Designation: D6795 – 08

Standard Test Method for Measuring the Effect on Filterability of Engine Oils After Treatment with Water and Dry Ice and a Short (30-min) Heating Time[1]

This standard is issued under the fixed designation D6795; 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 Engine Oils

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

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

E344 Terminology Relating to Thermometry and Hydrometry

3. Terminology

3.1 *Definitions:*

3.1.1 *calibrate*, *v*—to determine the indication or output of a measuring device with respect to that of a standard. **E344**

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

[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.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. 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:D02-1529.

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] Withdrawn.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

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 water added and dry ice 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 and dry ice. The sample is heated to 70°C for 30 min, 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 may 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 mm, (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*, 250 mL, with blade compatible with the blender.

6.4 *Syringe*, 1000 µL.

6.5 *Dry Ice (solid carbon dioxide).*

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.

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

7. Reagents

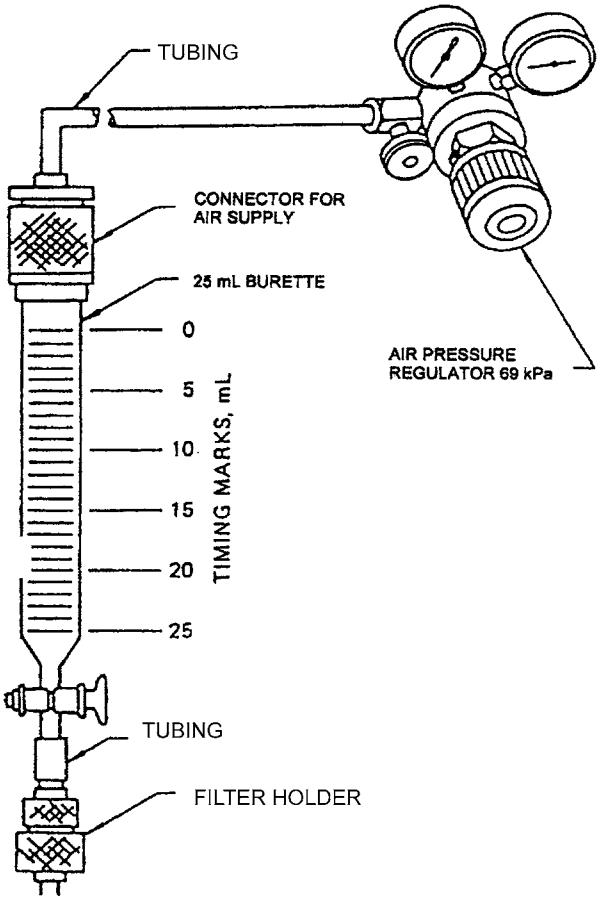
7.1 *Purity of Reagents*—Use reagent grade chemicals in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society,[7] where such specifications are available. Other grades may be 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 Specification D1193 Type III deionized water or water of equivalent purity.

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

[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.

[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.



NOTE—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

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 and approximately 10 g of dry ice in the blender for 30 ± 1 s at 18 000 rpm ±10 %. Cover the top of the container loosely to prevent oil spattering while allowing rapid vaporization of the dry ice.

9.2 Transfer the sample to a 60-mL wide-mouth glass bottle and place the loosely capped (¼ turn) bottle in an oven at 70.0 ± 1.0°C for 30 ± 2 min. Remove from the oven, tighten cap and allow to cool to room temperature (20 to 24°C).

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

9.4 Determine filterability 48 ± 2 h after removing the sample from the oven.

9.5 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 in accordance with 10.2. 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:

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

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 and 25 mL measured with the same filter disc) using Eq 2:

$$\text{percent change in flow rate} = \frac{E - D}{D} \times 100 \quad (2)$$

where:
D = final new oil flow rate, and
E = final test oil flow rate.

12. TMC Reference Oil Testing

12.1 Test a TMC-coded reference oil along with each batch of non-reference oil tests. Run the reference oil simultaneously with, and in the same batch as, the non-reference oils.

NOTE 2—Annex A1 discusses the involvement of the ASTM TMC with respect to the reference test-monitoring program.

12.1.1 Prior to conducting a reference oil test, procure a supply of reference oils directly from the TMC. These oils have been formulated or selected to represent specific chemistry types, or performance levels, or both. Each reference oil sample is identified using a unique set of identification codes on the container labels. The coded reference samples provide for a blind reference-testing program to protect against the possibility of bias in the results.

12.1.1.1 The testing laboratory tacitly agrees to use the TMC reference oils exclusively in accordance with the TMC's published Policies for Use and Analysis of ASTM Reference Oils, and to run and report the reference oil test according to TMC guidelines.

NOTE 3—Policies for the Use and Analysis of ASTM Reference Oils are available from the TMC.

12.1.2 Request a reference oil assignment from the TMC for this test method. The TMC will determine the specific reference oil to be tested by the laboratory. Assignments will be made by the unique identifying codes on the reference oil container labels.

12.1.3 Run the TMC reference oil test according to the test method and in the same manner as the non-reference oil test(s). Run the reference oil simultaneously with, and in the same batch as, the non-reference oils. Reference oils and non-reference oils in the same batch shall be run within the same 4-h time frame.

12.1.4 *Reporting of Reference Oil Test Results*—Report the results of all reference oil tests to the TMC according to the following directives:

12.1.4.1 The data report forms are available from the TMC for reporting all TMC reference oil test data to the TMC. Report only the reference oil results to the TMC. Do not include any non-reference test data. Complete all of the required blank fields on the forms.

12.1.4.2 Transmit reference test data to the TMC by electronic means or by telephone facsimile immediately upon completion of the test analysis. Include all of the reporting forms in the transmission.

NOTE 4—Specific protocols for the electronic transmission of test data to the TMC are available from the TMC.

12.1.5 *Evaluation of Reference Test Oil Results*—Upon receipt of the transmitted TMC reference oil test results, the TMC will review the test for operational adherence to the published test method. If the test is found to be operationally valid, the reference oil results will be evaluated using acceptance criteria established by the governing surveillance panel. The reference oil acceptance criteria are subject to change at the discretion of the surveillance panel.

12.1.5.1 If the transmitted test is found to be both operationally valid and statistically acceptable, the testing laboratory will be notified of the acceptable status of the reference test. The uncoded TMC reference oil identification will also be disclosed to the testing laboratory.

12.1.5.2 In the event that a TMC reference oil test is found to be unacceptable, an explanation of the problem relating to the failure will be provided to the testing laboratory. If there is an obvious operational reason for the failed test, the problem shall be corrected before requesting another TMC reference oil assignment. If the reason for failure is not obvious, all test-related equipment shall be rechecked for compliance to the test method and good laboratory practice. Following this recheck the TMC will assign another TMC reference oil for testing.

12.1.6 *Status of Non-Reference Oil Tests Relative to TMC Reference Oil Tests*—The batch of non-reference tests is considered valid only if the results of the TMC reference oil test meet the predetermined acceptance specifications for the particular reference oil tested.

13. Report

13.1 Report the following information:
13.1.1 Non-reference or reference oil identification.
13.1.2 Porosity of filter used (25 μm).
13.1.3 Percent change in final flow rate for each test.
13.1.4 Average percent change in final flow rate for the duplicate tests.
13.1.5 For TMC-monitored tests, contact the TMC for reporting requirements.
13.1.6 For tests not monitored by the TMC, an in-house quality assurance sample can be used to evaluate test acceptability.

14. Precision and Bias

14.1 The following criteria should be used for judging the acceptability of 25-μm filter results:

14.1.1 *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:

Repeatability = 11.4 %

14.1.2 *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:

Reproducibility = 23.6 %

14.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for this test method, bias cannot be determined.

15. Keywords

15.1 engine lubricating oil; filterability; storage

ANNEX

(Mandatory Information)

A1. THE ROLE OF THE ASTM TEST MONITORING CENTER AND THE CALIBRATION PROGRAM

A1.1 *Nature and Functions of the TMC*—The TMC is a non-profit organization located in Pittsburgh, PA, USA. The TMC is staffed to administer technical studies; conduct laboratory visits; perform statistical analyses of reference oil test data; blend, store, and ship reference oils; and provide associated administrative functions to maintain the referencing calibration program for various lubricant tests as directed by ASTM Subcommittee D02.B0 and the Test Monitoring Board. The TMC coordinates its activities with the test sponsors, the test developers, the surveillance panels, and the testing laboratories through a consensus process.

A1.2 *Rules of Operation of the TMC*—The TMC operates in accordance with the ASTM Charter, the ASTM Bylaws, the Regulations Governing ASTM Technical Committees, the Bylaws Governing ASTM Committee D02, and the Rules and Regulations Governing the ASTM Test Monitoring System.

A1.3 *Management of the TMC*—The management of the Test Monitoring System is vested in the Test Monitoring Board (TMB) elected by ASTM Subcommittee D02.B0. The TMB selects the TMC Administrator who is responsible for directing the activities of the TMC staff.

A1.4 *Operating Income of the TMC*—The TMC's operating income is obtained from fees levied on the reference oils supplied, and on the calibration tests conducted. Fee schedules are established and reviewed by ASTM Subcommittee D02.B0.

A1.5 *Conducting a Reference Oil Test*—For those laboratories choosing to utilize the services of the TMC in maintaining the calibration of test methods and apparatus, calibration testing is conducted at regular intervals as determined by ASTM D02.B0.07 Engine Oil Filterability Surveillance Panel. These tests are conducted using coded reference oils supplied by the TMC as outlined in 11.1 of this test method. It is the laboratories' responsibility to maintain the calibration in accordance with this test method. It is also the laboratories' responsibility to keep an on-site reference oil inventory at or above the minimum level specified by the TMC test representative.

A1.6 *New Laboratories:*

A1.6.1 Laboratories wishing to participate in the ASTM Monitoring System will be requested to conduct reference oil tests to ensure that the laboratory is using the proper testing techniques. Information concerning fees, laboratory inspections, reagents, testing practices, appropriate committee membership, and rater training can be obtained by contacting the TMC Administrator.

A1.6.2 The calibrating reference oils produce various filterability characteristics. When new reference oils are selected, member laboratories will be requested to run their share of the tests needed to enable the TMC to recommend proper industry performance and precision targets and performance acceptance limits. These donated tests will be run as required by ASTM D02.B0.07 Engine Oil Filterability Surveillance Panel to establish these targets and acceptance limits for new oils.

A1.7 *TMC Information Letters:*

A1.7.1 Occasionally it may become necessary to change the test method, and notify the test laboratories of the change, prior to consideration of the change by either ASTM Subcommittee D02.B0 on Automotive Lubricants, or ASTM Committee D02 on Petroleum Products and Lubricants. In such a case, the TMC will issue an Information Letter. Subsequently, prior to each semiannual ASTM Committee D02 meeting, the accumulated Information Letters are balloted by ASTM Subcommittee D02.B0. Following this action, the approved Information Letters are used to revise the affected standards, and these are balloted in the main committee, and finally, the Society. By this means, the Society's due process procedures are applied to these Information Letters.

A1.7.2 The review of an Information Letter prior to its original issue will differ according to its nature. In the case of an Information Letter concerning a part number change, which does not affect test results, the TMC is authorized to issue such a letter. Long-term studies by the Surveillance Panel to improve test procedures through improved operation and hardware control may result in a recommendation to issue an Information Letter. If obvious procedural items affecting test

results need immediate attention, the test sponsor and the TMC will issue an Information Letter and present the background and data to the Surveillance Panel for approval prior to the semiannual ASTM Subcommittee D02.B0 meeting.

A1.7.3 The ASTM Committee on Technical Committee Operations (COTCO) in 1984 gave authority for the issuance of Information Letters, as follows: "COTCO recognizes that D-2 has a unique and complex situation. The use of Information Letters is approved providing each letter contains a disclaimer to the effect that such has not obtained ASTM consensus. These Information Letters should be moved to such consensus as rapidly as possible."

A1.8 *TMC Memoranda*—In addition to the Information Letters discussed under A1.7, supplementary memoranda may be issued by the TMC. These memoranda are developed by the TMC, often under the guidance of the surveillance panel, and to participating laboratories. The memoranda convey such information as approval for test parts or materials, clarification of the test procedure, notes and suggestions of the collection and analysis of special data that the TMC may request, or for any other pertinent matters having no direct affect on test performance, results, and precision and bias.