

Designation: D7451 - 21

# Standard Test Method for Water Separation Properties of Light and Middle Distillate, and Compression and Spark-Ignition Engine Fuels<sup>1</sup>

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 ( $\varepsilon$ ) 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.
- 1.7 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:<sup>2</sup>

D86 Test Method for Distillation of Petroleum Products and Liquid Fuels at Atmospheric Pressure

D396 Specification for Fuel Oils

D975 Specification for Diesel Fuel

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

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

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 7 Fuel

D6985 Specification for Middle Distillate Fuel Oil—Military Marine Applications (Withdrawn 2010)<sup>3</sup>

2.2 ASTM Adjuncts:

Distillate Fuel Bar Chart<sup>4</sup>

Fuel Clarity Rating Standard (physical or digital versions)<sup>5,6</sup>

# 3. Terminology

3.1 Definitions:

<sup>&</sup>lt;sup>1</sup> 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 D02.14 on Stability, Cleanliness and Compatibility of Liquid Fuels. Current edition approved Oct. 1, 2021. Published October 2021. Originally approved in 2008. Last previous edition approved in 2020 as D7451 – 20. DOI: 10.1520/D7451-21.

<sup>&</sup>lt;sup>2</sup> 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.

<sup>&</sup>lt;sup>3</sup> The last approved version of this historical standard is referenced on www.astm.org.

<sup>&</sup>lt;sup>4</sup> Available from ASTM International Headquarters. Order Adjunct No. ADJD417601. Original adjunct produced in 1991.

<sup>&</sup>lt;sup>5</sup> Available from ASTM International Headquarters. Order Adjunct No. ADJD7451. Original adjunct produced in 2008.

<sup>&</sup>lt;sup>6</sup> Available from ASTM International Headquarters. Order Adjunct No. ADJD7451-E-PDF. Original adjunct produced in 2019.

- 3.1.1 For definitions of terms used in this test method, refer to Terminology D4175.
- 3.1.2 *film*, *n*—thin, translucent layer that does not adhere to the wall of the glass test tube.
- 3.1.3 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.
- 3.1.4 *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:
- 3.2.1 *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 mL to 10 mL and 2 mL graduations from 10 mL 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 =  $200 \text{ mm} \pm 5 \text{ mm}$ .
  - 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 mm  $\pm$  5 mm.
  - 6.1.5 Tip length = 30 mm with a  $45^{\circ}$  taper.

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

- 6.2 Shaking Apparatus (Optional)<sup>7</sup>—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.
- 6.3 Rating Chart—The line card and fuel clarity rating chart from ASTM Adjuncts ADJD417601 and ADJD7451 (or ADJD7451-E-PDF) respectively. (See 10.4.2.)

Note 2—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.

## 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—Non-chromium containing cleaning solution. (**Warning**—Corrosive. Health hazard. Oxidizing agent.)

Note 3—Contact Subcommittee D02.14 for a list of possible suppliers for the glass cleaning solutions.

7.4 Cleaning Solvent, Reagent Grade—Trisolvent (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.

<sup>&</sup>lt;sup>7</sup> The sole source of supply of the shaking apparatus known to the committee at this time is Part No. 215086, Laciny Brothers Inc., St. Louis, MO, http://www.lacinybros.com. 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, which you may attend.



#### **TABLE 1 Fuel Clarity Ratings**

Note 1—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      | Clear and bright, equal to the base fuel  |
| 2      | Very slight haze  |
|        | <ul> <li>Black 10 pt bold Times New Roman font on a white background readily readable through tube, or</li> <li>Volume markings and numbers on glassware readily readable through tube</li> </ul>     |
| 3      | Slight haze   |
|        | <ul> <li>Black 10 pt bold Times New Roman font on a white background visible but not readable through tube, or</li> <li>Volume marking and numbers on glassware visible through tube</li> </ul>       |
| 4      | Slight haze   |
|        | <ul> <li>Black 10 pt bold Times New Roman font on a white background not readable through tube, or</li> <li>Volume markings and numbers on glassware visible but not readable through tube</li> </ul> |
| 5      | Hazy/translucent  |
|        | <ul> <li>Black 10 pt bold Times New Roman font on a white background not visible through tube, or</li> <li>Volume markings and numbers on glassware barely visible through tube</li> </ul>            |
| 6      | Opaque  |

- 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 4—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 5—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 mL  $\pm$  1 mL at ambient temperature, typically 18 °C to 30 °C, of the fuel to be tested to the test tube. Measure 20 mL  $\pm$  1 mL of water or the desired aqueous phase, also at ambient temperature, typically 18 °C 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 cm 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 (or ADJD7451-E-PDF) 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  $45^{\circ}$ .