An American National Standard

Designation: D 2158 – 02~~4~~

Designation: 317/95

Standard Test Method for Residues in Liquefied Petroleum (LP) Gases[1]

This standard is issued under the fixed designation D 2158; 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 the extraneous materials weathering above 38°C that are present in liquefied petroleum gases.

1.2 Liquefied petroleum gases that contain alcohols to enhance their anti-icing behavio~~u~~r can give erroneous results by this test method.

1.3 The result can be expressed in terms of measured volumes or indices derived from these volumes. In either case, the test method provides an indication of the quantity and nature of materials in the product that are substantially less volatile than the liquefied petroleum gas hydrocarbons.

1.4 Although this test method has been used to verify cleanliness and lack of heavy contaminants in propane for many years, it may 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, may be required for some applications.

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.* ~~For specific precautionary statements, see 6.9.~~

2. Referenced Documents

2.1 *ASTM Standards:* [2]

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[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.

Current edition approved ~~April 10, 2002.~~ May 1, 2004. Published ~~June 2002.~~ May 2004. Originally ~~published as D 2158 – 63 T.~~ approved in 1963. Last previous edition ~~D 2158 – 97$^{\epsilon 1}$.~~ approved in 2002 as D 2158–02.

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

D 96 Test Methods for Water and Sediment in Crude Oil by Centrifuge Method (Field Procedure)[3]
D 1796 Test Method for Water and Sediment in Fuel Oils by the Centrifuge Method (Laboratory Procedure)
D 1835 Specification for Liquefied Petroleum (LP) Gases
E 1 Specification for ASTM Liquid-in-Glass Thermometers
2.2 *Other Documents:*
IP Appendix A–[4]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 ~~*residue*~~*O Number* ~~—the volume, measured to~~—10 divided by 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.~~ oil stain observation.

3.1.2 ~~*R Number —the residue multiplied by 200.*~~

~~3.1.3 *oil stain observation*~~—the volume of solvent-residue mixture required to yield an oil ring that persists for 2 min under specified conditions on a prescribed filter paper.

3.1.3 *R Number* —the residue multiplied by 200.

3.1.4 ~~*O Number residue*—10 divided by~~ —the volume, measured to the nearest 0.05 mL, of the residual ~~s~~ material boilin ~~og~~ abs ove 38°C resulting from the evaporation of 100 mL of sample under the specified conditions of 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 at 38°C is measured and recorded as is also the appearance of a filter paper to which the residue has been added in measured increments.

5. Significance and Use

5.1 Control over the residue content (required by Specification D 1835) is of considerable importance in end-use applications. In liquid feed systems residues may lead to troublesome deposits and, in vapor offtake 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. Apparatus

6.1 *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 given in Table 1. Detailed requirements for centrifuge tubes appear in Test Methods D 96 and D 1796.

6.2 *Cooling Coil*, 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.)

NOTE 1—Mechanical refrigeration is permitted provided that the coolant temperature is in the range from −46 to −48°C.

6.3 *Syringe*, 2-mL (ordinary medical syringe), graduated in 0.1 mL and equipped with a needle 200 ± 5 mm long. Alternatively, an equivalent liquid dispensing device capable of delivering 0.1- mL ~~p~~ ipncrements may be used, such as a 0.1-mL pipet.

6.4 *Thermometers*, conforming to Specification E 1 or IP Appendix ~~A.~~ A.

Low Range-Minus 38°C to + 50°C	IP 1C/ASTM 5C or IP 2C/ASTM 6C
High Range-Minus 20°C to + 50°C	ASTM 57C

NOTE 2—When a thermometer or a water bath, or both, are not available, for example, a field test, a satisfactory alternative for screening is to warm the tip of the centrifuge tube with the hand.

NOTE 3—For routine testing, a general purpose thermometer with 0.5°C subdivisions and a maximum scale error of 0.5°C may be used.

NOTE 4—Intrinsically safe digital thermometers with accuracies equal or better than Specification E 1 or IP Appendix A may also be used.

6.5 *Filter Paper*, medium-grade, rapid, white, 125-mm diameter.

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[3] ~~Discontinued; see~~

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For ~~*2000 Annual*~~ *Annual Book of ASTM Standards~~, Vol 05.01.~~ volume information, refer to the standard's Document Summary page on the ASTM website.*

~~*Annual Book of ASTM Standards, Vol 05.01.*~~

[3] Withdrawn.

~~*Annual Book of ASTM Standards,* Vol 14.03.~~

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

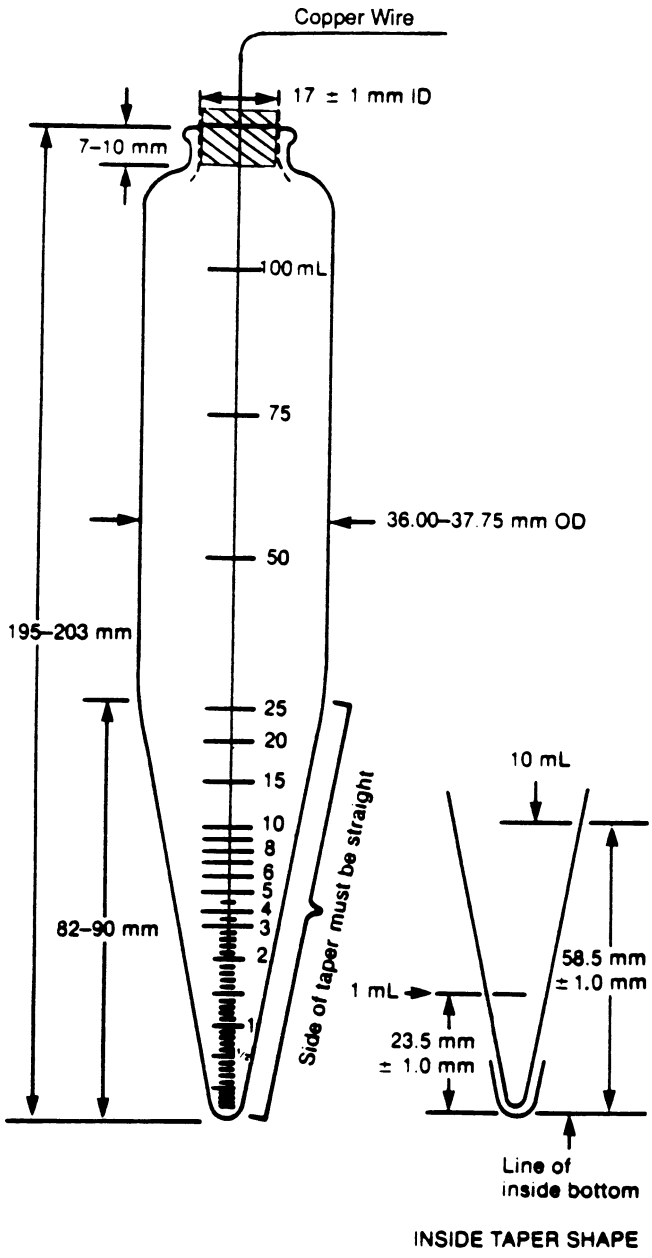


FIG. 1 Cone–Shaped Centrifuge Tube, 203 mm

NOTE 5—The technical description of the filter paper is currently under review.

6.6 *Solvent Wash Bottle.*

6.7 *Water Bath,* controlled at 38 ± 2°C.

6.8 *Copper Wire,* 1.6 ± 0.1-mm diameter, 300 ± 10-mm long.

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

7. Reagents and Materials

7.1 *Solvent*—Oil-free, reagent-grade pentane or cyclopentane.

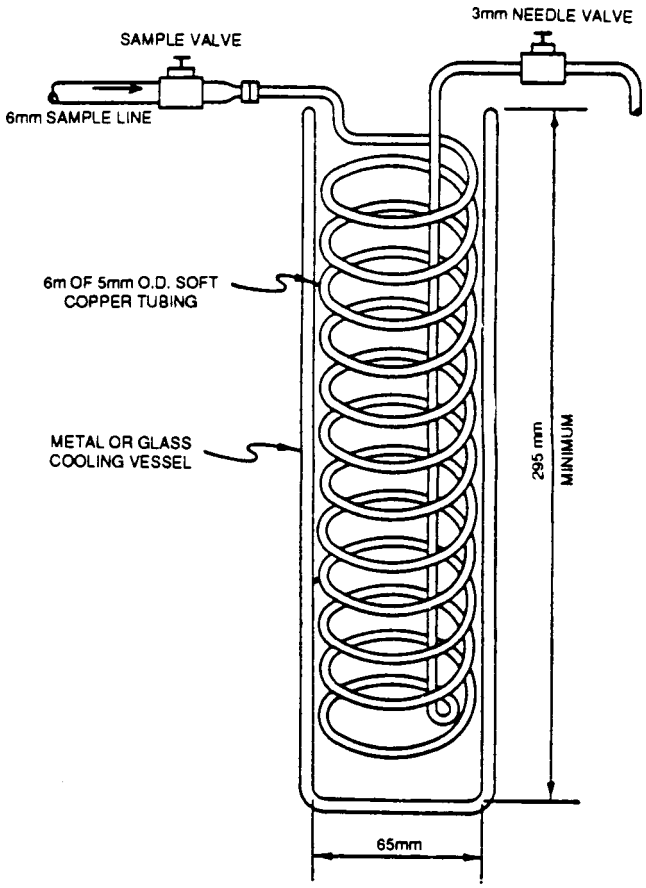
NOTE 6—Although pentane is the preferred solvent for use in this test method, cyclopentane can be substituted for pentane whenever the ambient temperature or altitude is too high to enable the convenient handling of pentane.

8. Preparation of Apparatus

8.1 Wash all glassware that is to be used in the test in the selected solvent. Add 10 mL of a new sample of solvent to the centrifuge tube. Mark the center of the filter paper. Fill the syringe or ~~pipet~~ equivalent liquid dispensing device (see 6.3) with a portion of the solvent drawn from the centrifuge tube and direct 0.1 mL of the solvent to the mark on the paper. Allow the solvent

TABLE 1 Centrifuge Tube Graduation Tolerances

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

to evaporate and note the persistence of an oil ring. Attempt to cover a circle of about 30 to 35 mm in diameter on the filter paper with each addition. If no oil ring appears after 1.5 mL of solvent has been added, the solvent and glassware are satisfactory. The appearance of an oil ring indicates either improperly cleaned glassware or contaminated solvent.

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

8.3 The solvent is added in 0.1-mL increments to confine the solvent ring to a circle of 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 the 250-mL beaker.

9. Procedure

9.1 *Residue*—Attach the cooling coil to the sample source, cool the coil to below the boiling point of the sample, and flush the coil and sampling line.

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

9.1.2 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. If more than 10 mL of the sample is lost because of bumping, obtain a new sample.

9.1.3 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 residue remains, place the tip of the tube in a water bath at 38°C for 5 min.

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

9.1.5 Perform the oil stain observation described in 9.2 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.

9.2 *Oil Stain Observation*—Add sufficient solvent to the centrifuge tube containing the residue described in 9.1.4 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 ~~with the syringe needle or pipet~~ so that any residue at the bottom of the tube is dissolved uniformly in the solvent. Stirring with the syringe needle (see 6.3) or pipet has been found satisfactory.

9.2.1 Mark the center of a clean white filter paper. Fill the syringe or ~~pipet~~ equivalent liquid dispensing device (see 6.3), 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.

9.2.2 If no oil ring persists after directing the entire 1.5 mL on the filter paper and a 2-min waiting period when holding the dry filter paper between the eye and a bright incandescent light or strong daylight, discontinue the test.

9.2.3 If a ring is discernible, determine the volume of the solvent-residue mixture at which the oil ring first persists for 2 min on a new filter paper by adding the solvent-residue mixture in 0.1-mL increments.

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

9.3 Storage of oil-free solvent in a polyethylene wash bottle for several days contaminates the solvent. Any solvent transferred to the wash bottle for purposes of running the test should either be used in testing during the same day or discarded.

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

NOTE 7—As an acceptable alternative to the procedure given in 9.2 for use in those cases where a product specification limit has been established, continued incremental additions of the solvent-residue mixture that is equivalent to the limiting specification can be made to the filter paper and, if no persistent oil ring appears, the result of the test shall be reported as passing.

10. Calculation

10.1 *R Number*—Multiply the volume of residue obtained in 9.1.4 by 200.

10.2 *O Number*—Divide 10 by the oil stain observation obtained in 9.2.4. If the oil stain observation exceeds 1.5 mL, the result is recorded as zero.

11. Expression of Results

11.1 *Volumetric*—The results shall be expressed as:

11.1.1 Residue on evaporation to the nearest 0.05 mL, and

11.1.2 Oil stain observation to the nearest 0.1 mL.

11.2 *Normalized*—The results shall be expressed as:

11.2.1 R Number to the nearest 10, and

11.2.2 O Number to the nearest 1.

12. Precision and Bias

12.1 Precision is only expressed in terms of the normalized reporting units.

12.2 *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 ~~0 to 20~~	4 ~~4~~	0 to 20 ~~0 to 20~~	5 ~~5~~

20 to 40	6	0 to 20	5
20 to 40	6	20 to 40	10
40 to 100	8	40 to 60	20

12.3 *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	6	0 to 20	0 to 20	6
20 to 40	8	20 to 40	20	
40 to 100	12	40 to 60	30	

12.4 *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.

13. Keywords

13.1 liquefied petroleum gases; LPG; residue

SUMMARY OF CHANGES

Subcommittee D02.H0 has identified the location of selected changes to this standard since the last issue (D 2158–02) that may impact the use of this standard.

(1) Updated 6.2, 6.3, 8.1, 9.2, and 9.2.1.