



Designation: D2274 – 03a (Reapproved 2008)



Designation: 388/97

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 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 or significant amounts of components derived from non-petroleum sources.

1.3 The values given in acceptable 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:²

- D381 Test Method for Gum Content in Fuels by Jet Evaporation
- D943 Test Method for Oxidation Characteristics of Inhibited Mineral Oils
- D1193 Specification for Reagent Water

¹ This test method is under the jurisdiction of 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.

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)

2.2 Military Specification:³

MIL-F-16884 Fuel, Navy Distillate

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *adherent insolubles* (formerly adherent gum)—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*—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*—the resistance to change when exposed to air, but in the absence of other environmental factors such as water, or reactive metallic surfaces and dirt.

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

3.1.5 *zero time*—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

³ Available from Standardization Documents Order Desk, Bldg. 4, 700 Robbins Ave., Philadelphia, PA 19111-5098. Attn: NPODS

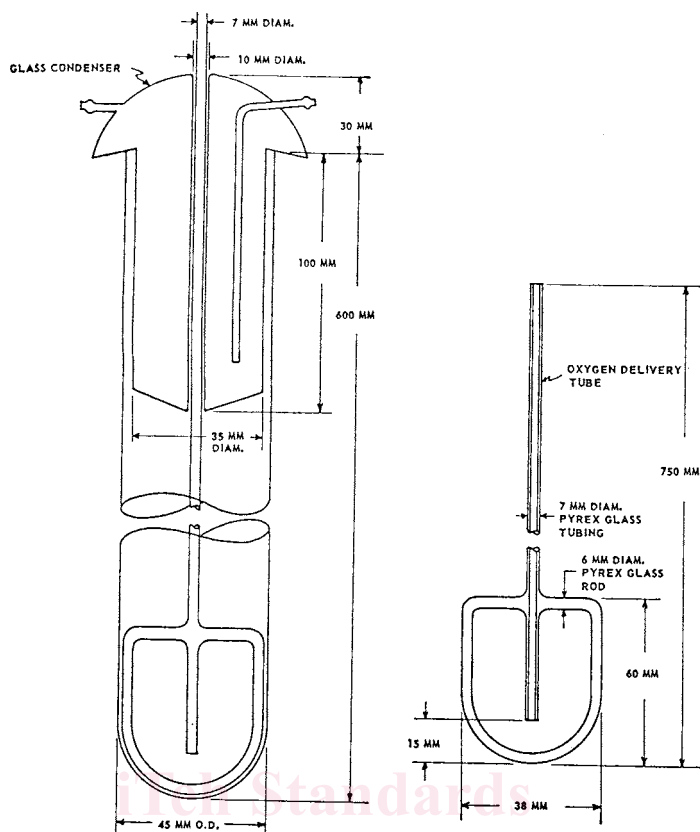


FIG. 1 Oxidation Cell

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 storage 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 2—It is suggested that all 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.

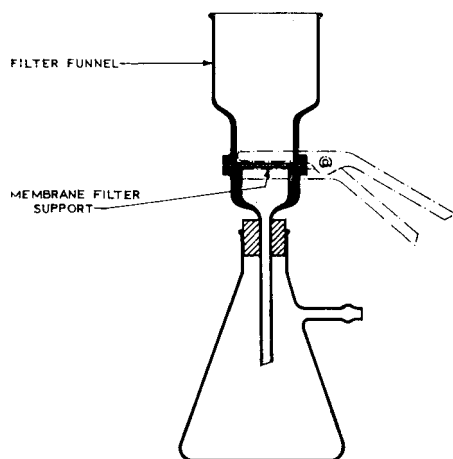


FIG. 2 Apparatus for Determining Filterable Insolubles

7.2 *Heating Bath*, with a thermostatically controlled liquid medium, shall be capable of maintaining the bath temperature at $95 \pm 0.2^\circ\text{C}$ ($203 \pm 0.4^\circ\text{F}$). 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.⁴

7.3 *Flowmeters*, shall have a capability of measuring 3 ± 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 $80^\circ \pm 2^\circ\text{C}$ ($176^\circ \pm 4^\circ\text{F}$) for the drying of filter materials.

7.5 *Glassware Drying Oven*, shall be capable of drying glassware at $105^\circ \pm 5^\circ\text{C}$ ($221 \pm 9^\circ\text{F}$).

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

7.7 *Filter Media*⁵, 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.

7.7.2 A matched weight pair of filters or alternatively, a preweighed control and sample, filters shall be used for determination of filterable insolubles

7.8 *Evaporating Vessel*, borosilicate glass beaker, 200-mL capacity, tall style.

7.9 *Hot Plate*, capable of heating a liquid in the evaporating vessel (7.8) to 135°C (275°F).

8. Reagents and Materials

8.1 *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.⁶ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean reagent water as defined by Type III of Specification D1193.

8.3 *2,2,4-trimethylpentane (isooctane)*, 99.75 % purity pre-filtered through a filter medium of the type specified in 7.7.

8.4 *Oxygen*, 99.5 % purity or better. When the oxygen is delivered through a plant system of piping, a filter shall be provided adjacent to the constant temperature bath to prevent the introduction of line debris or moisture into the oxidation cells; a pressure regulator adequate to maintain a constant flow of gas through the apparatus shall also be used. A tank of oxygen of the specified purity can be used provided it is equipped with a two-stage pressure regulator. (**Warning**—Oxygen vigorously accelerates combustion. Do not use equipment having exposed surfaces containing oil or grease.)

8.5 *Trisolvent*, a mixture of equal volumes of acetone, methanol, and toluene. See 8.1. (**Warning**—It is particularly important that technical, commercial, practical, or industrial grades (however they are designated by the particular manufacturer) are not to be used, as their use may lead to apparently increased levels of adherent insolubles.) (**Warning**—Fire hazard, toxic.)

9. Samples and Sampling

9.1 When obtaining samples for the laboratory, follow Practices D4057 or D4177, or other standard practice capable of providing representative samples.

9.2 Analyze fuel samples as soon as possible after receipt. When a fuel cannot be tested within one day, blanket it with an inert gas such as oxygen-free nitrogen, argon, or helium and store at a temperature no higher than 10°C (50°F) but not lower than the cloud point. (**Warning**—Plastic containers are not acceptable for samples due to the potential for leaching of plasticizers. Samples should be taken preferably in metal cans previously cleaned according to Practice D4057. Borosilicate glass containers can be used if they are wrapped or boxed to exclude light. Do not use soft (soda) glass containers.)

9.3 *Test Samples*—Reduction of the laboratory sample to test sample size (about 400 mL for each determination) depends upon the size of sample received by the laboratory. If the laboratory sample is stored in a tank, drum, or 19-L (5-gal) or larger can, use the pertinent procedures of Practice D4057. Thoroughly mix smaller laboratory samples by shaking, rolling, or other techniques before taking an aliquot portion by pouring, pipetting, or other means. Clean any tube, thief, pipet, beaker, or other substance that is to contact the laboratory sample with trisolvent and rinse with a portion of the sample prior to use. Prior to mixing thoroughly and taking an aliquot,

⁴ This apparatus is available from suppliers of specialty petroleum testing equipment.

⁵ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report D02-1012. Filters may be qualified using the procedure in this research report.

⁶ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.