



Designation: D2158 – 11

Designation: 317/95

Standard Test Method for Residues in Liquefied Petroleum (LP) Gases¹

This standard is issued under the fixed designation D2158; 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 determination of extraneous materials weathering above 38°C that are present in liquefied petroleum gases. The extraneous materials will generally be dissolved in the LPG, but may have phase-separated in some instances.

1.2 Liquefied petroleum gases that contain certain anti-icing additives can give erroneous results by this test method.

1.3 Although this test method has been used to verify cleanliness and lack of heavy contaminants in propane for many years, it might not be sensitive enough to protect some equipment from operational problems or increased maintenance. A more sensitive test, able to detect lower levels of dissolved contaminants, could be required for some applications.

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 Material Safety Data Sheet (MSDS) 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.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 *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.*

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.H0 on Liquefied Petroleum Gas.

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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)

D1835 Specification for Liquefied Petroleum (LP) Gases

E1 Specification for ASTM Liquid-in-Glass Thermometers

2.2 *Energy Institute Document*:

IP Test Methods - Appendix A. Specifications - IP standard thermometers⁴

3. Terminology

3.1 *Definitions of Terms Specific to This Standard*:

3.1.1 *oil stain observation, n*—the volume of solvent-residue mixture required to yield an oil stain or ring that persists for 2 minutes under specified conditions on absorbent paper.

3.1.2 *residue, n*—the volume, measured to the nearest 0.05 mL, of the residual material boiling above 38°C resulting from the evaporation of 100 mL of sample under the specified conditions of this test method.

3.1.3 *solvent-residue mixture, n*—a mixture (solution) of 10 mL of solvent with any residue remaining in the centrifuge tube at the conclusion of the first step in this test method.

4. Summary of Test Method

4.1 A 100-mL sample of liquefied petroleum gas is weathered in a 100-mL centrifuge tube. The volume of residue remaining after heating the tube to 38°C is measured and recorded.

4.2 To dissolve any residue, 10 mL of solvent is added to the centrifuge tube. Small, measured volumes of solvent-residue

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.

⁴ Available from Energy Institute, 61 New Cavendish St., London, WIG 7AR, U.K. www.energyinstpubs.org.uk

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

mixture are deposited on an absorbent paper in a specified manner. The appearance of the absorbent paper to which the residue solution has been added in measured increments is observed and recorded.

5. Significance and Use

5.1 Control over the residue content (required by Specification D1835) is of considerable importance in end-use applications of LPG. In liquid feed systems residues can lead to troublesome deposits and, in vapor withdrawal systems, residues that are carried over can foul regulating equipment. Those that remain will accumulate, can be corrosive, and will contaminate following product. Water, particularly if alkaline, can cause failure of regulating equipment and corrosion of metals.

6. Interferences

6.1 Solid contaminants such as rust, scale or dirt can interfere with this test method, which is not intended for representative measurement of solid, undissolved contaminants.

7. Apparatus

7.1 *Centrifuge Tube*, 100-mL graduated, conforming to dimensions given in Fig. 1. The first 0.5 mL shall be graduated in 0.05-mL increments. 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. Tubes shall be made of thoroughly annealed heat-resistant glass. Volumetric graduation tolerances, based on air-free water at 20°C, are

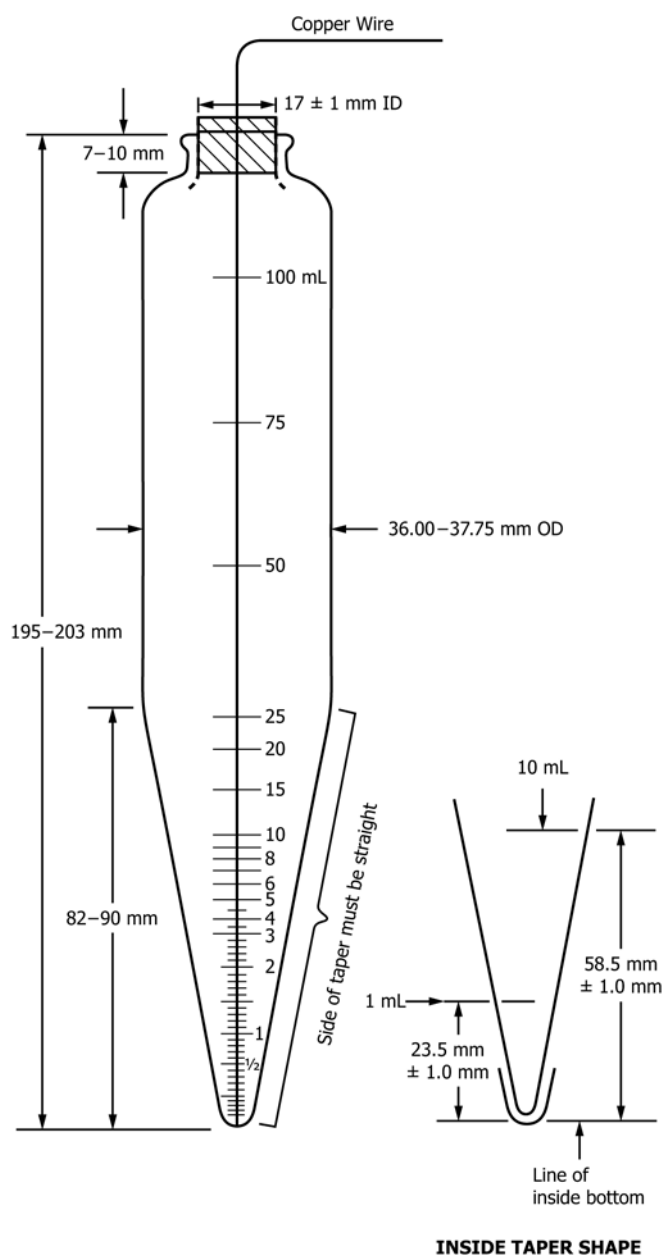


FIG. 1 Cone-Shaped Centrifuge Tube, 203 mm

given in Table 1. Detailed requirements for centrifuge tubes appear in Test Methods D96 and D1796.

7.2 *Cooling Coil and Cooling Bath*, a minimum length of 6 m of 5 to 7-mm outside diameter copper tubing wound to a diameter of 63.5 ± 1.5 -mm outside diameter, and assembled in a suitable cooling bath. (See Fig. 2 as an example.)

7.2.1 Mechanical refrigeration is permitted provided that the coolant temperature is below -43°C . If dry ice is used, a non-glass dewar or vessel is recommended.

7.3 *Syringe*, 2-mL graduated in 0.1 mL and equipped with a needle 200 ± 5 mm long. The needle may be either a sharp needle (ordinary medical syringe needle) or a safe, non-sharp syringe needle to avoid a puncture hazard. Alternatively, an equivalent liquid dispensing device capable of delivering 0.1-mL increments may be used, such as a 0.1-mL pipette.

7.4 *Temperature Measuring Device*, that is intrinsically safe, with accuracy equal to or better than Specification E1 or IP Test Methods - Appendix A shall be used. Thermometers conforming to Specification E1 or IP Appendix A have been found to be satisfactory.

| | | |
|------------|--|-----------------------------------|
| Low Range | -38°C to $+50^{\circ}\text{C}$ | IP 1C/ASTM 5C or IP 2C/ASTM 6C |
| High Range | -20°C to $+50^{\circ}\text{C}$ | ASTM 57C |

NOTE 1—When a thermometer or a water bath, or both, are not available, for example when conducting a field test, a satisfactory alternative for screening purposes is to warm the tip of the centrifuge tube with the hand.

7.4.1 For routine testing, a general purpose thermometer with 0.5°C subdivisions and a maximum scale error of 0.5°C may be used.

7.5 *Absorbent Paper*, white, at least 100-mm diameter. Medium grade or rapid filter paper has been found to be satisfactory. In this test method, the paper will be referred to as “filter paper.”

7.6 *Solvent Wash Bottle*, typically polyethylene.

7.7 *Water Bath*, controlled at $38 \pm 2^{\circ}\text{C}$.

7.8 *Copper Wire*, 1 to 2-mm diameter, at least 10 mm longer than the centrifuge tube’s height.

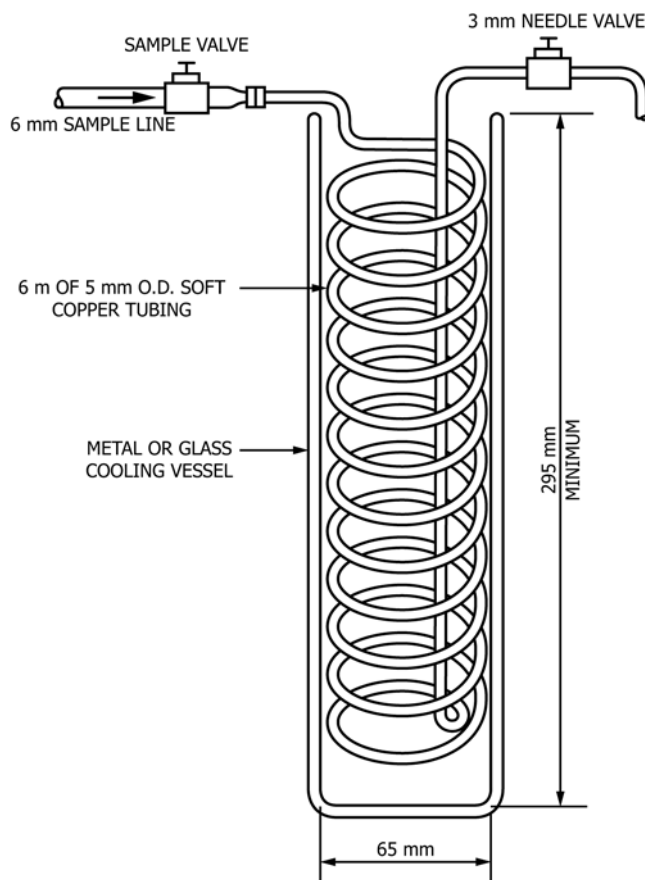
7.9 *Clamp*, suitable for holding the centrifuge tube during weathering.

8. Reagents and Materials

8.1 *Solvent*—HPLC-grade pentane or cyclopentane. Another grade of solvent may be used provided that it meets the requirements of 10.2.

TABLE 1 Centrifuge 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—Coils in the drawing are extended for clarity.

FIG. 2 Precooling Equipment

8.1.1 Although pentane is the preferred solvent for use in this test method, cyclopentane may be substituted for pentane whenever the ambient temperature or altitude is too high to enable the convenient handling of pentane.

8.1.2 Storage of solvent in a polyethylene wash bottle for several days contaminates the solvent. Therefore, do not use any solvent that has remained in a polyethylene wash bottle for more than one day.

9. Hazards

9.1 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.

9.2 Operators should wear a grounded antistatic wrist strap. The use of an antistatic floor mat and grounding the sample cylinder are also advised.

10. Preparation of Apparatus

10.1 Wash all glassware that is to be used in the test in the selected solvent.

10.2 Verification of cleanliness of glassware and solvent.

10.2.1 Add 10 mL of a new sample of solvent to the centrifuge tube.

10.2.2 Mark the center of the filter paper with a pencil or other non-pentane-soluble writing tool.

10.2.3 Fill the syringe or equivalent liquid dispensing device (see 7.3) with a portion of the solvent drawn from the centrifuge tube and direct 0.1-mL portions of the solvent to the mark on the paper.

10.2.3.1 The solvent is added in 0.1-mL increments to confine the solvent ring to a circle about 30 to 35 mm in diameter. The filter paper should be held level during the solvent addition. One method is to place it on a 250-mL beaker.

10.2.4 Allow the solvent to evaporate for 2 min, and note the persistence of an oil ring.

10.2.4.1 The presence of an oil ring shall be observed by holding the dry filter paper between the eye and a bright incandescent light or strong daylight.

10.2.4.2 If no oil ring appears after 1.5 mL of solvent has been added, the solvent and glassware are satisfactory.

10.2.4.3 The appearance of an oil ring indicates either improperly cleaned glassware or contaminated solvent.

11. Procedure

11.1 Residue Measurement:

11.1.1 If the LPG sample is expected to be clean, with no residues or contaminants that could result in an oil stain, the procedure may be performed without a cooling coil. However, if a residue or oil stain is detected using this screening procedure (that is, a residue is measured at greater than or equal to 0.05 mL in 11.1.7, or an oil stain is generated with 1.5 mL of test solution in 11.2.4), the test shall be repeated using a cooling coil.

11.1.2 Attach the cooling coil to the LPG sample source, cool the coil to below the boiling point of the sample, and flush the coil and sampling line.

11.1.3 Rinse and cool the centrifuge tube with the material to be sampled and then fill it to the 100-mL mark with a representative sample of LPG.

11.1.4 Immediately insert the copper wire through a clean, slotted cork or a clean, loose-fitting plug of cotton or cleansing tissue in the mouth of the centrifuge tube. The wire helps to prevent superheating and resulting bumping (erratic or excessive boiling), and the cork (or plug) will keep out air or moisture while the sample is weathering.

11.1.5 If more than 10 mL of the sample is lost because of bumping, obtain a new sample and repeat the test.

11.1.6 Allow the sample to weather, using artificial heating if the ambient temperature or type of sample requires it. If, when weathering has ceased and the tube has reached ambient temperature, a visible residue remains, place the tip of the tube in a water bath at 38°C for 5 min.

11.1.7 Record the volume of any remaining residue to the nearest 0.05 mL, and the presence of extraneous matter, if observed.

11.1.8 Perform the oil stain observation described in 11.2 even if there is no apparent or visible residue in the centrifuge tube. Experience has shown that there can be a thin film of oil on the inner surface of the centrifuge tube that is difficult to see

and does not give a measurable volume, but can still give a ring or stain in the Oil Stain Observation procedure.

11.2 Oil Stain Observation:

11.2.1 Add sufficient solvent to the centrifuge tube containing the residue described in 11.1.6 to restore the volume to 10 mL. Add the solvent from the wash bottle and carefully wash down the sides of the tube. Stir well so that any residue at the bottom of the tube is dissolved uniformly in the solvent. Stirring with the syringe needle (see 7.3) or pipette has been found satisfactory. This mixture will be referred to as the solvent-residue mixture.

11.2.2 Mark the center of a clean white filter paper.

11.2.3 Fill the syringe or equivalent liquid dispensing device (see 7.3), and direct 1.5 mL of the solvent-residue mixture at the center of the paper at an appropriate rate such that the wetted circle is maintained at about 30 to 35 mm in diameter. If desired, steps 11.2.3 and 11.2.4 may be omitted, and the test continued according to 11.2.5.

11.2.4 After directing the entire 1.5 mL of residue solution onto the filter paper and at the end of a 2-min waiting period, if no oil ring persists when holding the dry filter paper between the eye and a bright incandescent light or strong daylight, discontinue the test and go to 12.1.

11.2.5 If a stain or ring is discernible, determine the volume of the solvent-residue mixture at which the oil stain or ring first persists for 2 min on a new filter paper by adding the solvent-residue mixture in 0.1-mL increments, waiting 2 min after each addition, and inspecting the paper for an oil stain at the end of the 2-min period.

11.2.6 Record the volume in millilitres of the solvent-residue mixture required to yield a persistent oil stain or ring as the oil stain observation.

11.2.7 Alternatively, if a product specification calls for adding a specified amount of solvent-residue mixture to the filter paper, add the specified quantity in 0.1 mL increments, and report the result at the specified total amount.

11.2.7.1 Specification D1835 specifies that 0.3 mL of solvent-residue mixture shall be deposited on the filter paper in 0.1-mL increments.

11.2.7.2 If there is no oil stain after the addition of 0.3 mL and a 2-min waiting period, report the result as "Pass."

11.2.7.3 If an oil stain is observed at 0.3 mL after a 2-min waiting period, report the result as "Fail."

11.3 Any solvent transferred to the wash bottle for purposes of running the test shall either be used in testing during the same day or discarded.

11.4 It has been noted that at low ambient temperatures (below about 5°C) materials in the gasoline boiling range will leave an oil stain or ring that persists after 2 min. Oil stain determinations should be made in a protected area where the temperature is above 5°C. If it is necessary to determine the oil stain at temperatures below 5°C, allow 10 min for oil stain persistence.

12. Report

12.1 Report the results as:

12.1.1 Residue on evaporation to the nearest 0.05 mL, and

12.1.2 Oil stain observation to the nearest 0.1 mL.

12.2 Alternatively, if the application of this test method calls for passing or failing the oil stain requirement at a specified volume of solvent-residue mixture, report the result at the specified volume as “Pass” or “Fail” (see 11.2.7).

13. Precision and Bias

13.1 *Precision*—Precision has not been determined on this method in terms of residue and oil stain results. Precision had been determined in terms of R and O numbers, as given in [Appendix X1](#).

13.2 *Bias*—The procedure in this test method for measuring residues in LP Gas has no bias, because the residues are defined only in terms of this test method.

14. Keywords

14.1 contaminants; liquefied petroleum gases; LPG; oil stain; residue

APPENDIX

(Nonmandatory Information)

X1. CALCULATION OF R AND O NUMBERS

INTRODUCTION

Historically, D2158 has included indices for R and O which give the residue and oil stain results in whole numbers rather than the decimal results of this test method. These results have been referred to as “normalized results.” Since no ASTM standards call for R or O results, the description and calculation of these values have been moved to an appendix for information.

X1.1 Terminology

X1.1.1 *Definitions of Terms Specific to this Standard:*

X1.1.1.1 *O Number*, n , 10 divided by the oil stain observation (in millilitres).

X1.1.1.2 *R Number*, n , the residue volume in millilitres multiplied by 200.

X1.2 Calculation and Reporting

X1.2.1 *O Number*—Divide 10 by the oil stain observation obtained in 11.2.6, in millilitres, and report the result to the nearest whole number. If the oil stain observation exceeds 1.5 mL, record the result as zero.

X1.2.2 *R Number*—Multiply the residue volume obtained in 11.1.6, in millilitres, by 200, and express the result to the nearest 10.

X1.3 Precision and Bias

X1.3.1 Precision has been determined in terms of the normalized reporting units.

X1.3.2 *Repeatability*—The difference (r) between successive test results obtained by the same operator with 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 values below only in one case in twenty:

| O Number | r | R Number | r |
|-----------|-----|----------|-----|
| 0 to 20 | 4 | 0 to 20 | 5 |
| 20 to 40 | 6 | 20 to 40 | 10 |
| 40 to 100 | 8 | 40 to 60 | 20 |

X1.3.3 *Reproducibility*—The difference (R) between two test results independently obtained by different operators operating in different laboratories on nominally identical test material would, in the long run, in the normal and correct operation of the test method, exceed the values below only in one case in twenty:

| O Number | R | R Number | R |
|-----------|-----|----------|-----|
| 0 to 20 | 6 | 0 to 20 | 10 |
| 20 to 40 | 8 | 20 to 40 | 20 |
| 40 to 100 | 12 | 40 to 60 | 30 |

SUMMARY OF CHANGES

Subcommittee D02.H0 has identified the location of selected changes to this standard since the last issue (D2158–05) that may impact the use of this standard.

(1) Revised 7.5 and 7.8.

(2) Added new 11.1.1 and renumbered rest of Section 11.

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