Designation: D1837 – 17

Standard Test Method for Volatility of Liquefied Petroleum (LP) Gases[1]

This standard is issued under the fixed designation D1837; 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 is a measure of the relative purity of the various types of liquefied petroleum (LP) gases and helps to ensure suitable volatility performance. The test results, when properly related to vapor pressure and density of the product, can be used to indicate the presence of butane and heavier components in propane-type LP-gas, and pentane and heavier components in propane-butane and butane-type fuels. The presence of hydrocarbon compounds less volatile than those of which the LP-gas is primarily composed is indicated by an increase in the 95 % evaporated temperature.

1.2 When the type and concentration of higher boiling components is required, chromatographic analysis should be used.

1.3 The values stated in SI units are to be regarded as the standard.

1.3.1 *Exception*—The non-SI values are provided for information only.

1.4 **WARNING**—Mercury has been designated by many regulatory agencies as a hazardous material that can cause central nervous system, kidney and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury containing products. See the applicable product Safety Data Sheet (SDS) for details and EPA's website—http://www.epa.gov/mercury/faq.htm—for additional information. Users should be aware that selling mercury and/or mercury containing products into your state or country may be prohibited by law.

1.4.1 Note that thallium in a mercury-thallium thermometer is also a hazardous material.

1.5 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D96 Test Method for Water and Sediment in Crude Oil by Centrifuge Method (Field Procedure) (Withdrawn 2000)[3]

D1796 Test Method for Water and Sediment in Fuel Oils by the Centrifuge Method (Laboratory Procedure)

E1 Specification for ASTM Liquid-in-Glass Thermometers

3. Summary of Test Method

3.1 Refrigerate the sample by means of a cooling coil and collect 100 mL of liquid in a weathering tube. Allow to evaporate ("weather") at ambient pressure under specified conditions that approximate a single plate distillation. Measure the observed temperature when 5 mL of liquid test portion remains. Correct this observed temperature for barometric pressure and thermometer ice point error, and report as the 95 % evaporation temperature.

4. Significance and Use

4.1 Volatility, expressed in terms of the 95 % evaporated temperature of the product, is a measure of the amount of least volatile components present in the product. Coupled with a vapor pressure limit, it serves to ensure essentially single-component products in the cases of commercial grades of propane and butane. When volatility is coupled with a vapor pressure limit which has been related to density, as in the case of the commercial PB-mixture, the combination serves to assure essentially two component mixtures for such fuels. When coupled with a proper vapor pressure limit, this measurement serves to assure that special-duty propane products will be composed chiefly of propane and propylene and that propane will be the major constituent.

5. Apparatus

5.1 *Weathering Tube*—A centrifuge tube, cone-shaped, conforming to the dimensions given in Fig. 1 and made of

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.H0 on Liquefied Petroleum Gas.

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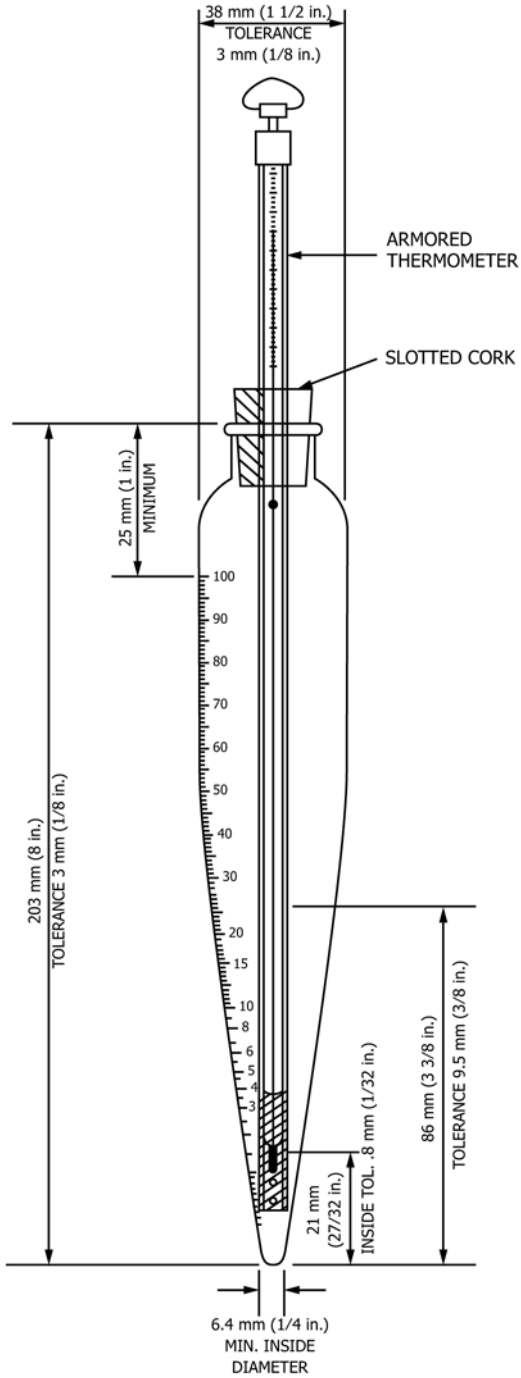
[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

*A Summary of Changes section appears at the end of this standard



NOTE 1—For graduation tolerances see Table 1.

FIG. 1 Weathering Tube

thoroughly annealed heat-resistant glass.[4] The shape of the lower tip of the tube is especially important. The taper shall be uniform and the bottom shall be rounded as shown in Fig. 1. The tubes shall comply in wall thickness to ASTM centrifuge tube requirements (Note 1). The graduation tolerances are given in Table 1.

[4] Borosilicate glass has been found satisfactory for this purpose.

TABLE 1 Weathering Tube Graduation Tolerances

Range, mL	Scale Division, mL	Limit of Error, mL
0.0 to 0.1	0.05	0.02
0.1 to 0.3	0.05	0.03
0.3 to 0.5	0.05	0.05
0.5 to 1.0	0.1	0.05
1.0 to 3.0	0.1	0.1
3.0 to 5.0	0.5	0.2
5.0 to 25.0	1.0	0.5
25.0 to 100.0	1.0	1.0

NOTE 1—Requirements for centrifuge tubes appear in Test Methods D96 and D1796.

5.2 *Tube Support*—Means shall be provided for supporting the weathering tube by its neck in a vertical position.

5.3 *Water Bath* (for use in tests on butane and propane-butane mixture types of liquefied petroleum gas only). A shallow container filled with clean water having a maintained temperature ranging from 15 °C to 21 °C (60 °F to 70 °F) and a depth of 38 mm (1½ in.).

5.4 *Thermometer*—ASTM Armored Weathering Test Thermometer having a range from −50 °C to 5 °C (−58 °F to 41 °F) and conforming to the requirements for Thermometer 99C–92 (99F–86) as prescribed in Specification E1. Do not remove the armor from the thermometer.

5.5 *Barometer*—A pressure measuring device capable of measuring local station pressure with an accuracy of 0.1 kPa (1 mm Hg) or better, at the same elevation relative to sea level as the apparatus in the laboratory. (**Warning—**Do not take readings from ordinary aneroid barometers, such as those used at weather stations and airports, since these are precorrected to give sea level readings.)

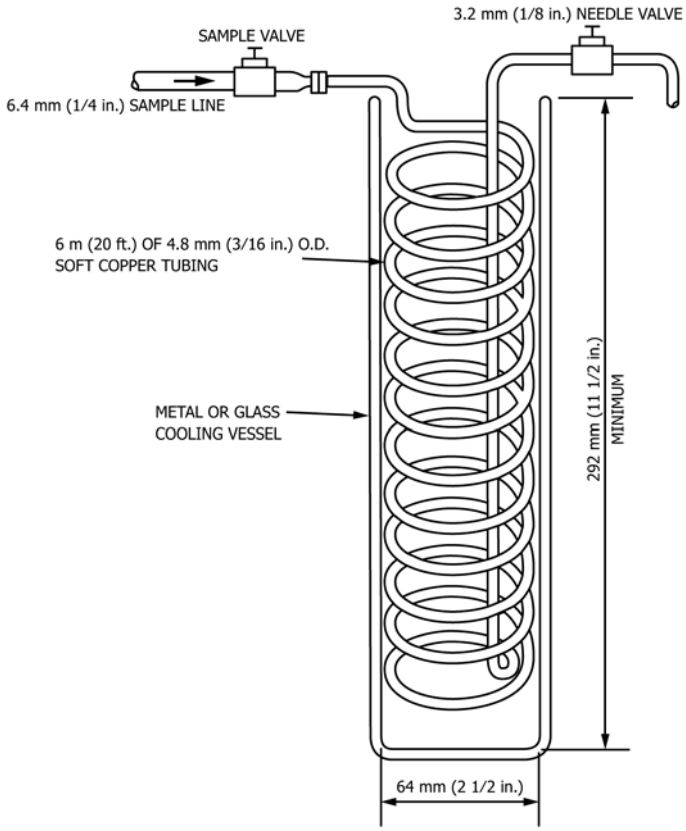
5.6 *Sampling Precooling Equipment:*

5.6.1 *Cooling Vessel*—Any suitable wide-mouthed metal container or Dewar flask at least 64 mm (2½ in.) in inside diameter by 292 mm (11½ in.) deep.

5.6.2 *Cooling Coil*—Approximately 6 m (20 ft) of 4.8 mm (3⁄16 in.) outside diameter soft copper tubing, wound around a hollow mandrel at least 54 mm (2⅛ in.) in outside diameter, with adjacent turns touching. Run the lower end of the tube up through the center of the mandrel before winding so that the finished coil will fit snugly inside the cooling vessel. When assembled, the top of the coil shall be at least 25 mm (1 in.) below the top of the cooling vessel and the open ends of the coil shall not be more than 100 mm (4 in.) above. Connect the downstream end of the coil to a 3.2 mm (⅛ in.) needle valve having an outlet connection not more than 76 mm (3 in.) long (see Fig. 2).

5.6.3 *Precoolant*—This may be the liquefied petroleum gas from the same container from which a sample is to be taken. Other refrigerants having a boiling point lower than the initial boiling point of the sample may be used. Use a nonflammable precoolant if required.

5.7 *Charcoal*—Four grains of activated charcoal, approximately 6 mesh to 14 mesh in size, are required. The four grains shall be similar in size (Note 2).



NOTE 1—The coils in the drawing are extended for clarity. Note additional detailed requirements in 5.6.2.

FIG. 2 Precooling Equipment

NOTE 2—Grains of charcoal are used as a boiling aid which reduces the tendency for the propane or butane to overflow the weathering tube as the propane or butane evaporates.

6. Hazards

6.1 Note that there is a significant fire hazard from LPG vapors, and since the boiling point of LPG can be as low as –41 °C, there is a risk of freezing "burns." Take appropriate safety precautions to prevent ignition or fire, and wear suitable protective equipment to protect against skin contact with liquid or vaporizing LPG.

6.2 When using a cooling bath of dry ice and a hydrocarbon solvent, be aware that the bath can 'bump' or splatter cold liquid when LPG is passed through the tubing.

7. Procedure

7.1 *Positioning the Thermometer*—Add water to the 5 mL line of the weathering tube. Add two grains of charcoal. Insert the armored thermometer as low as possible into the weathering tube. Observe and record the water level in the tube. Remove and discard the water and charcoal and clean and dry the weathering tube.

7.2 *Obtaining a Test Portion:*

7.2.1 Fill the cooling vessel with the precoolant so as to cover the cooling coil.

7.2.2 The source of the sample (test portion) may be a process or delivery line, or a sample cylinder, taking care to obtain a liquid sample.

7.2.3 Attach the inlet of the cooling coil to the source from which the sample is to be taken with a short line connection of 6.4 mm (¼ in.) pipe (or larger), having a sampling valve large enough to prevent vaporization of the material due to the drop in pressure across the valve seat.

7.2.4 Purge the sampling line and cooling coil by opening both the sampling valve and the 3.2 mm (⅛ in.) needle valve on the downstream end of the cooling coil.

7.2.5 Fill the weathering tube with the sample flowing through the cooling coil.

7.2.6 Empty this first sample.

7.2.7 Add two grains of similar-sized charcoal as was used in 7.1, and then refill the weathering tube to the 100 mL mark with fresh liquid sample passing through the cooling coil.

7.3 *Placement of Thermometer*—Carefully insert the precooled armored thermometer into the centrifuge tube, to the same position as in 7.1, and center it in the tube by means of a slotted cork. Take all 5 % residue readings at the level established in 7.1.

NOTE 3—Inadequate precooling of the sample will result in excessive vaporization and loss of light components from the sample collected in the weathering tube. This results in proportionally more of the higher boiling components in the sample in order to collect 100 mL of liquid. As a result, the test is made more severe, with a higher reported 95 % evaporation temperature, which is on the conservative side.

7.4 *Weathering Butane and Propane-Butane Mixture Types of Liquefied Petroleum Gas Products*—When the temperature of the sample is below −12 °C (10 °F), allow it to weather in the atmosphere until the temperature has reached −12 °C (10 °F). At this point, place the weathering tube, with the armored thermometer still in place, in the water bath in a vertical position, submerging it to the 1½ mL mark, and allow the contents to weather.

7.5 *Weathering Propane-Type Liquefied Petroleum Gas Products*—Allow the sample to weather in the atmosphere, taking care to disturb the frost on the tube as little as possible. An acetone or alcohol swab may be used to remove frost sufficient to permit reading of the temperature.

7.6 *Reading of Temperature*—When the liquid level in the weathering tube, with the armored thermometer still in place, corresponds to the level previously determined in 7.1, read and record the temperature of the sample in accordance with 7.4 and 7.5.

7.7 *Temperature Correction*—Following the final temperature reading (7.6), remove the armored thermometer from the weathering tube and place it in a bath of finely crushed ice up to the immersion point. Observe the reading of the thermometer when a constant reading is obtained. If the thermometer reading is less than 0 °C (32 °F), add the fraction of a degree it is low to the final test reading. If the thermometer reading is more than 0 °C (32 °F), subtract the fraction of a degree that it is high from the final test reading. If the thermometer reading varies more than 0.5 °C (1 °F), the test result is invalid. Repeat the procedure using an accurate thermometer.

NOTE 4—A high reading of the thermometer when it is placed in ice usually indicates that there is a break in the mercury-thallium thread. This can be corrected by warming the thermometer gently in a warm water bath to drive the break upward into the expansion chamber at the top of the thermometer. While the mercury-thallium is continuous in the upper chamber, tap the bottom of the thermometer on a hard, but cushioned surface, to join the liquid into a continuous thread. A low reading in ice usually indicates that some of the liquid has remained in the expansion chamber. To correct this, allow the thermometer to warm so the liquid enters the chamber and tap as previously instructed.

8. Interpretation of Results

8.1 Correct the thermometer reading at the 95 % boiling point (5 % residue) for the thermometer error (7.7).

8.2 Correct the observed temperature for the thermometer error from the observed atmospheric pressure in kilopascals (kPa) (millimetres of mercury) to a base barometric pressure of 101 kPa (760 mm).

8.3 In the weathering test for propane, add 0.3 °C (0.6 °F) to the corrected temperature for each 1.3 kPa (10 mm Hg) that the test is conducted below 101 kPa (760 mm) pressure, or subtract 0.3 °C (0.6 °F) from the observed temperature for each 1.3 kPa (10 mm Hg) that the test is conducted above 101 kPa (760 mm).

8.4 In the weathering test for butane and propane-butane mixtures, add 0.4 °C (0.7 °F) to the corrected temperature for each 1.3 kPa (10 mm Hg) that the test is conducted below 101 kPa (760 mm) pressure, or subtract 0.4 °C (0.7 °F) from the observed temperature for each 1.3 kPa (10 mm Hg) that the test is conducted above 101 kPa (760 mm).

9. Report

9.1 Report the corrected 95 % boiling point temperature to 0.1 °C, and reference this test method.

10. Precision and Bias

10.1 The following criteria should be used for judging the acceptability of results (95 % confidence):

10.1.1 *Repeatability*—The difference between two test results obtained by the same operator with the same apparatus under constant operating conditions on identical test materials would in the normal and correct operation of the test method, exceed the following value only in one case in twenty:

0.6 °C (1.0 °F)

10.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories 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:

1.0 °C (1.7 °F) for butane and propane-butane mixtures, and

1.3 °C (2.3 °F) for propane

10.2 *Bias*—The procedure in this test method for measuring volatility of LP-gases has no bias because the volatility is defined only in terms of this test method.

11. Keywords

11.1 butane; liquefied petroleum (LP) gases; LPG; propane; volatility

SUMMARY OF CHANGES

Subcommittee D02.H0 has identified the location of selected changes to this standard since the last issue (D1837 – 11) that may impact the use of this standard. (Approved Jan. 1, 2017.)

(1) Updated all numbers and symbols to proper SI form and style.
(2) Revised subsection 1.3 by adding new subsection 1.3.1, to make the units statement consistent with current practice.
(3) In subsection 1.4, changed Material Safety Data Sheet (MSDS) to Safety Data Sheet (SDS).
(4) Added new subsection 1.4.1 to note that thallium is also a hazardous material.
(5) Revised subsection 3.1 for clarity.
(6) Added subsection 5.5, Barometer (description from Test Method D86), and re-numbered subsequent sections.
(7) Modified Note 1 to Fig. 2 to emphasize the detailed requirements in subsection 5.6.2.
(8) Added Section 6, Hazards.
(9) Revised subsection 8.1.
(10) Added Section 9, Report.