Designation: ~~D6794 – 13~~ D6794 – 14

Standard Test Method for
Measuring the Effect on Filterability of Engine Oils After Treatment with Various Amounts of Water and a Long (6 h) Heating Time[1]

This standard is issued under the fixed designation D6794; 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.

INTRODUCTION

Any properly equipped laboratory, without outside assistance, can use the procedure described in this test method. However, the ASTM Test Monitoring Center (TMC)[2] provides reference oils and an assessment of the test results obtained on those oils by the laboratory (see Annex A1). By these means, the laboratory will know whether their use of the test method gives results statistically similar to those obtained by other laboratories. Furthermore, various agencies require that a laboratory utilize the TMC services in seeking qualification of oils against specifications. For example, the U.S. Army imposes such a requirement in connection with several Army engine lubricating oil specifications.

Accordingly, this test method is written for use by laboratories that utilize the TMC services. Laboratories that choose not to use those services may simply ignore those portions of the test method that refer to the TMC.

This test method may be modified by means of information letters issued by the TMC. In addition, the TMC may issue supplementary memoranda related to the test method (see Annex A1).

For other information, refer to the research report of this test method.[3]

1. Scope*

1.1 This test method covers the determination of the tendency of an oil to form a precipitate that can plug an oil filter. It simulates a problem that may be encountered in a new engine run for a short period of time, followed by a long period of storage with some water in the oil.

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

2. Referenced Documents

2.1 *ASTM Standards:*[4]

D1193 Specification for Reagent Water

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4485 Specification for Performance of Active API Service Category Engine Oils

D5844 Test Method for Evaluation of Automotive Engine Oils for Inhibition of Rusting (Sequence IID) (Withdrawn 2003)[5]

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum ~~Products~~ Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.B0 on Automotive Lubricants.

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[2] ASTM Test Monitoring Center, 6555 Penn Ave., Pittsburgh, PA 152006-4489. This test method is supplemented by Information Letters and Memoranda issued by the ASTM Test Monitoring Center. This edition includes all information letters through No. 13–1. Users of this test method can contact the ASTM Test Monitoring Center to obtain the most recent of these.

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1492.

[4] 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.

[5] The last approved version of this historical standard is referenced on www.astm.org.

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

D5862 Test Method for Evaluation of Engine Oils in Two-Stroke Cycle Turbo-Supercharged 6V92TA Diesel Engine (Withdrawn 2009)[5]

3. Terminology

3.1 *Definitions:*

3.1.1 *calibrate, v*—to determine the indication or output of a device (e.g., thermometer, manometer, engine) with respect to that of a standard.

3.1.2 *calibration test, n*—a test, using a coded oil, conducted as specified in the test method.

3.1.2.1 *Discussion*—

The test result is used to determine the suitability of the testing facility/laboratory to conduct such tests on non-reference oils.

3.1.3 *candidate oil, n*—an oil that is intended to have the performance characteristics necessary to satisfy a specification and is tested against that specification. **D5844**

3.1.4 *engine oil, n*—a liquid that reduces friction or wear, or both, between the moving parts within an engine; removes heat, particularly from the underside of pistons; and serves as a combustion gas sealant for the piston rings.

3.1.4.1 *Discussion*—

It may contain additives to enhance certain properties. Inhibition of engine rusting, deposit formation, valve train wear, oil oxidation, and foaming are examples. **D5862**

3.1.5 *non-reference oil, n*—any oil other than a reference oil–such as a research formulation, commercial oil, or candidate oil. **D5844**

3.1.6 *reference oil, n*—an oil of known performance characteristics, used as a basis for comparison.

3.1.6.1 *Discussion*—

Reference oils are used to calibrate testing facilities, to compare the performance of other oils, or to evaluate other materials (such as seals) that interact with oils. **D5844**

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *effective filter area, n*—that surface of a test filter that can receive the material to be filtered.

3.2.2 *new oil, n*—an unused oil having the identical formulation and base stock as the test oil.

3.2.3 *test oil, n*—the new oil with various amounts of water added.

3.2.3.1 *Discussion*—

A potential precipitate in the test oil is induced by heating the oil and aging.

4. Summary of Test Method

4.1 The test oil is treated with deionized water. The sample is heated to 70 °C for 6 h, followed by storage at room temperature. The sample is filtered and the flow rate is calculated determining the engine oil filterability characteristics.

5. Significance and Use

5.1 It is normal for some of the combustion products of an internal combustion engine to penetrate into the engine lubricant and be retained in it.

5.2 When an engine is run for a period of time and then stored over a long period of time, the by-products of combustion might be retained in the oil in a liquefied state.

5.3 Under these circumstances, precipitates can form that impair the filterability of the oil the next time the engine is run.

5.4 This test method subjects the test oil and the new oil to the same treatments such that the loss of filterability can be determined.

5.5 Reference oils, on which the data obtained by this test method is known, are available.

5.6 This test method requires that a reference oil also be tested and results reported. Two oils are available, one known to give a low and one known to give a high data value for this test method.

NOTE 1—When the new oil test results are to be offered as candidate oil test results for a specification, such as Specification D4485, the specification will state maximum allowable loss of filterability (flow reduction) of the test oil as compared to the new oil.

6. Apparatus

6.1 The apparatus consists of a 25 mL burette, a filter holder with 25 µm automotive oil filter paper, and a source of (69 ± 2) kPa air pressure. Discs of filter paper are cut to fit the holder and installed (see Fig. 1).

6.1.1 *Burette (glass or plastic),* 25 mL, with polytetrafluoroethylene (PTFE) stopcock and (1.8 ± 0.1) mm burette tip opening.

6.1.2 *Air Regulator,* capable of regulating air to a pressure of (69 ± 2) kPa.

6.1.3 *Filter Holder,* with effective filter area approximately 0.8 cm^2.

6.1.4 *Automotive Oil Filter Paper,* 25 µm porosity.[6]

6.2 *Blender,* capable of 18 000 rpm ± 10 % without the container.

6.2.1 *Timer,* capable of timing (30 ± 1) s.

6.3 *Container with Blade,* 250 mL, compatible with the blender.

6.4 *Syringe,* 1000 µL.

6.5 *Glass Jars,* 60 mL, with inert lined lids.

6.6 *Mechanical Convection Oven,* capable of maintaining (70 ± 1) °C.

6.7 *Sensors (or equivalent timing devices),* capable of measuring sequential events to 1 s resolution.

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[6] The sole source of supply of the automotive oil filter paper known to the committee at this time is The Central Parts Distributor, OH Technologies Inc., P.O. Box 5039, Mentor, OH 44061-5039. 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.
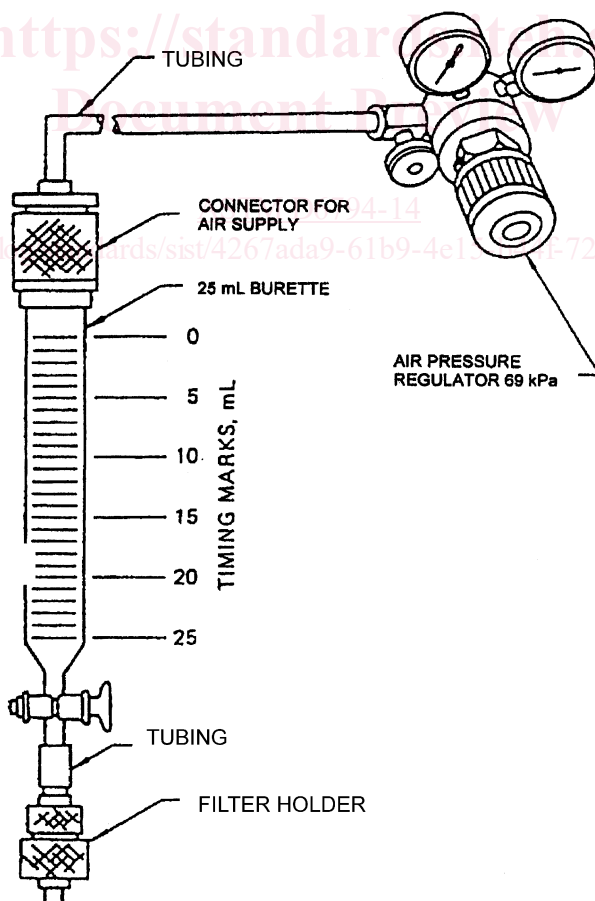
NOTE 1—Filter holder has approximate area of 0.8 cm^2. It contains automotive oil filter paper (25 µm porosity).
FIG. 1 Apparatus To Measure Engine Oil Filterability

7. Reagents

7.1 *Purity of Reagents*—Use reagent grade chemicals in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society,[7] where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Test Method D1193 Type III deionized water or water of equivalent purity.

7.3 *TMC Reference Oils*—These are available from the Test Monitoring Center.

8. Sampling

8.1 Take samples in accordance with the instructions in Practice D4057.

9. Preparation of Test Oil Sample and Filter

9.1 Mix (49.7 ± 0.1) g of test oil, (0.3 ± 0.05) g [(0.3 ± 0.05) mL with the 1000 µL syringe] of deionized water in the blender for (30 ± 1) s at 18 000 rpm ±10 %. Cover the top of the container loosely to prevent oil spattering.

9.2 Transfer the sample to a 60 mL wide mouth glass bottle and place the loosely capped (1/4 turn) bottle in an oven at (70.0 ± 1.0) °C for 6 h. Remove from the oven, tighten cap, and allow to cool to room temperature (20 to 24) °C.

9.3 Repeat 9.1 and 9.2 with ~~0.5 g water, 1.0 g water, and 1.5 g~~ (49.5 ± 0.1) g of test oil and (0.5 ± 0.05) g water, (49.0 ± 0.1) g of test oil and (1.0 ± 0.05) g water, and (48.5 ± 0.1) g of test oil and (1.5 ± 0.05) g water.

9.4 Store in dark at room temperature (20 to 24) °C.

9.5 Determine filterability (48 ± 2) h after removing the sample from the oven.

9.6 Dry filters in an oven at (70 ± 2) °C for (30 ± 2) min and store in a desiccator until used.

10. Procedure

10.1 Assemble apparatus as shown in Fig. 1 with filter installed in proper orientation (25 µm smooth side up).

10.2 Determine the new oil flow rate by placing a sample of the new oil in the burette. Pressurize the system and force at least 10 mL of oil through the filter to saturate the filter with oil and remove any air bubbles. Disconnect the air line and fill the burette with new oil to a level (1 to 2) cm above the *0* mark. Pressurize the system to (69 ± 2) kPa, open the stopcock, and measure the flow time for each successive 5 mL of oil between the (0 and 25) mL graduations.

10.3 To determine the test oil flow rate, the flow times of the new oil are first determined. Using the same filter disc, filter holder, and burette, reduce the new oil level in the burette to the lowest level that allows no air bubbles below the stopcock. Disconnect the air line and fill the burette with a well-mixed sample of test oil to a level (1 to 2) cm above the *0* mark. Pressurize the system to (69 ± 2) kPa, open the stopcock, and measure the flow time for each successive 5 mL of oil between the (0 and 25) mL graduations.

10.4 Run each non-reference and reference oil in duplicate; repeat Section 9 and 10.1 to 10.4 for each non-reference and each reference oil.

10.5 For TMC-monitored tests, run the TMC reference oil on the same day as the non-reference oil.

10.6 For tests not monitored by the TMC, an in-house quality assurance oil can be used in place of the TMC reference.

11. Calculation

11.1 Calculate the flow rate for the new oil and the test oil for each 5 mL portion of oil using Eq 1:

$$\text{flow rate} = \frac{A}{B} \qquad (1)$$

where:
A = volume of oil, and
B = flow time.

11.2 Calculate the percent change in flow rate of the test oil relative to the new oil with the final oil flow rates (between 20 mL and 25 mL measured with the same filter disc) using Eq 2:

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[7] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.