



Designation: D322 – 97 (Reapproved 2016)

Standard Test Method for Gasoline Diluent in Used Gasoline Engine Oils by Distillation¹

This standard is issued under the fixed designation D322; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This test method covers determination of the amount of dilution in crankcase oils of engines when gasoline has been used as the fuel.

NOTE 1—There may be cases in dispute, therefore, the user of this test method is advised to establish whether this method will be accepted. There may be cases where Test Method D3525 results will be set as the referee value.

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

1.3 *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 statement, see 6.4, 7.1, and 9.3.

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 ASTM Standards:²

D235 Specification for Mineral Spirits (Petroleum Spirits) (Hydrocarbon Dry Cleaning Solvent)

D3525 Test Method for Gasoline Diluent in Used Gasoline Engine Oils by Gas Chromatography

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

2.2 *IP Methods for Analyses and Testing, Vol II, Part I³*

3. Terminology

3.1 Definitions:

3.1.1 *fuel diluent, n*—in used oil analysis, unburnt fuel components that enter the engine crankcase causing dilution of the oil.

3.1.1.1 *Discussion*—In this test method, the fuel diluent components being determined are from gasoline.

3.1.2 *used oil, n*—any oil that has been in a piece of equipment (for example, an engine, gearbox, transformer, or turbine) whether operated or not. **D4175**

3.1.2.1 *Discussion*—In this test method, used oil is from a gasoline engine.

4. Summary of Test Method

4.1 The sample, mixed with water, is placed in a glass still provided with a reflux condenser discharging into a graduated trap connected to the still. Heat is applied, and the contents of the still are brought to boiling. The diluent in the sample is vaporized with the water and then liquefied in the condenser. The diluent collects at the top of the trap, and the excess water runs back to the still where it is again vaporized, carrying over an additional quantity of diluent. The boiling is continued until all the diluent has been boiled out and recovered in the trap. The volume is recorded.

5. Significance and Use

5.1 Some fuel dilution of the engine oil may take place during normal operation. However, excessive fuel dilution is of concern in terms of possible performance problems.

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 D02.06 on Analysis of Liquid Fuels and Lubricants.

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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 Available from Energy Institute, 61 New Cavendish St., London, WIG 7AR, U.K., <http://www.energyinst.org.uk>.

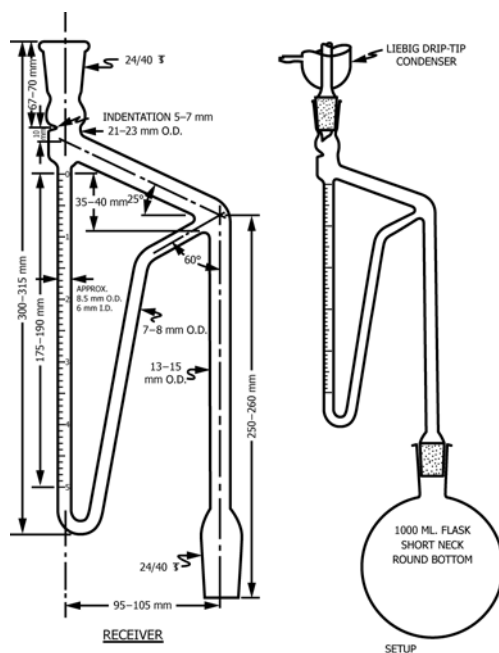


FIG. 1 Apparatus for Determining Diluent in Gasoline Engine Crankcase Oil

6. Apparatus

- 6.1 *Flask*, round-bottom type (see Fig. 1 and A1.1).
- 6.2 *Condenser*, Liebig straight-tube type (see A1.2).
- 6.3 *Trap*, 5 mL, graduated in 0.1 mL increments (see Fig. 1 and Fig. 2 and A1.3).
- 6.4 *Heater*—Any suitable gas burner or electric heater may be used with the glass flask. (**Warning**—Hot exposed surface. Avoid contact by wearing protective equipment as required.)

7. Reagents and Materials

- 7.1 *Ethanol, Denatured*, conforming to either Formula No. 30 or 3A of the US Bureau of Internal Revenue. (**Warning**—Flammable. Denatured. Cannot be made non-toxic.)
- 7.2 *Mineral Spirits (Petroleum Spirits)*, conforming to Specification D235.

NOTE 2—In Annex A1.3, the use of reagent grade heptane may be the preferred solvent because the use of commercial grade heptane or mineral spirits can cause complications of container disposal that may not be required for the disposal of the containers for reagent grade heptane.

8. Sampling

- 8.1 Obtain a representative sample using either Practices D4057 or D4177.

9. Procedure

- 9.1 Mix the sample thoroughly, measure $25 \text{ mL} \pm 5 \text{ mL}$ by means of a 25 mL graduated cylinder, and transfer as much as possible of the contents of the cylinder by pouring it into the

flask. Wash the graduated cylinder with successive portions of hot water until only a negligible amount of oil is left in the cylinder. Add additional water to the flask to make a total of approximately 500 mL of water. Fill the trap with cold water and add $1 \text{ mL} \pm 0.1 \text{ mL}$ of denatured ethanol to the water in the trap.

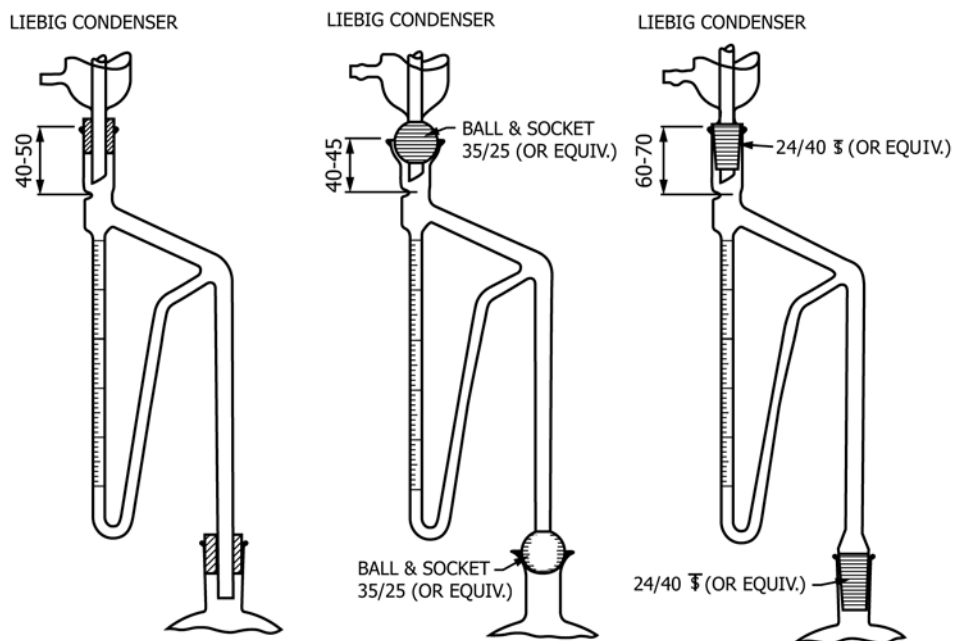
- 9.2 Assemble the apparatus as shown in Fig. 1, so that the tip of the condenser is directly over the indentation in the trap.

9.3 Apply heat to the flask at such a rate that refluxing starts within 7 min to 10 min after heat is applied, with the water and sample being at 21°C to 38°C prior to application of heat. After boiling and condensation has commenced, adjust the rate of boiling so that condensed distillate is discharged from the condenser at a rate of 1 to 3 drops per s. (**Warning**—Hot exposed surface. Avoid contact by wearing protective equipment as required.)

NOTE 3—Bumping with a tendency to froth over is often experienced with dirty oils. The use of *boiling stones*, steel wool, or about 5 mL of concentrated hydrochloric acid (HCl) in the flask is often helpful in eliminating this difficulty.

- 9.4 Obtain readings of the amount of diluent at the following times, taken from the time that refluxing starts: 5 min, 15 min, and 30 min, ± 1 min, and each 15 min, ± 1 min, following until the test is complete. Completion of the test shall be determined on the basis of either or both of the following criteria:

9.4.1 The test is complete when the volume of diluent increases by not more than 0.1 mL in any 15 min period during the course of the test.



All dimensions are in millimetres

FIG. 2 Alternate Closures for Diluent Trap

9.4.2 The test is complete when the volume of diluent obtained in a given time indicates completion, as follows:

Time from Start of Refluxing	Test is Complete if Apparent Volume of Diluent Collected is Equal to or Less Than:
5 min	no visible diluent ^A
30 min	2.0 mL
60 min	4.0 mL
90 min	5.0 mL

^A Report as "no dilution;" otherwise the test should be continued at least 30 min.

9.5 When the test continues without reaching the limit defined in 9.4.1, to a point at which any of the conditions described in 9.4.2 are encountered, the latter shall define the completion of the test.

9.6 When the test is complete by either of the criteria defined in 9.4.1 and 9.4.2, turn off the heat. Allow the equipment to stand for at least 30 min to allow the distillate to separate completely and to cool to approximately room temperature. Read the volume of diluent collected in the trap. If the volume of diluent exceeds the calibrated capacity of the trap, discontinue the test and report the results as 20 % plus.

10. Calculation

10.1 The diluent content of the sample, expressed as volume percent, is equal to the volume of diluent in millilitres multiplied by 4.

NOTE 4—In some cases with samples containing large amounts of diluent, equipment limitations do not permit collection and measurement of the full 5 mL of diluent even when more is present. This condition exists when the upper limit of the collected diluent is above the zero

calibration mark on the trap. When it occurs, finish the test as prescribed in 9.6, read the maximum volume of diluent collected, calculate the corresponding percentage x , and report the results as x percent plus.

11. Report

11.1 Report the result as the Diluent Content, ASTM D322.

12. Precision and Bias

12.1 The precision of this test method as obtained by statistical examination of interlaboratory test results is as follows:

12.1.1 *Repeatability*—The difference between successive test results, obtained by the same operator with the same apparatus under constant operating conditions of 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:

0.6 volume % (1)

12.1.2 *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:

1.4 volume % (2)

12.2 *Bias*—The procedure in this test method has no bias because the volume percent of gasoline diluent can only be defined in terms of this test method.

13. Keywords

13.1 distillation; engine oils; gasoline distillation; used

ANNEX**(Mandatory Information)****A1. APPARATUS**

A1.1 *Flask*, round-bottomed, short-necked, having a nominal capacity of 1 L. **Fig. 1** and **Fig. 2** show recommended designs and glass connections.

A1.2 *Condenser*, Liebig straight type with a jacket not less than 400 mm long, and with an inner tube having an outside diameter of 10 mm to 13 mm. **Fig. 1** and **Fig. 2** show characteristic details of suitable condensers.

A1.3 *Trap*, in accordance with the details of construction shown in **Fig. 1** and **Fig. 2**, and conforming to the following requirements: It shall be graduated from 0 mL to 5 mL in

0.1 mL divisions. It shall be calibrated at four or more points by first filling it with water and then adding Stoddard solvent or *n*-heptane from a standard buret having a calibrated capacity at least equal to that of the trap. The Stoddard solvent should conform to Specification **D235**. The *n*-heptane should conform to the current IP specification (see **2.1**). The error of the indicated volume shall not exceed 0.05 mL.

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