

Standard Test Method for Water and Sediment in Middle Distillate Fuels by Centrifuge¹

This standard is issued under the fixed designation D2709; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope*

1.1 This test method covers the determination of the volume of free water and sediment (as a percentage of the sample) that is suspended in the bulk fuel in middle distillate fuels with viscosities in the range of 1.0 mm²/s to 4.1 mm²/s at 40 °C (1.0 cSt to 4.1 cSt at 104 °F) and densities in the range of 770 kg/m³ to 900 kg/m³ at 15 °C.

Note 1—Fuels corresponding to Specification D396 Grades No. 1 and 2, D975 Grades No. 1-D and 2-D, Specification D2880 Grades No. 0-GT, 1-GT and 2-GT, and Specification D3699 Grades No. 1-K and 2-K and similar middle distillate fuels and blendstocks will usually fall in this viscosity and density range. Test Method D1796 is intended for higher viscosity fuel oils.

- 1.2 The values stated in SI units are to be regarded as standard.
- 1.2.1 *Exception*—The non-SI values are for information only.
- 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:²

D396 Specification for Fuel Oils

D975 Specification for Diesel Fuel Oils

D1796 Test Method for Water and Sediment in Fuel Oils by the Centrifuge Method (Laboratory Procedure)

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

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

3. Terminology

- 3.1 For definitions of terms used in this test method, refer to Terminology D4175.
 - 3.2 Definitions:
- 3.2.1 *free water, n*—water in excess of that soluble in the fuel at the temperature of the test and appearing in the fuel as a haze, cloudiness, droplets, or water layer.
- 3.2.1.1 *Discussion*—Note that when there is a water layer in a biodiesel fuel blend, there can be water-soluble components present in the free water.
 - 3.3 Abbreviations:
 - 3.3.1 *rcf*—relative centrifugal force.

4. Summary of Test Method

4.1 A 100 mL sample of the undiluted fuel is centrifuged at a relative centrifugal force (see 6.2) of 800 for 10 min at 21 $^{\circ}$ C to 32 $^{\circ}$ C (70 $^{\circ}$ F to 90 $^{\circ}$ F) in a specified centrifuge tube. After centrifugation, the volume of free water and sediment that has settled into the tip of the centrifuge tube is read to the nearest 0.005 mL and reported as the volumetric percent water and sediment by centrifuge.

5. Significance and Use

- 5.1 This test method is used as an indication of free water and sediment suspended as haze, cloudiness, or droplets in middle distillate fuels such as Grades No. 1 and 2 fuel oil (Specification D396), Grades No. 1-D and 2-D diesel fuel (Specification D975), and Grades No. 0-GT, 1-GT, and 2-GT gas turbine fuels (Specification D2880), similar fuels and blendstocks used to make these fuels.
- 5.2 Appreciable amounts of free water and sediment in a fuel oil tend to cause fouling of fuel-handling facilities and to give trouble in the fuel system of a burner or engine. An accumulation of sediment in storage tanks and on filter screens can obstruct the flow of oil from the tank to the combustor. Free water in middle distillate fuels can cause corrosion of

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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.

TABLE 1 Rotation Speeds Applicable for Centrifuges of Various Diameters of Swing

| Diameter of Swing ^A | | r/min ^B at 800 |
|--------------------------------|------|---------------------------|
| in. | cm | rcf |
| 12 | 30.5 | 2160 |
| 13 | 33.0 | 2080 |
| 14 | 35.6 | 2000 |
| 15 | 38.1 | 1930 |
| 16 | 40.6 | 1870 |
| 17 | 43.2 | 1820 |
| 18 | 45.7 | 1770 |
| 19 | 48.3 | 1720 |
| 20 | 50.8 | 1680 |
| 21 | 53.3 | 1640 |
| 22 | 55.9 | 1600 |
| 23 | 58.4 | 1560 |
| 24 | 61.0 | 1530 |

^A Measured between tips of opposite tubes when in rotating position.

^B "r/min" is the correct SI symbol for the former term "rpm."

tanks and equipment, and if detergent is present, the water can cause emulsions or a hazy appearance. Free water can support microbiological growth at fuel-water interfaces in fuel systems.

6. Apparatus

6.1 Centrifuge, capable of spinning one or more pairs of filled centrifuge tubes at a speed which can be controlled to give a relative centrifugal force (rcf) of 800 ± 60 at the tip of the tubes. The revolving head, trunnion rings, and trunnion cups, including the cushions, shall be soundly constructed to withstand the maximum centrifugal force capable of being delivered by the power source. The trunnion cups and cushions shall support the tubes when the centrifuge is in motion. The centrifuge shall be enclosed by a metal shield or case strong enough to eliminate danger if any breakage occurs.

6.2 The data in Table 1 may be used to determine the centrifuge speed setting required for the centrifuge to meet relative centrifugal force requirements for this method and was developed using the following equations:

$$r/\min = 265 \sqrt{\text{rcf/}d}$$
 (1)

where:

rcf = relative centrifugal force, and

d = diameter of swing, in **inches**, measured between tips of opposite tubes when in rotating position or

$$r/\min = 422\sqrt{\operatorname{rcf}/d}$$
 (2)

where:

rcf = relative centrifugal force, and

 d = diameter of swing, in centimetres, measured between tips of opposite tubes when in rotating position.

6.3 *Centrifuge Tube*, equipped with a stopper, either:

6.3.1 Cone-shaped, 100 mL with capillary tip capable of measuring 0.01 mL and readable by estimation to 0.005 mL, or

6.3.2 Pear-shaped, 100 mL, with tube tip having graduations of 0.01 mL over the range of 0 mL to 0.2 mL.

7. Sampling

7.1 Sampling shall be consistent with the procedures of Practices D4057 or D4177.

7.2 The sample for a laboratory test will normally be an aliquot of a much larger sample taken for full or partial specification testing. Allow the sample container and its contents to equilibrate between 21 °C to 32 °C (70 °F to 90 °F). The laboratory test temperature should not be lower than the temperature at which the fuel is stored or used as too low a temperature can cause water haze to form from additional free water separation, making the test more severe.

8. Procedure

8.1 *Temperature Control*—After the sample container and its contents have equilibrated to laboratory temperature, between 21 °C to 32 °C (70 °F to 90 °F), agitate the full sample by hand or preferably by a mechanical shaker for 10 min to ensure homogeneity.

8.2 As soon as possible, to prevent losing any water or sediment by settling, fill the centrifuge tube to the 100 mL mark directly from the sample container. Stopper and place in a trunnion cup in the centrifuge.

8.3 Prepare a second centrifuge tube to the same mass as the centrifuge tube in 8.2, and place in the trunnion cup opposite the first tube to establish a balanced condition. It is practical to fill the second tube with another portion of the sample being evaluated. This practice gives a second tube of consistent mass to the first tube, and allows the sample to be analyzed in duplicate.

8.4 Close the centrifuge cover, turn on and centrifuge for 10 min at a speed sufficient to produce a relative centrifugal force (rcf) of 800 ± 60 at the tip of the whirling tubes. (For the relationship between diameter of swing, rcf, and r/min, see Table 1.)

8.5 When the centrifuge has stopped moving after the 10 min spin, remove the centrifuge tube, estimate and record the combined water and sediment volume at the bottom of the tube to the nearest 0.005 mL.

8.6 Since a 100 mL sample is used, the volume of water and sediment in millilitres is numerically the same as the percent by volume.

9. Report

9.1 Report the volume of the combined water and sediment read from the tube as the percentage of the total sample to the nearest 0.01 % and reference this test method.

9.1.1 Report results lower than 0.01 mL as <0.01 % by volume.

10. Precision and Bias³

10.1 *Precision*—The precision of the procedure in this test method, for measuring the percent by volume amount of combined free water and sediment in middle distillate fuels by centrifuge was determined by an interlaboratory study (ILS) performed by six individuals using 13 samples at a common site.

³ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1308.

Note 2-Precision data for this test used pear-shaped tubes.

10.1.1 Repeatability—The difference between successive measured volume percent amounts of combined water and sediment obtained by the same operator using the same centrifuge and type of pear-shaped tube under constant operating conditions on identical middle distillate fuel samples at the same site would, in the long run and in the normal and correct operation of the test method, exceed 0.014 % by volume in 1 case in 20.

10.1.2 *Reproducibility*—The difference between two single independent measurements of volume percent amounts of combined water and sediment obtained by different operator/

centrifuge pairs using the same type of pear-shaped tube on identical middle distillate fuel samples at the same site would, in the long run, in the normal and correct operation of the test method, exceed 0.041 % by volume in 1 case in 20.

10.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for this procedure for measuring water, the bias is not available for this test method.

11. Keywords

11.1 centrifuge; centrifuge tube; middle distillate fuel; water and sediment

SUMMARY OF CHANGES

Subcommittee D02.14 has identified the location of selected changes to this standard since the last issue $(D2709 - 96 (2011)^{\epsilon 1})$ that may impact the use of this standard. (Approved April 1, 2016.)

- (1) Numerous editorial changes were made throughout the standard to align it with current Form and Style requirements and correct SI units.
- (2) Scope—1.1, 1.2, and Note 1were revised to include Specification D396, related products and blendstocks, and update the formatting of units.
- (3) Subsection 2.1—Terminology D4175 and Practice D4177 were added.
- (4) Distillate fuel definition was deleted from Section 3; subsection 3.2.1, a definition for free water, was added.
- (5) Subsection 3.1 was added to reference Terminology D4175.
- (6) Subsection 3.3, Abbreviations, specifically 3.3.1 "rcf" was added.
- (7) Subsection 4.1 was modified to simplify the description of the centrifuge tube.

- (8) Subsections 5.1 and 5.2 were clarified.
- (9) Table 1, the column for "Rpm at 500 rcf" was deleted as not being required; added new footnote B for "r/min."
- (10) Wording in subsections 6.1, 6.2, and 6.3 was clarified.
- (11) Subsection 7.1, added reference to Practice D4177.
- (12) Subsection 7.2, clarified wording.
- (13) Subsection 8.1, "Warning—flammable" was deleted.
- (14) Subsection 8.2 was modified and rephrased to generate new subsections 8.3, 8.4, 8.5, and 8.6.
- (15) Report subsection 9.1 was revised, and new subsection 9.1.1 was added to correct the reporting of low results.
- (16) Precision subsection 10.1 was revised.

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