected test temperature.

5. Significance and Use

- 5.1 Information on the vapor pressures of liquefied petroleum gas is pertinent to selection of properly designed storage vessels, shipping containers, and customer utilization equipment to ensure safe handling of these products.
- 5.2 Determination of the vapor pressure of liquefied petroleum gas is important for safety reasons to ensure that the maximum operating design pressures of storage, handling, and fuel systems will not be exceeded under normal operating temperature conditions.
- 5.3 For liquefied petroleum gases, vapor pressure can be considered a semi-quantitative measure of the amount of the most volatile material present in the product.
- 5.4 This test method uses a small sample volume and excludes any manual handling of a measuring chamber under high pressure.

6. Apparatus

6.1 Vapor Pressure Apparatus⁴—The type of apparatus suitable for this test method employs a small volume, cylindrically shaped measuring chamber with associated equipment to control the chamber temperature within the range of 5 to 70°C. The measuring chamber shall contain a movable piston with a maximum dead volume of less than 1 % of the total volume at the lowest position to allow sample introduction into the measuring chamber and expansion to the desired vaporliquid ratio. A static absolute pressure transducer shall be incorporated in the piston. The measuring chamber shall contain an inlet/outlet valve combination for sample introduc-

tion and expulsion. The piston and the valve combination shall be at the same temperature as the measuring chamber to avoid any condensation or excessive evaporation.

6.1.1 The test chamber shall be designed to contain a total of 5 mL of liquid and vapor and be capable of maintaining a vapor-liquid ratio of 0.5:1 with a maximum deviation of 0.02.

Note 3—The test chamber employed by the instruments used in generating the precision and bias statements were constructed of nickel-plated aluminum and stainless steel.

Note 4—Test chambers exceeding a 5 mL capacity can be used, but the precision and bias statements (see Section 15) are not known to apply.

- 6.1.2 Electronic temperature control shall be used to maintain the measuring chamber at the prescribed temperature within $\pm 0.1^{\circ}\text{C}$ for the duration of the vapor pressure measurement.
- 6.1.3 The pressure transducer shall have a range of 0 to 3500 kPa with a minimum resolution of 1 kPa. The minimum accuracy shall be ± 1 kPa for pressures up to 700 kPa, ± 2 kPa for pressures up to 1750 kPa, and ± 4 kPa for pressures up to 3500 kPa.
- 6.1.4 A platinum resistance thermometer, or equivalent, shall be used for measuring the temperature of the test chamber. The minimum temperature range of the measuring device shall be from 0 to 80°C with a resolution of 0.1°C and a minimum accuracy of ± 0.1 °C.
- 6.2 *Vacuum Pump for Calibration*, capable of reducing the pressure in the measuring chamber to less than 0.01 kPa absolute.
- 6.3 McLeod Vacuum Gage or Calibrated Electronic Vacuum Measuring Device for Calibration, to cover at least the range from 0.01 to 0.67 kPa (0.1 to 5 mm Hg). The calibration of the electronic vacuum measuring device shall be regularly verified in accordance with A6.3 of Test Method D 2892.
- 6.4 Pressure Measuring Device for Calibration, capable of measuring local station pressure with an accuracy and a resolution of 0.1 kPa (1 mm Hg) or better, at the same elevation relative to sea level as the apparatus in the laboratory.

Note 5—This test method does not give full details of instruments suitable for carrying out this test. Details on the installation, operation, and maintenance of each instrument may be found in the manufacturer's manual.

7. Reagents and Materials

7.1 Purity of Reagents—Use chemicals of at least 99 % purity for quality control checks. Commonly used quality control check materials are propane, butane, and pentane (see Section 11). Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society⁵ where such specifications are available. Lower purities can be used,

⁴ The sole source of supply of the apparatus known to the committee at this time is Grabner Instruments, A-1220 Vienna, Dr. Otto Neurathgasse 1, Austria. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee¹, which you may attend.

⁵ Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see Annual Standards for Laboratory Chemicals, BDH Ltd., Poole, Dorset, U.K., and the United States Pharmacopeia and National Formulary, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

provided it is first ascertained that the reagent is of sufficient purity to permit its use without lessening the accuracy of the determination.

Note 6—The chemicals in this section are suggested for quality control procedures (see Section 11) and are not used for instrument calibration.

7.2 Cleaning Solvents—Use noncorrosive solvents capable of cleaning the measuring chamber, the valves, and the inlet and outlet tubes. A commonly used solvent is acetone. (Warning—propane, butane, pentane, and acetone are flammable and health hazards.)

8. Sampling and Sample Introduction

- $8.1\,$ Samples shall be obtained and stored in accordance with Practice D 1265, or IP 181, unless the test samples can be taken directly from the source of the material to be tested. Use a container of not less than $100\,$ mL in size and filled at least $70\,$ % with sample up to a maximum fill density as specified in regulations, which is typically no more than $80\,$ %. Consult the specified regulations for more details.
- 8.2 Any method of coupling the vapor pressure apparatus to the sample source can be employed. Tubing, 3 to 7 mm in diameter, of suitable-working pressure, and made of material corrosion-resistant to the products being sampled, is satisfactory for this purpose. A flexible transparent polyperfluoro-alkoxyethylene (PFA) tubing greatly facilitates the purging and sampling operations.
- 8.3 Practice D 1265 cylinders shall be equilibrated above the sample cell temperature of 5°C to ensure sufficient sample cylinder pressure to fill the sample cell entirely. Practice D 3700 cylinders shall be maintained at a pressure above the vapor pressure of the LPG at 5°C (approximately 655 kPa for special duty propane and approximately 175 kPa for commercial butanes).

9. Preparation of Apparatus

- 9.1 Prepare the instrument for operation in accordance with the manufacturer's instructions. Since LPG is discharged at the outlet of the apparatus, connect a tubing to the outlet and lead the other end of the tubing to a safe exhaust system or a gas recovery system to protect the environment from LPG.
- 9.2 If contaminated, clean the measuring chamber with a solvent. Acetone has been used successfully. Cleaning is performed by drawing the solvent into the chamber by the integrated piston and expelling the solvent into a waste container.

10. Calibration

- 10.1 Pressure Transducer:
- 10.1.1 Check the calibration of the transducer when needed as indicated from the quality control checks performed according to Section 11. The calibration of the transducer is checked using two reference points, zero pressure (that is, < 0.1 kPa) and the ambient barometric pressure.
- 10.1.2 Connect a McLeod gage or a calibrated electronic vacuum-measuring device to the vacuum source in line with the measuring chamber (Note 7). Apply vacuum to the measuring chamber. When the vacuum measuring device registers a pressure less than 0.1 kPa (0.8 mm Hg), adjust the transducer

control to zero or to the actual reading on the vacuum measuring device as dictated by the instrument design or manufacturer's instructions.

Note 7—Refer to Annex A6.3 of Test Method D 2892 for further details concerning the calibration of electronic vacuum measuring devices and proper maintenance of McLeod gages.

- 10.1.3 Open the measuring chamber of the apparatus to atmospheric pressure and observe the corresponding pressure value of the transducer. Ensure that the apparatus is set to display the total pressure and not a calculated or corrected value. Compare this pressure value with the pressure obtained from a pressure-measuring device (see 6.4), as the pressure reference standard. The pressure-measuring device shall measure the local station pressure at the same elevation as the apparatus in the laboratory, at the time of pressure comparison. When the instrument is used over the full pressure range, a calibration with a dead weight balance shall be carried out. (Warning—Many aneroid barometers, such as those used at weather stations and airports, are pre-corrected to give sea level readings. These shall not be used for calibration of the apparatus.)
- 10.1.4 Repeat 10.1.2 and 10.1.3 until the zero and barometric pressures read correctly without further adjustments.
- 10.2 Temperature Sensor—Verify the calibration of the resistance thermometer or equivalent (see 6.1.4) used to monitor the measuring chamber temperature when needed as indicated from the quality control checks performed according to Section 11 against a temperature sensing device which is traceable to National Institute of Standards and Technology (NIST) or national authorities in the country the equipment is used.

11. Quality Control Checks

- 11.1 Use a verification fluid or gas of known vapor pressure as an independent check against the instrument calibration each day the instrument is in use. For pure compounds, multiple test specimens may be taken from the same container over time.
- 11.2 A possible pure gas for verification of the instrument and its corresponding vapor pressure at 37.8°C and a vapor to liquid ratio of 0.5:1 is:

Propane
$$VP_{tot}$$
 (37.8°C) = 1301 kPa⁶

- 11.2.1 If the observed total pressure differs from the reference value by more than 7.0 kPa, check the instrument calibration (see Section 10).
- 11.3 A second possible pure gas for verification of the instrument and its corresponding vapor pressure at 37.8 $^{\circ}$ C and a vapor to liquid ratio of 0.5:1 is:

Butane
$$VP_{tot}$$
 (37.8°C) = 356.5 kPa⁶

11.3.1 If the observed total pressure differs from the reference value by more than 6.0 kPa, then check the instrument calibration (see Section 10).

⁶ Data from Physical Constants of Hydrocarbon and Non-Hydrocarbon Compounds, ASTM Data Series DS 4B, ASTM International, W. Conshohocken, PA.

- 11.4 If a liquid is used to check the performance of the test, cool and air saturate the liquid according to the corresponding sections in the sample preparation procedure of Test Method D 5191.
- 11.5 A possible liquid for verification of the instrument and its corresponding vapor pressure at 70°C and a vapor to liquid ratio of 0.5:1 is:

Pentane
$$VP_{tot}(70^{\circ}\text{C}) = 310 \text{ kPa}$$

11.5.1 If the observed total pressure differs from the reference value by more than 6.0 kPa, check the instrument calibration (see Section 10). (Warning—The values given in 11.2, 11.3, and 11.5 for the vapor pressures of propane, butane, and pentane are the total pressure values. If the instrument reading corresponds to the automatically corrected LPG vapor pressure relative to atmospheric pressure (see 13.2), add 101.3 kPa to the value displayed by the instrument before comparing to the above values for pure compounds.) (Warning—The use of single component verification materials such as listed in 11.2, 11.3, and 11.5 will only prove the calibration of the equipment. It will not check the accuracy of the entire test method, including sample handling, because losses due to evaporation will not decrease the sample vapor pressure for single component materials as happens with losses of light ends in multi-component mixtures.)

Note 8—The value for pentane was derived from the 1999 interlaboratory cooperative test program and represents the total pressure value of the air saturated liquid at the specified temperature.

Note 9—It is recommended that at least one type of verification fluid or gas used have a vapor pressure representative of the samples regularly tested by the equipment. The vapor pressure measurement process (including operator technique) can be checked periodically by performing this test method on previously prepared samples from one batch of product, in accordance with the procedure described in Section 12. Samples should be stored in an environment suitable for long term storage without significant sample degradation. Analysis of result(s) from these quality control samples can be carried out using control chart techniques, such as those outlined in Practice D 6299.

12. Procedure

- 12.1 Connect the pressurized sample container to the inlet of the apparatus and position it in such a way that the outlet valve of the container is below the liquid level. Open the outlet valve of the pressurized sample container.
- 12.2 *Rinsing*—Open the inlet valve and draw in the sample by moving the piston from zero-volume to the filling position. Close the inlet valve and open the outlet valve, move the piston to zero-volume position. Close the outlet valve. Repeat this procedure two more times.
- 12.3 Filling—Regulate the measuring chamber to the filling temperature of 5 ± 0.5 °C. When the measuring chamber is at the filling temperature, close the outlet valve and open the inlet valve. Draw in the sample from the pressurized sample container by moving the piston from zero-volume to the filling position. Close the inlet valve.
- 12.4 Expansion—Move the piston to the final volume to provide the necessary vapor to liquid ratio (the overall volume of the measuring chamber is 1.5 times the fill volume of liquid for a vapor to liquid ratio of 0.5:1).

12.5 Total Pressure Determination—Adjust the temperature regulator of the measuring chamber to the test temperature of interest, such as 37.8° C. After the temperature equilibrium observe the pressure reading. If two consecutive readings remain constant within ± 3 kPa after 1 min, record the observed pressure as total pressure of the sample at test temperature.

13. Calculation

- 13.1 Record the total pressure readings from the instrument.
- 13.2 Calculate the LPG vapor pressures relative to the normal atmospheric pressure of 101.3 kPa by using Eq 1. Ensure that the instrument reading used in this equation corresponds to the absolute pressures (relative to vacuum) and has not been corrected by an automatically programmed correction factor:

$$VP(LPG) = P_{tot} - 101.3 \text{ kPa}$$
 (1)

where:

 P_{tot} = measured total vapor pressure, kPa.

13.3 The calculation described by Eq 1 can be accomplished automatically by the instrument, if so equipped, and in such cases the user shall not apply any further corrections.

14. Report

14.1 Report the corrected total pressure as the LP-Gas vapor pressure test result and specify the test temperature and vapor to liquid ratio if not equal to 0.5:1.

$$VP(T_m \, ^{\circ}C) = #### \, kPa$$

where:

 T_m = measuring temperature.

15. Precision and Bias

- 15.1 *Precision*—The precision of this test method as determined by the statistical examination of interlaboratory test results is as follows:
- 15.1.1 Repeatability—The difference between successive test results obtained by the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following value only in one case in twenty:

Repeatability =
$$7.4 \text{ kPa} (1.1 \text{ psi})$$

Note 10—The repeatability was calculated from triplicate determinations on 20 samples of propane measured under single site repeatability over the range of 300 to 1463 kPa (44 to 212 psig) using a vapor to liquid ratio of 0.5:1, at a temperature of 37.8°C. No trend in variance was detected over this range.

- 15.1.2 *Reproducibility*—Reproducibility will be determined in an interlaboratory cross-check program in accordance with the ASTM requirements within five years.
- 15.2 Relative Bias to Test Method D 1267—Relative bias will be determined in an interlaboratory cross-check program in accordance with the ASTM requirements within five years.

16. Keywords

16.1 expansion method; liquefied petroleum gas; vapor pressure

APPENDIXES

(Nonmandatory Information)

X1. ADDITIONAL CALIBRATION EQUIPMENT FOR PRESSURE TRANSDUCER

X1.1 Dead-Weight Tester—A dead-weight tester of satisfactory range and accuracy of ± 0.1 kPa may be used as a means for checking the accuracy of vapor pressure gages.

X2. WARNING STATEMENTS

X2.1 Propane/Butane, or Mixture of Both

- X2.1.1 Vapors may cause flash fires.
- X2.1.2 Keep away from heat, sparks, and open flame.
- X2.1.3 Keep container closed.
- X2.1.4 Use with adequate ventilation.

- X2.1.5 Avoid buildup of vapors and eliminate all sources of ignition, especially non-explosion-proof electrical apparatus and heaters.
 - X2.1.6 Avoid prolonged breathing of vapor or spray mist.
 - X2.1.7 Avoid prolonged or repeated skin contact.

SUMMARY OF CHANGES

Subcommittee D02.08 has identified the location of selected changes to this standard since the last issue (D 6897 – 03) that may impact its use.

(1) Updated 2.1 to replace the Test Method D 1160 reference with Test Method D 2892.

(2) Updated 6.3 to reference Test Method D 2892 requirements

and 10.1.2 to explicitly reference Note 7.

(3) Added Note 7 and renumbered notes.

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