Designation: ~~D7563 – 10 (Reapproved 2016)~~ D7563 – 23

Standard Test Method for Evaluation of the Ability of Engine Oil to Emulsify Water and Simulated Ed85 Fuel[1]

This standard is issued under the fixed designation D7563; 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.

1. ~~Scope~~ Scope*

1.1 This test method describes a qualitative procedure to measure the ability of a specific volume of engine oil to emulsify a specific added volume of combined water and simulated Ed85 fuel upon agitation in a high-speed blender and to retain this emulsified state for at least 24 h at temperatures of both 20 °C to 25 °C and –5 °C to 0 °C.

1.2 Information Letters are published periodically by the ASTM Test Monitoring Center (TMC) to update this and other test methods under the jurisdiction of Subcommittee D02.B0. Copies of these letters can be obtained by writing the Center.[2]

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

1.4 *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~~safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *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:*[3]

D1193 Specification for Reagent Water
D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants
D4485 Specification for Performance of Active API Service Category Engine Oils
D4806 Specification for Denatured Fuel Ethanol for Blending with Gasolines for Use as Automotive Spark-Ignition Engine Fuel
D5798 Specification for Ethanol Fuel Blends for Flexible-Fuel Automotive Spark-Ignition Engines
D5854 Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products
E1272 Specification for Laboratory Glass Graduated Cylinders

3. Terminology

3.1 *Definitions:*

---

[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.B0.07 on Development and Surveillance of Bench Tests Methods.

Current edition approved ~~April 1, 2016~~June 1, 2023. Published ~~May 2016~~July 2023. Originally approved in 2010. Last previous edition approved in ~~2010~~2016 as ~~D7563 – 10.~~D7563 – 10 (2016). DOI: ~~10.1520/D7563-10R16.~~10.1520/D7563-23.

[2] Information letters may be obtained from the ASTM Test Monitoring Center, ~~6555 Penn Avenue, Pittsburgh, PA 15206-4489. www.standards.astmtmc.emu.edu.~~ 203 Armstrong Drive, Freeport, PA 16229, www.astmtmc.org.

[3] 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.

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

3.1.1 *denatured fuel ethanol, n*—fuel ethanol made unfit for beverage use by the addition of denaturants. **D4806**

3.1.2 *fuel ethanol (Ed75-Ed85), n*—blend of ethanol and hydrocarbon of which the ethanol portion is nominally 75 to 85 volume % denatured fuel ethanol. **D5798**

3.1.3 *engine oil, n*—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 piston rings.

3.1.3.1 *Discussion*—

It may contain additives to enhance certain properties. Inhibition of engine rusting, deposit formation, valve train wear, oil oxidation, and foaming are examples. **D4175**

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *aqueous presence, n*—translucent or semi-transparent or transparent appearance, laciness, bubbling, or layer in the lower region of the glass container.

3.2.2 *simulated Ed85 fuel, n*—laboratory blend made to simulate Ed85 fuel by mixing 85 % denatured fuel ethanol and 15 % unleaded gasoline by volume.

3.2.3 *test oil, n*—any engine oil subjected to evaluation in this test method.

4. Summary of Test Method

4.1 Distilled water, simulated Ed85 fuel, and the test oil are emulsified in a high-speed blender. Portions of the resulting emulsion are stored in two graduated cylinders (or suitably dimensioned alternative containers) at temperatures of 20 °C to 25 °C and –5 °C to 0 °C for 24 h. ~~Presence~~Existence or absence of an aqueous presence layer at the bottom of either of the containers after this time interval is observed and reported.

5. Significance and Use

5.1 During engine operation, engine oil can become contaminated by water and fuel. In the case of Ed85 fuels, this contamination can result in a non-emulsified aqueous bottom layer in the oil that can affect the lubrication and detergency of the engine oil. To avoid field problems, engine oil should be capable of emulsifying water contamination to the extent that no aqueous ~~layer~~presence appears.

5.2 The test described in this method is designed to evaluate the ability of an engine oil, contaminated with a specified amount of water (volume fraction of 10 % of the original oil sample) and simulated Ed85 fuel (also a volume fraction of 10 % of the original oil sample), to emulsify the water after agitation in a blender and to maintain this emulsion at temperatures of 20 °C to 25 °C and –5 °C to 0 °C for at least 24 h.

5.3 This test method has potential use in specifications of engine lubricating oils, such as Specification D4485.

6. Apparatus and Supporting Equipment

6.1 *Blender*—Commercial laboratory blender with its associated glass or stainless steel liquid container having suitable blending capacity for this use and a blade rotating at approximately 10 000 r/min ± 2000 r/min as measured by a stroboscopic tachometer.

NOTE 1—This speed is generally equivalent to the low-speed setting on two-speed commercial mixers or the “2” or “3” setting on seven-speed commercial mixers.

6.2 *Glass, Graduated Cylinders*—Unless otherwise stated, all graduated cylinders shall conform to Specification E1272, Class B, Style I.

6.2.1 *For Measuring Volume of Test Oil and Unleaded Fuel*—Having a capacity of 250 mL with graduation marks of 2 mL.

6.2.2 *For Measuring Volumes of Simulated Ed85 Fuel and Water*—Having a capacity of 25 mL with graduation marks of 0.2 mL.

6.2.3 *For Preparing the Simulated Ed85 Fuel*—Glass-stoppered and conforming to Specification E1272, Class B, Style II and having a capacity of 1 L with graduation marks of 10 mL.

6.3 *Containers for Observation of Emulsified Samples*—Use either of the following types:

6.3.1 Glass-stoppered, graduated cylinders conforming to Specification E1272, Class B, Style II and having a capacity of 100 mL with graduation marks of 1 mL (see Fig. 1).

6.3.2 Glass, flat-bottomed, sample bottles with an outer diameter of approximately 30 mm, a minimum height of 150 mm and capable of being closed by a solvent resistant screw cap (see Fig. 1). The height of the bottles should be such that 100 mL of fluid comes below the shoulder to allow accurate measurement of the height of 100 mL of emulsion (see 9.6 and X1.2.1).

NOTE 2—A simple way of marking the sample bottles is to pour 100 mL of water from a graduated cylinder into each clean and dry bottle, mark the level of the water meniscus on the bottle (suggest using a glass scribing tool or triangular metal file), and dry the bottle before proceeding.

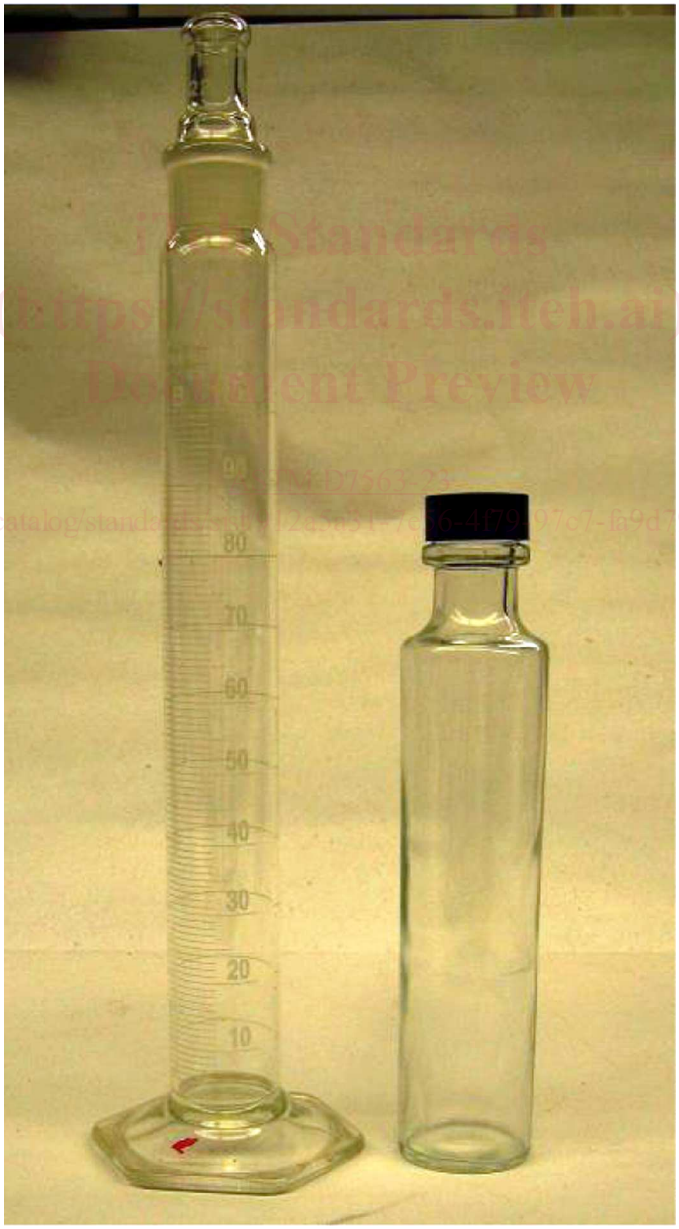

FIG. 1 Observation Containers: Graduated Cylinder with Ground Glass Stopper and Flat-bottom Sample Bottle with Cap

6.4 *Temperature Chambers:*

6.4.1 Cold cabinet or refrigerator capable of controlling temperature within a range of –5 ºC to 0 °C.

6.4.2 Warm cabinet or oven capable of controlling temperature within a range of 20 ºC to 25 °C.

6.5 *Time-Measuring Equipment:*

6.5.1 Stopwatch or other timer capable of measuring 1 min with a minimum precision of ±1 s.

6.5.2 Clock or other timer capable of measuring 24 h with a minimum precision of ±5 min.

6.6 *Laboratory Hood Vented Exteriorly or Other Well-Ventilated Work Space*—Suitable for pouring volatile, flammable fluids such as denatured fuel ethanol and gasoline into graduated cylinders and containers. Ensure there is no ignition source within the hood or nearby when using an open work space, particularly at lower levels where hydrocarbon volatiles tend to congregate.

7. Reagents and Materials

7.1 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water that meets or exceeds that defined as Type IV of Specification D1193.

7.2 *Denatured Fuel Ethanol*—Use product conforming to Specification D4806. Refer to Specification D4806 and Practice D5854 for information on container selection, storage and handling. Product stored for more than three months should be checked to ensure it has not deteriorated or been contaminated before use. (**Warning—**Danger! Extremely flammable. Vapors may cause flash fire.)

7.3 *Unleaded Gasoline*—Use only Haltermann EEE unleaded fuel coded HF003.[4] Fuel stored for more than three months should be checked to ensure it has not deteriorated or been contaminated before use. (**Warning—**Danger! Extremely flammable. Health Hazard.)

7.4 *Cleaning Solvents*—Reagent grade acetone and *iso*octane. (**Warning—**Danger! Extremely flammable. Vapors may cause flash fire.)

8. Hazards

8.1 *Specific Hazards*—Because of fire danger, carefully pour denatured fuel ethanol and gasoline into graduated cylinders and containers in a laboratory hood, preferably from containers of capacity no greater than about 2 L. See also 7.2 and 7.3.

9. Procedure

9.1 Clean blender and other glassware with *iso*octane followed by acetone (see warning in 7.4).

9.1.1 First, rinse total inner surface of all vessels three times with *iso*octane followed by similarly rinsing three times with acetone and dry thoroughly.

9.2 *Blending the Simulated Ed85 Fuel:*

9.2.1 In a laboratory hood, carefully pour denatured fuel ethanol (see warning in 7.2 and Section 8) into a clean, dry, graduated cylinder with capacity of 1 L (see 6.2.3) so that the bottom of the meniscus is ~~exactly at the mark at~~ at approximately 850 mL.

9.2.2 Carefully pour the gasoline (see warning in 7.3 and Section 8) into the same graduated cylinder so that the bottom of the meniscus is exactly at the mark at 1000 mL. Close the graduated cylinder with the glass stopper.

[4] The sole source of supply of this fuel known to the committee at this time is Haltermann Products, 1201 Sheldon Road, P.O Box 429, Channelview, TX 77530-0429 (www.dow.com/haltermann). 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.

9.2.3 Place the graduated cylinder in a refrigerated cabinet held at –5 °C to 0 °C for 10 min. Remove, hold the glass stopper firmly in place, and upend the graduated cylinder ten times to mix the contents.

NOTE 3—When dealing with fuels in a closed container, it is good laboratory practice to chill the container to prevent any build-up of vapor pressure during agitation. Mixing the two fluids by upending is preferable to shaking because, if too vigorous, the latter may cause a build up of vapor pressure. Moreover, shaking two miscible components in an elongated graduated cylinder does not assure thorough mixing.

9.2.3.1 Unless using immediately, store the simulated Ed85 fuel in a suitable container such as a closed metal container or the stoppered, graduated cylinder in a refrigerated cabinet held at –5 °C to 0 °C. For next use, allow the graduated cylinder and contents to return to ambient temperature and upend ten times to ensure contents are mixed. Discard any unused simulated Ed85 fuel after three months.

NOTE 4—Such storage avoids the loss of the more volatile components of the simulated Ed85 fuel which otherwise may adversely affect the test.

9.3 Using a graduated cylinder (see 6.2.1), pour 185 mL ± 2 mL of the test oil into the clean, dry blender.

9.4 Using separate graduated cylinders (see 6.2.2), pour 18.5 mL ± 0.3 mL each of the simulated Ed85 fuel and water into the blender.

9.5 Place the lid on the blender and blend for 60 s ± 1 s at 10 000 r/min ± 2000 r/min.

9.6 When blending is completed, pour the blended emulsion into each of two, clean graduated cylinders (see 6.3.1) or two previously marked bottles (see 6.3.2 and Note 2) to the mark at 100 mL ± 1 mL (or use a graduated cylinder to pour 100 mL ± 1 mL into each of two, clean sample bottles; see 6.3.2). Close the graduated cylinders with their glass stoppers (or appropriately cap the sample bottles). Place one of the two containers in a refrigerated cabinet held at –5 °C to 0 °C for 24 h ± 0.5 h and the other in a warm cabinet held at 20 °C to 25 °C for 24 h ± 0.5 h.

9.7 At the completion of the 24 h ± 0.5 h storage period, remove the containers from the temperature controlling cabinets and within 10 min, observe the ~~presence~~existence or absence of an aqueous presence or layer at the bottom of either or both the containers.

9.7.1 Fig. A1.1 and Fig. A1.2 show examples of different appearances of emulsion and aqueous ~~layers.~~presence.

9.7.1.1 Fig. A1.1 shows a comparison of samples with and without aqueous ~~layers~~prescence at the bottom of the 100 mL graduated cylinders.

9.7.1.2 The material at the bottom of the cylinder in Fig. A1.1~~(d)~~ (photo #4) is considered to be an oil-continuous emulsion, as opposed to an aqueous ~~layer,~~presence, because the layer is not obviously transparent or clear, is of a milky-white/semi-opaque appearance (as opposed to the samples designated as showing an aqueous ~~layer)~~presence) and has no separation or space between the milky-white fluid volume and the volume of fluid material above it.

9.7.1.3 Fig. A1.2 photo #8 shows samples in the sample bottles in which the water is completely emulsified.

9.7.2 An aqueous ~~layer~~presence is considered present if there is ~~a~~any translucent or semi-transparent or transparent ~~lowest~~ appearance, laciness, bubbling, or or layer in the ~~graduated cylinder.~~lower region of the glass container.

9.7.3 If there is no translucent or semi-transparent or transparent ~~lowest layer~~ appearance in the graduated cylinder, no aqueous ~~layer is considered present.~~presence occurs.

9.7.4 If it is desired to quantify the amount of each phase, proceed as directed in Appendix X1.

9.8 Clean blender and other glassware thoroughly as described in 9.1.

10. Report

10.1 *Observable Aqueous ~~Layer~~Presence at the Bottom of the Container:*

10.1.1 If an aqueous ~~layer~~presence was observed at the bottom of the glass container in 9.7.2 for the blended test mixture stored at 20 °C to 25 °C, report this fact as observable aqueous ~~layer~~presence at 20 °C to 25 °C.

10.1.2 If an aqueous ~~layer~~presence was observed at the bottom of the glass container in 9.7.2 for the blended test mixture stored at –5 °C to 0 °C, report this fact as observable aqueous ~~layer~~presence at –5 °C to 0 °C.

10.2 *No Observable Aqueous ~~Layer~~Presence at the Bottom of the Container:*

10.2.1 If no aqueous ~~layer~~presence was observed at the bottom of the glass container in 9.7.3 for the blended test mixture stored at 20 °C to 25 °C, report this fact as no observable aqueous ~~layer~~presence at 20 °C to 25 °C.

10.2.2 If no aqueous ~~layer~~presence was observed at the bottom of the glass container in 9.7.3 for the blended test mixture stored at –5 °C to 0 °C, report this fact as no observable aqueous ~~layer~~presence at –5 °C to 0 °C.

10.3 Reference this ASTM test method when reporting results obtained using the test method.

11. Precision and Bias[5]

11.1 No statistical information is presented about either the precision or bias of Test Method D7563 for measuring the emulsion retention properties of engine oil since the test result is non-quantitative.

11.1.1 However, a round robin involving seven laboratories and six, fully-formulated test oils representing five different additive technologies has shown that the test method detected:

11.1.1.1 One hundred percent of the time, those oils tested that did not form an aqueous ~~layers~~presence at both 25 °C and 0 °C.

11.1.1.2 One hundred percent of the time, those oils tested that formed an aqueous ~~layers~~presence at 25 °C.

11.1.1.3 Ninety-three percent of the time, those oils tested that formed an aqueous ~~layers~~presence at 0 °C.

12. Keywords

12.1 denatured fuel ethanol; engine oil; engine oil emulsion; simulated Ed85 fuel; water emulsion

ANNEX

(Mandatory Information)

A1. PHOTOGRAPHS SHOWING SAMPLES WITH AND WITHOUT AQUEOUS ~~LAYERS~~PRESENCE

A1.1 See Figs. A1.1 and A1.2.

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1681. Contact ASTM Customer Service at service@astm.org.

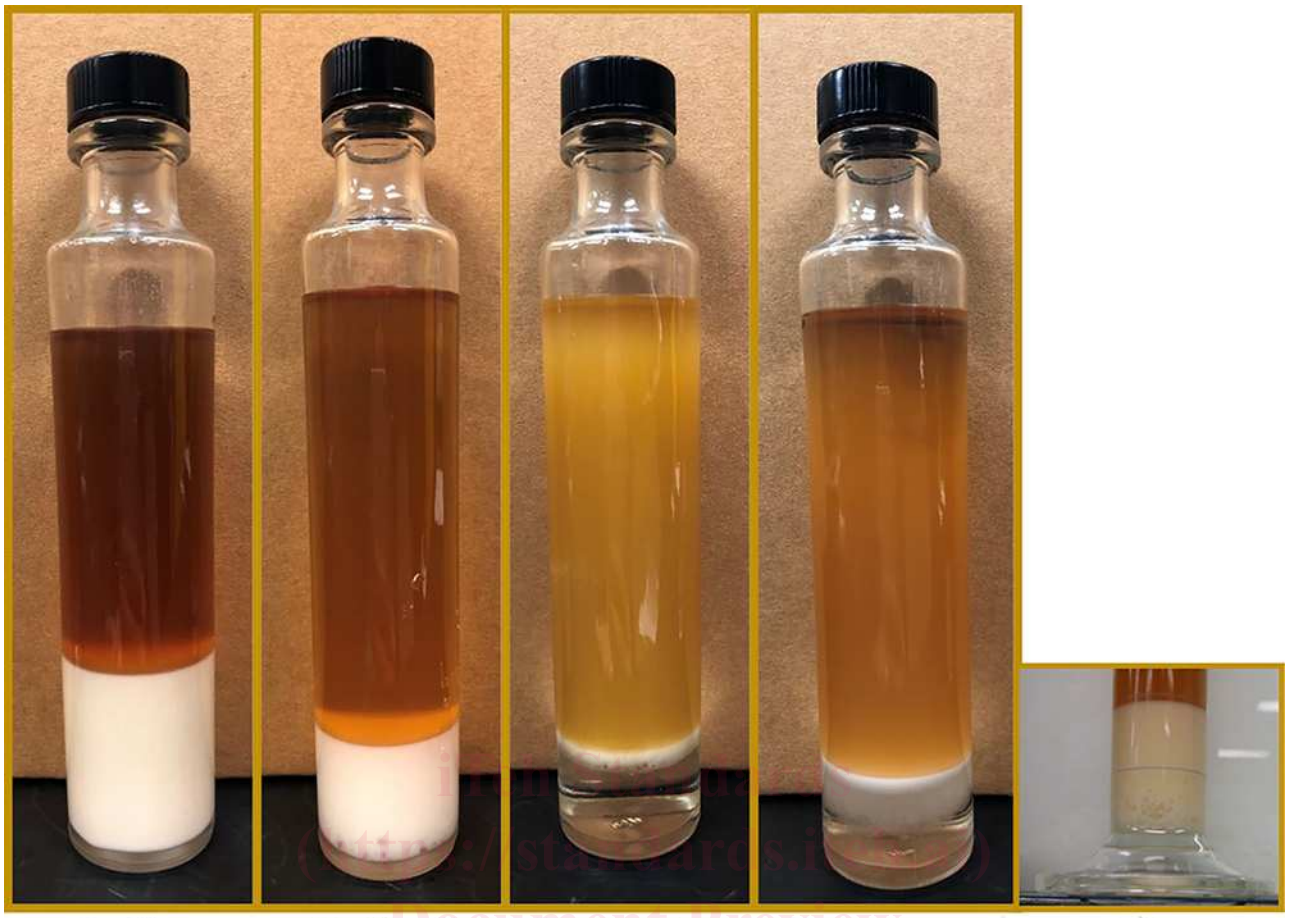

FIG. A1.1 Examples of Aqueous Presence and Absence ~~of Aqueous Layers~~