Designation: D2274 – 14 (Reapproved 2019)

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Standard Test Method for Oxidation Stability of Distillate Fuel Oil (Accelerated Method)[1]

This standard is issued under the fixed designation D2274; 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 measurement of the inherent stability of middle distillate petroleum fuels under specified oxidizing conditions at 95 °C.

NOTE 1—Fuels used in establishing the precision measures for this test method were described as gas oil, diesel fuel, No. 2 heating oil, and DFM, a Navy distillate fuel suitable for diesels, boilers, and gas turbines. (The term DFM is no longer used when referring to fuel meeting MIL-F-16884 requirements; rather it is called F76 as it conforms to NATO F76 requirements.) While the test method may be used for fuels outside the range of these fuels, the precision measures may not apply.

1.2 This test method is not applicable to fuels containing residual oil. This test method has not been validated for testing biodiesel, such as meeting Specification D6751 or blends of middle distillates and biodiesel, such as meeting Specification D7467, or both. Test Method D7462 has been determined to be suitable for testing B100 and all blends of middle distillates and biodiesel.

NOTE 2—No. 1 and No. 2 grades in Specifications D396 or D975 currently allow up to 5 % biodiesel meeting Specification D6751. Samples containing biodiesel can result in partial dissolution or compromise of the membrane filter and give erroneous results.

1.3 The values given in SI units are to be regarded as the standard. The values in parentheses are for information only.

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, 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:*[2]

D381 Test Method for Gum Content in Fuels by Jet Evaporation

D396 Specification for Fuel Oils

D943 Test Method for Oxidation Characteristics of Inhibited Mineral Oils

D975 Specification for Diesel Fuel

D1193 Specification for Reagent Water

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

D4625 Test Method for Middle Distillate Fuel Storage Stability at 43 °C (110 °F)

D6751 Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels

D7462 Test Method for Oxidation Stability of Biodiesel (B100) and Blends of Biodiesel with Middle Distillate Petroleum Fuel (Accelerated Method) (Withdrawn 2016)[3]

D7467 Specification for Diesel Fuel Oil, Biodiesel Blend (B6 to B20)

2.2 *Military Specification:*[4]

MIL-F-16884 Fuel, Navy Distillate

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

[1] This test method is under the jurisdiction of 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.

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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] The last approved version of this historical standard is referenced on www.astm.org.

[4] Available from Standardization Documents Order Desk, Bldg. 4, 700 Robbins Ave., Philadelphia, PA 19111-5098. Attn: NPODS

3.1.1 *adherent insolubles* (formerly adherent gum), *n*—material which is produced in the course of stressing distillate fuel under the conditions of this test and which adheres to the glassware after fuel has been flushed from the system.

3.1.2 *filterable insolubles, n*—material, which is produced in the course of stressing distillate fuel under the conditions of this test, which is capable of being removed from the fuel by filtration. This includes both material suspended in the fuel and material easily removed from the oxidation cell and oxygen delivery tube with hydrocarbon solvent.

3.1.3 *inherent stability, n*—the resistance to change when the fuel is exposed to air, but in the absence of other environmental factors such as water, or reactive metals and dirt.

3.1.4 *total insolubles, n*—arithmetic sum of the adherent and filterable insolubles.

3.1.5 *zero time, n*—the time the first of a batch of oxidation cells is placed in the heating bath.

3.1.5.1 *Discussion*—This is the time taken as the start of the 16 h of residence in the heating bath.

4. Summary of Test Method

4.1 A 350 mL volume of filtered middle distillate fuel is aged at 95 °C (203 °F) for 16 h while oxygen is bubbled through the sample at a rate of 3 L/h. After aging, the sample is cooled to approximately room temperature before filtering to obtain the filterable insolubles quantity. Adherent insolubles are then removed from the oxidation cell and associated glassware with trisolvent. The trisolvent is evaporated to obtain the quantity of adherent insolubles. The sum of the filterable and adherent insolubles, expressed as milligrams per 100 mL, is reported as total insolubles.

5. Significance and Use

5.1 This test method provides a basis for the estimation of the oxidation stability of middle distillate fuels such as No. 2 fuel oil.

5.2 The test method may not provide a prediction of the quantity of insolubles that will form in field storage over any given period of time. The amount of insolubles formed in such field storage is subject to the specific conditions which are too variable for this test method to predict accurately.

5.3 Test Method D2274 yields results more rapidly than Test Method D4625, the 43 °C bottle test. However, as a result of the significantly elevated temperature and the pure oxygen atmosphere, the nature and amount of insolubles may deviate to a greater extent than Test Method D4625 from those formed in field storage.

6. Interferences

6.1 Oxidation is a major chemical process causing adherent and filterable insolubles to form. Any substance such as copper or chromium that catalyzes oxidation reactions will cause greater quantities of insolubles to form. Since the apparatus used in this test can also be used in Test Method D943, where coils of copper and steel are used, it is important that any residues that could contain these metals be eliminated from the apparatus by thorough cleaning prior to use. Similarly, to preclude the presence of chromium ions, as well as to protect laboratory personnel from potential harm, chromic acid shall not be used for cleaning glassware in the practice of this method.

6.2 It has been found that commercial grades of acetone, if used in the trisolvent, can have impurities which cause an apparently greater level of adherent insolubles to be measured. It is, therefore, critical that only reagent (or higher) grade materials be used in preparing the trisolvent mixture.

6.3 Ultraviolet light exposure has been found to increase the amount of total insolubles. Therefore, the fuel being tested shall be shielded from direct exposure to ultraviolet light (sunlight or fluorescent). Conduct all sampling, measuring, filtration, and weighing away from direct sunlight and in as dark an area as would be compatible with other laboratory operations. Storage before stress, the stress period and cool-down after stressing shall be in the dark.

7. Apparatus

NOTE 3—It is suggested that all measuring equipment be calibrated according to manufacturer's instructions on a periodic basis to assure consistency of results.

7.1 *Oxidation Cell,* of borosilicate glass, as shown in Fig. 1, shall consist of a test tube, condenser, and oxygen delivery tube. This cell is identical to that used in Test Method D943.

7.2 *Heating Bath,* with a thermostatically controlled liquid medium, shall be capable of maintaining the bath temperature at 95 °C ± 0.2 °C (203 °F ± 0.4 °F). It shall be fitted with a suitable stirring device to provide a uniform temperature throughout the bath. It shall be large enough to hold the desired number of oxidation cells immersed to a depth of approximately 350 mm. Further, the bath construction must permit shielding the fuel samples in the oxidation cells from light while they are undergoing oxidation.[5]

7.3 *Flowmeters,* shall have a capability of measuring 3 L/h ± 0.3 L/h of oxygen. One flowmeter shall be provided for each oxidation cell.

7.4 *Filter Drying Oven,* shall be capable of safely evaporating the solvent at 80 °C ± 2 °C (176 °F ± 4 °F) for the drying of filters.

7.5 *Glassware Drying Oven,* shall be capable of drying glassware at 105 °C ± 5 °C (221 °F ± 9 °F).

7.6 *Filtration Assembly,* see Fig. 2, shall be capable of holding the filters described in 7.7.

7.7 *Filter Media*[6], 47 mm diameter cellulose ester surfactant-free membrane filters with a nominal pore size of 0.8 µm.

7.7.1 Single filters are to be used for prefiltration.

[5] This apparatus is available from suppliers of specialty petroleum testing equipment.

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1012. Filters may be qualified using the procedure in this research report.