



Standard Test Method for Water Separation Properties of Light and Middle Distillate, and Compression and Spark Ignition Fuels¹

This standard is issued under the fixed designation D7451; 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*

1.1 This test method covers the evaluation of the tendency of water and fuels with a final boiling point of less than 390°C, as measured in Test Method D86, to separate cleanly rather than create emulsions when they may contain potential emulsion forming additives or components, or have been additized with potential emulsion forming additives, or components.

1.2 This test method applies primarily to gasoline, diesel, kerosine, and distillate grades of gas turbine, marine, home heating oils and furnace fuels (see Specifications D396, D975, D2880, D3699, D4814, and D6985). For fuel components such as biodiesel or alcohol, refer to X1.2 and X1.3.

1.3 This test method is not meant to certify or qualify fuels for sale, but it is intended for use by additive suppliers to determine the need for demulsifier components in their additive packages.

1.4 This test method is not meant for testing of fuels containing large amounts of aqueous soluble components, such as E85, or for testing of water emulsified fuels, or for testing of aviation fuels.

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

1.6 *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:²

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.14 on Stability and Cleanliness of Liquid Fuels.

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² 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.

- D86 Test Method for Distillation of Petroleum Products at Atmospheric Pressure
- D396 Specification for Fuel Oils
- D975 Specification for Diesel Fuel Oils
- D1193 Specification for Reagent Water
- D2274 Test Method for Oxidation Stability of Distillate Fuel Oil (Accelerated Method)
- D2880 Specification for Gas Turbine Fuel Oils
- D3699 Specification for Kerosine
- D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
- D4176 Test Method for Free Water and Particulate Contamination in Distillate Fuels (Visual Inspection Procedures)
- D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
- D4814 Specification for Automotive Spark-Ignition Engine Fuel
- D6985 Specification for Middle Distillate Fuel Oil—Military Marine Applications

2.2 ASTM Adjuncts:

- Distillate Fuel Bar Chart³
- Fuel Clarity Rating Standard⁴

3. Terminology

3.1 Definitions:

3.1.1 *film, n*—thin, translucent layer that does not adhere to the wall of the glass test tube.

3.1.2 *heavy scum, n*—assessment that the fuel/water interface is covered with more than 50% scum that extends into either of the two layers or forms an emulsion (1 mL or greater in volume), or both.

3.1.3 *scum, n*—layer thicker than film (up to 1 mL in volume) or that adheres to the wall of the glass test tube, or both.

3.2 Definitions of Terms Specific to This Standard:

³ Available from ASTM International Headquarters. Order Adjunct No. ADJD417601. Original adjunct produced in 1991.

⁴ Available from ASTM International Headquarters. Order Adjunct No. ADJD7451. Original adjunct produced in 2008.

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

3.2.1 *interface condition rating, n*—qualitative assessment of the tendency of a mixture of water and fuel to form interface films or precipitates.

3.2.2 *water separation rating, n*—qualitative assessment of the tendency of components in the fuel to produce emulsions or precipitates, or both, in separated fuel and water layers.

3.2.3 *water volume change, n*—qualitative indication of the presence of water-soluble components in fuels, or the decrease in water returned during the test due to the formation of scum or emulsions.

3.2.3.1 *Discussion*—For example, the alcohol component in an ethanol-blended gasoline would be extracted into the aqueous phase, thus increasing the volume of the aqueous phase by the approximate volume of the ethanol present in the sample.

4. Summary of Test Method

4.1 The cleanliness of the glass test tube is tested prior to use in the test.

4.2 A sample of the fuel is shaken at room temperature using a standardized technique with an aqueous phase in thoroughly cleaned glassware.

4.3 The change in volume of the aqueous layer, the appearance of the interface and the clarity of the fuel are reported as an indication of the water separation properties of the fuel.

5. Significance and Use

5.1 The primary use of this test method is to evaluate new additive packages in specific fuels to ensure that fuel-water separability will not be compromised by the use of the additive package, either at their normal treat rates or at several times the intended treat rate to evaluate the impact of potential overtreatment.

5.1.1 *Water Volume Changes*—Using this technique reveal the presence of water-soluble components, such as alcohols, in the fuel.

5.1.2 *Interface Condition Ratings*—Using this technique reveal the presence of partially soluble components, such as surfactants, in the fuel.

5.2 Additives or contaminants that affect the interface could harm water separation properties of fuels in equipment and quickly inhibit the free flow of fuel through filters and injection equipment, causing a decrease in combustion performance.

6. Apparatus

6.1 *Graduated Glass Tube*—Sealable, blunt-tipped, 100-mL with ½-mL graduations from 1 to 10 mL and 2-mL graduations from 10 to 100 mL not encompassing the whole diameter of the tube as to interfere with the clarity rating. The dimensions of the tube are as follows:

6.1.1 Overall length = 200 ± 5 mm.

6.1.2 Outer diameter of the tube portion = 38 mm.

6.1.3 Neck length = 25 mm.

6.1.4 Headspace from 100-mL graduation to tube neck = 50 ± 5 mm.

6.1.5 Tip length = 30 mm with a 45° taper.

NOTE 1—Contact Subcommittee D02.14 for a list of possible suppliers for the tubes and caps.

6.2 *Shaking Apparatus (Optional)*—An explosion-proof apparatus capable of shaking the tubes as prescribed in 10.2 is preferred for testing consistency. However, any method that facilitates shaking at the prescribed stroke length, frequency, and sample orientation as outlined in 10.2 is acceptable.

NOTE 2—No suitable vendor for such a machine has been identified. It remains the responsibility of the user to select an apparatus that meets these criteria.

6.3 *Rating Chart*—The line card and fuel clarity rating chart from ASTM Adjuncts [ADJD417601](#) and [ADJD7451](#), respectively. (See 10.4.2.)

NOTE 3—If the line card is not available or fuel clarity is not readily discernible from using the line card, then printed text as described in [Table 1](#) may be used to assess fuel clarity.

TABLE 1 Fuel Clarity Ratings

NOTE—These rating systems have been determined for use in the tubes described in 6.1. The use of narrower or wider tube would give potentially different ratings than those outlined in the tables.

Rating	Appearance
1	<ul style="list-style-type: none"> • Clear and bright, equal to the base fuel
2	<ul style="list-style-type: none"> • Very slight haze • Black 10 pt bold Times New Roman font on a white background readily readable through tube, or • Volume markings and numbers on glassware readily readable through tube
3	<ul style="list-style-type: none"> • Slight haze • Black 10 pt bold Times New Roman font on a white background visible but not readable through tube, or • Volume marking and numbers on glassware visible through tube
4	<ul style="list-style-type: none"> • Slight haze • Black 10 pt bold Times New Roman font on a white background not readable through tube, or • Volume markings and numbers on glassware visible but not readable through tube
5	<ul style="list-style-type: none"> • Hazy/translucent • Black 10 pt bold Times New Roman font on a white background not visible through tube, or • Volume markings and numbers on glassware barely visible through tube
6	<ul style="list-style-type: none"> • Opaque

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used 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, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficient 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 distilled water or water of equivalent or greater purity. Any type of water specified in Specification **D1193** may be used.

7.3 *Glass Cleaning Solution*—Acidic non-chromium containing cleaning solution. (**Warning**—Corrosive. Health hazard. Oxidizing agent.)

NOTE 4—Contact Subcommittee D02.14 for a list of possible suppliers for the glass cleaning solutions.

7.4 *Cleaning Solvent, Reagent Grade*—Trisolvant (TAM) (as defined by Test Method **D2274**), n-hexane, n-heptane, methylbenzene, dimethylbenzene, isopropyl alcohol, methanol, or acetone. (**Warning**—Flammable. Health hazard.)

8. Sampling

8.1 Sampling shall be consistent with the procedures of Practice **D4057** or Practice **D4177**.

8.2 A sample of at least 100 mL is required to perform this test.

8.2.1 An aliquot of 100 mL may be removed from a larger sample for use in this test, provided the container is adequately shaken to homogenize the fuel prior to extracting the sample.

8.3 Sample containers shall be free from all contaminants. The sample container must be compatible with the hydrocarbon being sampled. If water is suspected in the hydrocarbon, then lined cans are required to prevent corrosion by-products from contaminating the fuel; otherwise, they are preferred.

NOTE 5—Test method results are known to be sensitive to trace contamination from sampling containers.

8.4 Under no circumstances shall the sample be pre-filtered after collection. Filtration media can remove surfactants, the detection of which is one of the purposes of this test method. If the test fuel sample is contaminated with particulate matter, allow it to settle before testing.

9. Preparation of Apparatus

9.1 Clean the test tubes thoroughly before carrying out this test. Only test tubes that are adequately cleaned can be used.

9.1.1 Remove traces of fuel from the test tubes and screw caps by first rinsing with a solvent as per 7.4. Follow this initial rinsing of the tubes with washing in hot, non-ionic detergent, rinsing with deionized water, then with a final rinse with acetone.

9.1.2 Following the washing described in 9.1.1, immerse the test tubes in a glass cleaning solution described in 7.3. The type of cleaning solution and conditions for its use may be established in each laboratory. The criterion for satisfactory cleaning shall be a matching of the quality of that obtained with chromic

acid cleaning solution. The use of a cleaning solution such as that described in 7.3 is the reference cleaning practice. Following cleaning with the glass cleaning solution, rinse with tap water, then distilled water and drain.

9.1.3 To check for proper cleanliness, completely fill the tube with distilled water, or buffer solution, then rapidly pour out the contents keeping the tube inverted. Glassware that drains in a manner such that the glass is evenly water-wet, with no voids or un-wetted areas immediately thereafter, is considered adequately cleaned.

NOTE 6—Inadequately cleaned glassware used in this test can give misleading indications of fuel contaminants. Use only test tubes that are adequately cleaned.

10. Procedure

10.1 Add 80 ± 1 mL at ambient temperature, typically 18 to 30°C, of the fuel to be tested to the test tube. Measure 20 ± 1 mL of water or the desired aqueous phase, also at ambient temperature, typically 18 to 30°C, into the test tube. Seal the test tube with a clean screw cap.

10.2 Shake the sealed test tube for $2 \text{ min} \pm 5 \text{ s}$, two to three strokes per second using 12 to 25-cm length strokes in a straight-line horizontal plane orientation, thus causing the fluids to roll from end to end. (**Warning**—Take care to avoid a swirling motion during shaking of the test tube, since swirling action tends to break any emulsion that might be formed.)

10.3 Immediately place the test tube on a vibration-free surface, and allow the contents to settle, undisturbed, for 5 min.

10.4 Without picking up the test tube, record the following while viewing the test tube in diffused light:

10.4.1 The volume of the aqueous layer to the nearest 0.5 mL.

10.4.2 The visual rating of the fuel using the line card and fuel clarity rating chart from ASTM Adjuncts **ADJD417601** and **ADJD7451**, respectively, as described in **Table 1**. (See also Test Method **D4176**.)

10.4.3 The degree of separation of the two phases in accordance with **Table 2**.

10.4.4 The condition of the interface in accordance with **Table 3**. The interface of the backlit sample should be viewed at an angle of 45°.

11. Report

11.1 The report shall include a reference to this test method and the following:

11.1.1 Total volume of the aqueous layer (not including unresolved emulsion) to the nearest 0.5 mL.

11.1.2 Rating of the fuel clarity (see **Table 1**).

TABLE 2 Fuel-Water Separation Rating

Rating	Appearance
1	Complete absence of all emulsions, or precipitates, or both, within either layer or upon the fuel layer
2	Same as Rating No. 1, except small air bubbles or small water droplets in the fuel layer
3	Emulsions or precipitates, or both, within either layer or upon the fuel layer, and droplets in the water layer or adhering to the test tube walls, or both, excluding the walls above the fuel layer

TABLE 3 Interface Condition Rating^A

Rating	Appearance
1	Clear and clean
1b	Clear bubbles covering not more than an estimated 50% of the interface and no film at the interface
2	Film at the interface
3	Scum at the interface (up to 1-mL emulsion)
4	Heavy scum at the interface (1-mL or greater emulsion)

^A Section 3 contains definitions of terms used in this table.

11.1.3 Rating of the degree of separation (see Table 2).

11.1.4 Rating of the condition of the interface (see Table 3).

11.1.5 Type of water or aqueous phase used in the test, if other than distilled water.

12. Precision and Bias

12.1 *Precision*—It is not practical to specify the precision of the fuel-water separation rating, interface condition rating, or

fuel clarity rating because the results of the ratings described in Tables 1-3, respectively, are purely qualitative. The tables assign numbers to descriptions of fuel-water separation, interface condition and fuel clarity ratings as a convenient guide to qualitative ratings.

12.2 *Bias*—This test method has no bias because water separation properties as defined only in terms of this test method.

13. Keywords

13.1 compression ignition fuel; demulsibility; diesel fuel; emulsion forming; fuel contaminants; fuel water interface; gasoline; light distillate fuel; middle distillate fuel; non-aviation fuel; spark ignition fuel; surfactants; water separation; water-soluble additives

APPENDIX

(Nonmandatory Information)

X1. ADDITIONAL INFORMATION

X1.1 *Aqueous Phase*—This test method specifies the use of distilled water, and the aqueous phase should be interpreted as distilled water unless otherwise noted. However, the methodology and observations contained in this test method are also applicable for testing with other aqueous phases, such as pH buffers, deionized water, process water, storage tank water samples, etc. The type of aqueous phase used in the test should be reported with the observations from the test.

X1.2 *Alcohols and Non-alcohol Oxygenates in Gasoline*—It is common for gasoline to contain certain alcohols or ethers as oxygen enriching agents for combustion improvement. When testing gasoline known to contain alcohols such as ethanol, for instance, one can expect to see the amount of recovered water increase due to the extraction of such species from the fuel by the aqueous phase. Since the amount of ethanol in the gasoline is usually known, it is possible to anticipate the volume increase to expect assuming that the aqueous phase will extract nearly all of the ethanol. This increase will not change how the fuel and interface are rated.

X1.2.1 Ether type oxygenates such as MTBE, ETBE, or TAME, or alcohols such as butanol, on the other hand, are not readily extracted into the aqueous phase and therefore do not cause an increase over the anticipated volume of aqueous phase recovered. Again, the presence of these oxygenates will not change how the fuel and interface are rated.

X1.3 *Biodiesel Blends*—The presence of biodiesel in distillate fuel may cause water to become emulsified in the fuel, resulting in haze in much the same manner as detergents and other surfactant additives. One can anticipate that as the level of biodiesel in the distillate fuel is increased from B5 (5% biodiesel in petroleum distillate fuel) to B20 (20% biodiesel in petroleum distillate fuel), the severity of the emulsion formed at the interface will increase and in some cases, may become quite stable. In any case, the water separation performance of a biodiesel tested under this method would be rated no differently than a traditional petroleum distillate fuel containing typical additives.

SUMMARY OF CHANGES

Subcommittee D02.14 has identified the location of selected changes to this standard since the last issue (D7451–08) that may impact the use of this standard.

(I) Promoted original Note 1 to 1.4 to further define the types of fuels that are not meant to be tested using this test method.

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