Designation: D4176 – 21a

Standard Test Method for Free Water and Particulate Contamination in Distillate Fuels (Visual Inspection Procedures)[1]

This standard is issued under the fixed designation D4176; 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 two procedures for estimating the presence of suspended free water and solid particulate contamination in distillate fuels having distillation end points below 400 °C and an ASTM color of 5 or less.

1.1.1 Both procedures can be used as field tests at storage temperatures, or as laboratory tests at controlled temperatures.

1.1.2 Procedure 1 provides a rapid pass/fail method for contamination. Procedure 2 provides a gross numerical rating of haze appearance.

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

1.4 *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:*[2]

D1500 Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)

D2276 Test Method for Particulate Contaminant in Aviation Fuel by Line Sampling

D2709 Test Method for Water and Sediment in Middle Distillate Fuels by Centrifuge

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)

D4860 Test Method for Free Water and Particulate Contamination in Middle Distillate Fuels (Clear and Bright Numerical Rating)

D8148 Test Method for Spectroscopic Determination of Haze in Fuels

2.2 *ASTM Adjuncts:*

Distillate Fuel Bar Chart[3]

Distillate Fuel Haze Rating Standard[4]

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this test method, refer to Terminology D4175.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *clear-and-bright* (also termed *clean-and-bright*), *n*—a condition in which the fuel is free of haze or cloudiness.

3.2.2 *free water, n*—water in excess of that soluble in the liquid sample (fuel) at the temperature of the test and usually appearing in the liquid sample (fuel) as a haze (cloudiness), droplets, or water layer.

3.2.3 *particulates, n*—small solid or semisolid particles, sometimes referred to as silt or sediment, that may or may not be suspended in the fuel as a result of contamination by air-blown dusts, corrosion by-products, fuel instability, or protective-coating deterioration.

4. Summary of Test Method

4.1 In Procedure 1 approximately 900 mL of fuel is placed into a clear, glass, 1 L jar and is examined visually for clarity.

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricantsand is the responsibility of Subcommittee D02.14 on Stability, Cleanliness and Compatibility 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.

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

[4] Available from ASTM International Headquarters. Order Adjunct No. ADJD417602. Original adjunct produced in 1991.

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

The sample is then swirled and examined for visual sediment or water drops below the vortex.

4.2 In Procedure 2 approximately 900 mL of fuel is placed into a clear, glass, 1 L jar and is examined visually for clarity. Fuel clarity is rated by placing a standard bar chart behind the sample and comparing its visual appearance with the standard haze rating photos. The sample is then swirled and examined for visual sediment or water drops below the vortex.

4.3 When field testing, both Procedures 1 and 2 are performed immediately after sampling and at storage temperature conditions.

4.4 When lab testing, both Procedures 1 and 2 are performed after the sample has equilibrated at the test temperature of interest.

5. Significance and Use

5.1 It has long been the practice to include in fuel specifications a requirement that the fuel be *clear and bright and free of visible particulate matter* (see Note 1). However, there has been no standard method for making this determination so that practices have differed. This test method provides standard procedures for the test.

NOTE 1—*Clean and bright* is sometimes used in place of *clear and bright.* The meaning is identical.

5.2 Procedure 1 provides a rapid pass/fail method for contamination in a distillate fuel. Procedure 2 provides a gross numerical rating of haze appearance, primarily as a communication tool. Other test methods, including Test Methods D2276, D2709, and D4860, permit quantitative determinations of contaminants. No relationship has been established between Procedure 2 and these quantitative methods.

5.2.1 Test Method D8148 has established a correlating relationship with Procedure 2 appearance rating numbers by reporting a correlating instrument haze rating (IHR) based upon its spectroscopically determined haze clarity index (HCI). Supporting data can be found in RR:D02-1876.[5]

5.3 Limited laboratory evaluations of samples that have failed this *clear and bright* test indicate that an experienced tester can detect as little as 40 ppm of free water in the fuel.

6. Interferences

6.1 When a fuel is tested at low temperatures at or below the cloud point temperature of the fuel, small amounts of solid wax particles may be confused with a water-induced haze or cloudiness.

6.2 If an attempt is made to use the test with fuels darker than a color rating of 5 in Test Method D1500, the presence of free water or particulate could be obscured and missed by the viewer.

7. Apparatus

7.1 *Cylindrical Container,* clear-glass, capable of holding 1.0 L ± 0.1 L of fuel and having a diameter of 100 mm ± 10 mm.

7.2 *Paper Card (Bar Chart),* [3] laminated in clear plastic, having five parallel lines of different widths and meeting the following description:

7.2.1 *Characteristics of Card Stock,* white paper 120 mm by 180 mm long.

7.2.2 *Line Color, Width, and Spacing,* five black lines of increasing widths, commencing with a line 0.6 mm wide, the second line 1.6 mm wide, and each succeeding line 1.6 mm wider to a maximum of 6.4 mm.

7.2.3 The lines shall be numbered from 1 through 5, with the thinnest line being No. 1.

7.3 A series of standard photographs[4] of the bar chart through samples of differing haze levels, numbered from 1 through 6. Photograph No. 1 is the clearest, while No. 6 represents the densest haze.

7.4 The differences between these haze levels are arbitrary and are not intended to represent equivalent increases in suspended water content or particulates. It is essential, therefore, that only the proper approved photos be used.

7.5 *Temperature Sensing Device* (TSD), capable of monitoring the observed test temperature to within an accuracy of ±0.5 °C (±1 °F) for use in laboratory tests that require measurements to be made at a specific temperature.

7.6 *Temperature-Controlled Bath,* of suitable dimensions and capable of controlling the sample container temperature within ±0.5 °C (±1 °F) of the desired temperature for laboratory tests that require measurements to be made at a specific temperature.

8. Sampling

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

8.2 Draw the sample directly into the sample container using the following procedure:

8.2.1 Be sure the sampling valve is free of loose solid contaminants. If rust or other loose encrustations are present, remove with a cloth; then flush the sampling valve prior to taking the actual sample.

8.2.2 Rinse a clean test container thoroughly with the fuel being sampled. (**Warning—**Flammable. See Annex A1.1.)

8.2.3 Draw approximately 900 mL of fuel into the container as rapidly as possible. Use a full flush rather than permitting the fuel sample to trickle out.

9. Sample Preparation

9.1 *Field Testing*—Both Procedures 1 and 2 are to be performed immediately after drawing the sample. Record the approximate sample storage temperature and the approximate ambient temperature at which the test is performed.

9.2 *Laboratory Testing:*

9.2.1 Do not subsample or transfer the sample to a secondary container. Perform the test with the sample drawn in the original sample container.

9.2.2 Replace the sample container's closure with an air tight closure through which a calibrated temperature sensing device is immersed in the sample. Allow the sample container

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1876. Contact ASTM Customer Service at service@astm.org.

to equilibrate in a temperature-controlled bath, bringing it to desired test temperature within the allowed tolerance. Periodically agitate the sample in a manner sufficient to homogenize the bulk of the sample (water droplets and particulates, if present, do not need to be evenly dispersed).

9.2.3 Remove the sample container from the temperature-controlled bath, wipe dry with an absorbent material (if a liquid bath is used), and perform the desired procedure(s) with minimal delay after removal. Remove the temperature sensing device after recording the sample test temperature.

10. Procedure

10.1 *Procedure 1*—Check visually for evidence of water or particulate contamination. Hold the sample up to the light and visually examine for haze or lack of clarity. Swirl the sample to produce a vortex and examine the bottom of the vortex for particulate matter. Record the visual clarity as clear and bright or not clear and bright. Record if particulate matter or water was or was not viewed at the bottom of the vortex.

10.2 *Procedure 2*—Place the sample container into a well lighted area, avoiding light reflections on the front of the container as much as possible. Place the bar chart directly behind the container, with the lines toward the container and parallel with the container bottom. The narrowest line should be at the bottom of the chart.

10.2.1 Directly facing the container and bar chart, compare the appearance of the bar chart through the sample with the standard photographs. Place the photographs next to the container so that they are lighted similarly to the sample. Select the photograph closest in appearance to the sample. Ignore differences in fuel color. Notice that the differences between photographs consist both of the successive disappearance of lines as well as a gradual lightening of all the lines. Record the number of the photograph closest in appearance as the rating of the sample.

10.2.1.1 As a non-mandatory supplement to Procedure 2, data gathered using Test Method D8148 may be used to document the visual appearance ratings by providing a quantitative measure of dispersed water or other suspended matter known as the haze clarity index (HCI). However, Test Method D8148 is not an approved replacement for Test Method D4176.

10.2.2 Remove the bar chart and swirl the sample container to produce a vortex. Examine the bottom of the vortex for particulate matter and water droplets. Record the presence of any particulates or water.

10.2.3 Also record any special observation, such as a particularly heavy contamination with water or solids or a darker than usual color which made ratings difficult.

11. Report

11.1 For field tests, the report shall provide an adequate description of the sample including the type of fuel, the source of the fuel (the sampling point), and the date, time, and approximate temperature of the sample. The report shall also indicate the approximate temperature at which the test was run and that a field test was performed.

11.1.1 For lab tests, the report shall include the test temperature at which the sample was analyzed. The report shall also indicate that a lab test was performed.

11.2 *Procedure 1*—The results of the test shall be shown as *pass* if: (A) The sample has been found to be *clear and bright* on visual observance, and (B) If there is no water or particulates observed at the bottom of the vortex. The results shall be reported as *fail* if (A) or (B) conditions are not met. The reason for any failure should also be recorded.

11.2.1 In addition to the *pass/fail* reporting requirements in 11.2, the individual sample qualities may be reported as follows:

Clear and Bright—Pass or Fail
Free Water—Pass (absent) or Fail (present)
Particulates—Pass (absent) or Fail (present)

11.3 *Procedure 2*—The report shall include the numerical rating of the sample and a note as to whether any particles or water droplets were found on the bottom of the sample container. Any special or unusual observations, such as darker than usual fuel color, shall also be reported.

11.3.1 If available, in addition to the reporting requirements in 11.3, Test Method D8148 HCI data may be reported.

12. Precision and Bias

12.1 *Procedure 1*—It is not practical to specify the precision of the procedure because the test is a *pass/fail* test, not a quantitative measurement.

12.2 *Procedure 2*—A rigorous precision statement cannot be developed according to ASTM Research Report RR:D02-1007 because the intervals between the rating steps are not known to be equal. However, if the intervals are assumed to be equal, the following estimates of precision will apply. Examination of the results of a cooperative test program supports these estimates.

12.2.1 *Repeatability*—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test samples would, in the long run, in the normal and correct operation of the test method, exceed one number in only one case in twenty.

12.2.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test materials would, in the long run, exceed two numbers only in one case out of twenty.

NOTE 2—The reproducibility values above were estimated from results obtained at the same location and on the same day by different operators/instruments testing identical samples at the same time as closely as possible. The instability of typical fuel hazes introduces unpredictable variations for samples tested at different times and makes the shipment of samples to different locations impractical. The basis of this precision statement and the program comparing results obtained by this procedure by 12 operators on 24 fuel samples are available from ASTM Headquarters.

12.3 No justifiable statement can be made on the bias of either procedure in Test Method D4176 because a fuel haze can result from a number of causes and relationship with a single absolute quantitative method is not possible.

13. Keywords

13.1 cleanliness; distillate fuel; free water; particulate contamination; visual inspection; visual ratings