



Designation: D 322 – 97 (Reapproved 2002)<sup>ε1</sup>

An American National Standard



Designation: 23/2000

## Standard Test Method for Gasoline Diluent in Used Gasoline Engine Oils by Distillation<sup>1</sup>

This standard is issued under the fixed designation D 322; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

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

<sup>ε1</sup> NOTE—Warning notes were editorially moved into the standard text in March 2003.

### 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 D 3525 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.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

- D 235 Specification for Mineral Spirits (Petroleum Spirits) Hydrocarbon Dry Cleaning Solvents<sup>2</sup>
- D 3525 Test Method for Gasoline Diluent in Used Gasoline Engine Oils by Gas Chromatography<sup>3</sup>
- D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products<sup>3</sup>

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.06 on Analysis of Lubricants.

This test method was adopted as a joint ASTM-IP standard in 1964. In the IP, this method is under the jurisdiction of the Standardization Committee.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 06.04.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 05.02.

D 4175 Terminology Relating to Petroleum, Petroleum Products, and Lubricants<sup>3</sup>

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products<sup>3</sup>

2.2 *IP Methods for Analyses and Testing, Vol II, Part I*<sup>4</sup>

### 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.

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.

<sup>4</sup> Available from the Institute of Petroleum, 61 New Cavendish St., London, W1G 7AR.

### 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.

### 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 D 235.

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 D 4057 or D 4177.

### 9. Procedure

9.1 Mix the sample thoroughly, measure  $25 \pm 5$  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  $1 \pm 0.1$  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 to 10 min after heat is applied, with the water and sample being at 21 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, 15, and 30 min,  $\pm 1$  min, and each 15 min,  $\pm 1$  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.

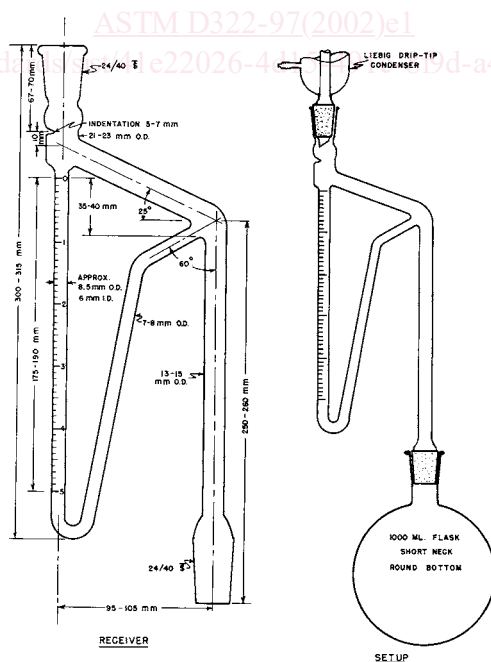


FIG. 1 Apparatus for Determining Diluent in Gasoline Engine Crankcase Oil