Designation: D5304 – 20

Standard Test Method for Assessing Middle Distillate Fuel Storage Stability by Oxygen Overpressure[1]

This standard is issued under the fixed designation D5304; 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 a procedure for assessing the potential storage stability of middle distillate fuels such as Grade No. 1D and Grade No. 2D diesel fuels, in accordance with Specification D975.

1.2 This test method is applicable to either freshly refined fuels or fuels already in storage.

1.3 This test method is suitable for fuels containing stabilizer additives as well as fuels containing no such additives. However, fuels additized with dispersant additives, including dispersant-containing stability additives, may be ranked inaccurately using this test method compared to fuels that are not additized with dispersant additives.

1.4 Appendix X1 provides information on other suggested test times and temperatures for which this test method may be used.

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

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.* For specific warning statements, see 4.1, 6.2, 6.3, and 7.4.

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:*[2]

D525 Test Method for Oxidation Stability of Gasoline (Induction Period Method)

D975 Specification for Diesel Fuel

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

D4306 Practice for Aviation Fuel Sample Containers for Tests Affected by Trace Contamination

E1 Specification for ASTM Liquid-in-Glass Thermometers

3. Terminology

3.1 *Definitions:*

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

3.1.2 *membrane filter, n*—a porous article of closely controlled pore size through which a liquid is passed to separate matter in suspension.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *oxygen overpressure, n*—partial pressures of oxygen higher than that of air at atmospheric pressure.

3.2.2 *potential storage stability, n*—the tendency of a fuel to form insolubles under the conditions of this test method.

3.2.3 *reactor, n*—any vessel capable of sustaining pressures and temperatures above ambient, sometimes designated pressure vessel.

3.2.4 *weighing assembly, n*—a set of two filters and two aluminum weighing dishes used to determine total insolubles for each sample or blank.

[1] 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.

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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.

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

4. Summary of Test Method

4.1 A 100 mL aliquot of filtered fuel is placed in a borosilicate glass container. The container is placed in a pressure vessel which has been preheated to 90 °C. The pressure vessel is pressurized with oxygen to 800 kPa (absolute) (100 psig) for the duration of the test. The pressure vessel is placed in a forced air oven at 90 °C for 16 h. (**Warning—** Observe all normal precautions while using oxygen under pressure and at high temperatures in the presence of combustible liquids. Appropriate shielding should be used for any containers under pressure. Pressurize and depressurize the containers *slowly* using appropriate personnel shielding. Never attempt to open the pressure vessel while it is pressurized. All fuel and solvent handling should be done in an appropriate fume hood only.) After aging and cooling, the total amount of fuel insoluble products is determined gravimetrically and corrected according to blank determinations.

5. Significance and Use

5.1 The results of this test method are useful in ranking a specific fuel sample against other specific fuel samples or standards when tested under identical conditions. Specific fuel samples containing dispersant additives, such as dispersant-containing stability additives, have shown inaccurate ranking against fuel samples that do not contain dispersant additives using this test method.[3] This test method is not meant to relate a specific fuel to specific field handling and storage conditions. The formation of insolubles is affected by the material present in the storage container and by the ambient conditions. Since this test method is conducted in glass under standardized conditions, the results from different fuels can be compared on a common basis.

6. Apparatus

6.1 *Sample or Blank Container,* a brown borosilicate glass bottle capable of holding 100 mL of sample but with total volume less than 200 mL, or a Test Method D525 glass insert. A top closure of aluminum foil, perforated with small holes for breathing, will be required if there is more than one sample per pressure vessel.

6.2 *Pressure Vessel(s) (Reactor(s)),* designed for safe operating pressures of 800 kPa (100 psig) in oxygen service (**Warning—**See 4.1.) equipped with a pressure gauge. (**Warning—**The pressure for the procedure in this test method is 800 kPa (absolute) (100 psig). Many pressure gauges are calibrated in kPa (gauge). For such gauges, the test pressure would be 700 kPa (gauge). Maximum gauge gradations should be 20 kPa (5 psig)). The gauge should be calibrated against standards, and capable of holding the four sample containers. Pressure vessels having internal volumes from 250 mL to 8000 mL have been used and found to be suitable. If 250 mL vessels such as Test Method D525 oxidation vessels are used, four will be required. The larger volume pressure vessels can accommodate multiple sample or blank containers). The pressure vessel(s) (reactor(s)) must be obtained only from commercial sources.

6.3 *Heater,* capable of maintaining the test temperature at 90 °C ± 1 °C for the duration of the test. Ensure heater temperature uniformity. Heater shall be capable of holding the pressure vessel(s) (reactor(s)) described in 6.2. Static (non-forced air) ovens and unstirred liquid medium baths, such as the Test Method D525 water bath, are unsuitable. Use of these heaters will give erroneous results due to nonuniformity of temperature.) The reactor should be placed in an oven so that the entire reactor is uniformly receiving heat. (**Warning—**Use of an explosion-proof oven is required.)

6.4 *Drying Oven,* forced air operated at 110 °C ± 5 °C. Static ovens or vacuum ovens are not suitable.

6.5 *Water Aspirator or Vacuum Pump,* as a source of vacuum.

6.6 *Aluminum Dish* (disposable), capable of holding 47 mm diameter filters and 30 mL of adherent insolubles solvent.

6.7 *Analytical Balance,* capable of weighing to the nearest 0.1 mg.

6.8 *Filtration System*—Arrange the following components as shown in Fig. 1.

6.8.1 *Funnel and Funnel Base,* with filter support for a 47 mm diameter membrane and a locking ring or spring action clip.

6.8.2 *Ground/Bond Wire,* 0.912 mm to 2.59 mm (No. 10 through No. 19) bare-stranded, flexible, stainless steel, or copper installed in the flasks and grounded as shown in Fig. 1.

6.8.3 *Receiving Flask,* 1.5 L or larger borosilicate glass vacuum filter flask, which the filtration apparatus fits into, equipped with a sidearm to connect to the safety flask.

6.8.4 *Safety Flask,* 1.5 L or larger borosilicate glass vacuum filter flask equipped with a sidearm to connect the vacuum system. A fuel and solvent resistant rubber hose through which the grounding wire passes shall connect the sidearm of the receiving flask to the tube passing through the rubber stopper in the top of the safety flask.

6.9 *Hot Plate,* capable of operating at low heat so that 10 mL of toluene placed in the aluminum dish described in 6.6 will require 10 min to 25 min to evaporate.

6.10 *Thermometer,* conforming to the requirements prescribed in Specification E1. Temperature measuring devices such as ASTM 61C (IP No. 63C), liquid-in-glass thermometers, thermocouples, or platinum resistance thermometers that provide equivalent or better accuracy and precision may be used.

6.11 *Forceps,* approximately 12 cm long, flat-bladed, with non-serrated, non-pointed tips.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where

[3] Williams, S., "Engineering Investigation of 2004/05 East Coast F-76 Rapid Fuel Degradation," NAVAIRSYSCOM REPORT 4451/06-006, August 14, 2006.