Designation: D 3607 – 97 (Reapproved 2002)$^{\epsilon 1}$

An American National Standard

Standard Test Method for Removing Volatile Contaminants from Used Engine Oils by Stripping[1]

This standard is issued under the fixed designation D 3607; 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.

ϵ^1 NOTE—Warning notes were editorially moved into the standard text in March 2003.

1. Scope

1.1 This test method covers a standardized procedure for removing volatile materials such as gasoline and water from used engine oils prior to further oil analysis.

1.2 It also provides an estimate of such volatiles in used engine oils.

1.3 When an accurate value of the gasoline contaminant is required either Test Methods D 322 or D 3525 shall be used.

NOTE 1—Test Method D 322 determines the amount of gasoline by distillation with water. Test Method D 3525 determines the amount of material boiling below the boiling point of n–tetradecane by gas chromatography.

NOTE 2—When the amount of gasoline is required to be known, the user of this test method is advised to determine which method is to be used. There are cases where D 3525 may be set as the referee method.

1.4 The values stated in SI units are to be regarded as the standard. The values in parentheses are for information only.

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 consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific precautionary statement, see 6.1 and 8.5.

2. Referenced Documents

2.1 *ASTM Standards:*

D 322 Test Method for Gasoline Diluent in Used Gasoline Engine Oils by Distillation[2]

D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (the Calculation of Dynamic Viscosity)[2]

D 3525 Test Method for Gasoline Diluent in Used Gasoline Engine Oils by Gas Chromatography[3]

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products[3]

D 4175 Terminology Relating to Petroleum, Petroleum Products, and Lubricants[3]

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products[3]

E 1 Specification for ASTM Thermometers[4]

3. Terminology

3.1 *Definitions:*

3.1.1 *stripping, n*—the process whereby volatile fractions are removed from a liquid material.

3.1.1.1 *Discussion*—In this test method, lighter components such as water and gasoline are removed by the application of heat while passing an inert gas through the liquid.

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*—This test method refers specifically to used lubricating oils from gasoline engines.

4. Summary of Test Method

4.1 A known weight of sample is heated to 90°C under a nitrogen flow for 4.5 h, cooled, and reweighed.

5. Significance and Use

5.1 The removal of volatile materials such as gasoline dilution from used engine oils is especially important if the mechanical shear stability or oxidative stability of the oil is being monitored by measuring a kinematic viscosity change in the oil after it has been used in a gasoline engine.

6. Apparatus

6.1 *Nitrogen Cylinder*, equipped with regulator. (**Warning—**Using pressurized gas is hazardous.)

---

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.06 on Analysis of Lubricants.

Current edition approved Dec. 10, 2002. Published March 2003. Originally approved in 1977. Last previous edition approved in 1997 as D 3607–97.

[2] *Annual Book of ASTM Standards*, Vol 05.01.

[3] *Annual Book of ASTM Standards*, Vol 05.02.

[4] *Annual Book of ASTM Standards*, Vol 14.03.

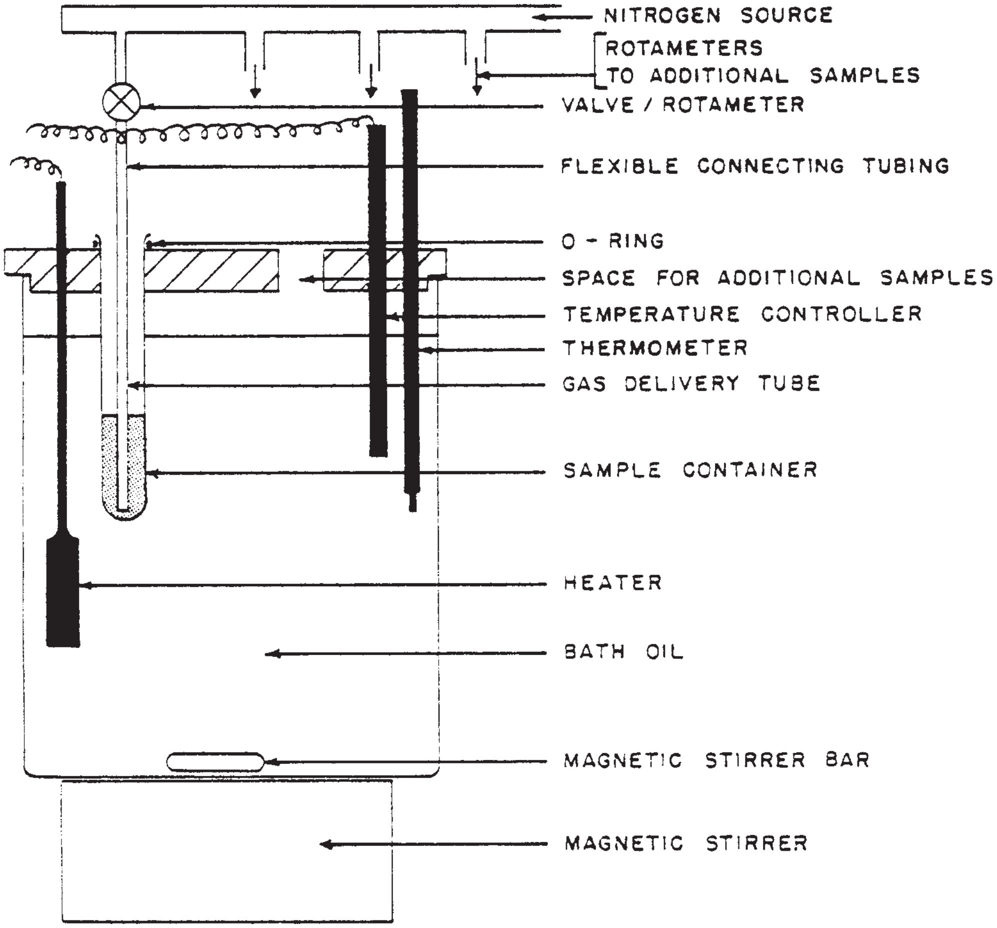

FIG. 1 Schematic of Test Apparatus

6.2 *Rotameters or Nitrogen Flowmeters* [5], one or more, each capable of supplying 400 cm^3/min of nitrogen at atmospheric pressure.

NOTE 3—If several rotameters are available, it is preferable to control the nitrogen flow with an individual rotameter to each tube. Consistent flow rates can be maintained to several tubes by one rotameter if the nitrogen pressure is controlled in a common manifold to all tubes.

6.3 *Connecting Tubing*, of 4.8-mm (3⁄16 in.) inside diameter and 7.9-mm (5⁄16 in.) outside diameter is a convenient size.

NOTE 4—No tolerances for the dimensions of the I.D. and O.D. of the tubing are given because these are nominal sizes.

6.4 *Sample Tubes or Test Tubes*, glass, 25 by 150-mm. As many as eight tubes can usually be handled in a typical apparatus assembly (Note 3).

6.5 *Tubing*, glass, 5-mm outside diameter, 3-mm or smaller inside diameter and about 200 mm long, for nitrogen flow into the sample in the above tubes. This tubing should be long enough to rest just off the bottom of the sample tube when connected to the nitrogen flow assembly with the flexible connecting tubing.

6.6 *Oil Bath*, with suitable cover for inserting glass sample tubes. Rubber O-rings or rubber stoppers between the tops of the tubes and the cover are convenient for steadying and positioning the tubes.

6.7 *Stirring Device*, for oil bath. When stripping several samples at once, the use of a magnetic stirrer and stirring bar is convenient as their use allows more room for the sample tubes.

6.8 *Thermoregulator and Heater*, capable of maintaining the oil bath at 90.0 ± 0.2°C.

6.9 *Thermometer*, such as ASTM Medium Aniline Point Thermometer having a range from 25 to 105°C and conforming to the requirements for Thermometer 34C as prescribed in Specification E 1.

NOTE 5—A typical schematic arrangement of the apparatus is shown in Fig. 1.

7. Sampling

7.1 Using Practices D 4057 (Manual) or D 4177 (Automatic) obtain a representative sample of the material to be tested.

---

[5] The sole source of supply of the apparatus known to the committee at this time is Brooks Instrument Co., Inc., Hatfield, PA 19440, as Sho-Rate 50, Model 1350. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee [1], which you may attend.

7.2 The sample should be well mixed by physical agitation for at least 1 min immediately before selecting the aliquot for testing.

8. Procedure

8.1 Turn on the heater and thermoregulator of the oil bath and set the bath at 90.0 ± 0.2°C (Note 1). If a magnetic stirrer is being used, ensure that it is properly positioned beneath the oil bath.

8.2 Weigh the empty 25 by 150-mm glass tube together with the 5-mm glass tubing to be used with it for nitrogen inlet flow. Weigh the assembly to the nearest 0.1 mg.

8.3 Add 25 mL of well-mixed sample to the assembly of 8.2 and reweigh to the nearest 0.1 mg.

8.4 Place the tubes containing oil into the preheated oil bath. The tubes should be positioned so that the entire oil sample is immersed in the bath and between 20 and 40 mm of the tube extends above the bath oil level.

8.5 Before connecting the gas inlet tubing, turn on the nitrogen flow and set it at 400 cm^3/min. This flow rate should be maintained to each tube. (**Warning—**Using pressurized gas is hazardous.)

8.6 Attach the nitrogen delivery flexible tubing to the 5-mm gas inlet tubing in the sample tube and ensure that the inlet tubing is resting just off the bottom of the sample tube.

NOTE 6—If excessive frothing occurs, decrease the nitrogen flow rate to approximately 50 cm^3/min and maintain this lesser rate until frothing subsides before increasing it to 400 cm^3/min. Frothing will usually subside within 30 min.

8.7 Maintain the 400-cm^3/min nitrogen flow rate after frothing subsides for a total sparging time of 4.5 h.

8.8 Turn off the heater, thermoregulator, stirrer, and nitrogen flow, remove the flexible tubing from the glass inlet tubes, and remove the sample tubes (still containing the glass inlet tubes) from the oil bath.

8.9 Wipe the outside of the sample tube completely dry, allow the assembly to cool to room temperature, and reweigh to the nearest 0.1 mg. The oil sample in the tube is now free of diluent.

NOTE 7—If the kinematic viscosity of the stripped sample is desired, the sample should be reheated to about 60°C and filtered while still warm before undertaking the kinematic viscosity measurement. This is especially important if the used oil contains solid particles. See Test Method D 445 for determining the kinematic viscosity and filtering of the oil.

9. Calculation

9.1 Calculate the volatile contaminants content of the sample as weight percent volatile contaminants as follows:

$$\text{Volatile contaminants, mass \%} = [(A - B)/A] \times 100 \quad (1)$$

where:

A = weight of original sample, g, and

B = weight of sample after stripping, g.

10. Report

10.1 Report the result as volatile contaminants content, mass percent ASTM D 3607.

10.2 Report the time, if any, that a nitrogen flow rate of 50 cm^3/min was used due to excessive frothing.

11. Precision and Bias

11.1 The following criteria should be used for judging the acceptability of results (95 % confidence) (Note 8 and Note 9):

11.1.1 *Repeatability*—The difference 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 following value only in one case in twenty: 0.27 mass %.

11.1.2 *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 out of twenty: 1.40 mass %.

NOTE 8—The precision reported here was obtained by a round-robin program using a stripping temperature of 87.8°C and a stripping time of 4.5 h. Kinematic viscosity results on the stripped samples were determined at 98.9°C.

NOTE 9—The precision of kinematic viscosity determinations on oils after stripping by this test method will not usually be as good as the precision stated in Test Method D 445. Results of duplicate stripping operations on two portions of the same oil by the same operator in the same laboratory should be considered suspect if the difference in the measured kinematic viscosities after stripping is greater than 1.4 % of their mean. Results on the same oil after stripping in each of two laboratories should be considered suspect if the difference in the measured kinematic viscosities after stripping is greater than 2.8 % of their mean. The precision of kinematic viscosity measurements on gelled used engine oils may be much poorer than this. Gelled oils are defined as oils that develop structure on standing, but that become much more fluid with light agitation.

11.2 *Bias*—The procedure in this test method for measuring volatile contaminants in used engine oils has no bias because the mass percent of volatile materials can only be defined in terms of the test method.

12. Keywords

12.1 contaminants; engine oils; gasoline dilution; used engine oils; water contamination

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).