Designation: D 5304 – 03

An American National Standard

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

This standard is issued under the fixed designation D 5304; 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 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 D 975.

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.

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 in SI units are to be regarded as the 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 and health practices and determine the applicability of regulatory limitations prior to use.* For specific warning statements, see 4.1, 6.2, 6.3, 7.4, 10.1, and 10.2.

2. Referenced Documents

2.1 *ASTM Standards:*

D 525 Test Method for Oxidation Stability of Gasoline (Induction Period Method)[2]

D 975 Specification for Diesel Fuel Oils[2]

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products[3]

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products[3]

D 4306 Practice for Aviation Fuel Sample Containers for Tests Affected by Trace Contamination[3]

D 4625 Test Method for Distillate Fuel Storage Stability at 43°C (110°F)[3]

E 1 Specification for ASTM Thermometers[4]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *membrane filter, n*—a thin medium of closely controlled pore size through which a liquid is passed and on which particulate matter in suspension is retained.

3.1.2 *oxygen overpressure*—partial pressures of oxygen higher than that of air at atmospheric pressure.

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

3.1.4 *reactor*—any vessel capable of sustaining pressures and temperatures above ambient, sometimes designated pressure vessel or bomb.

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

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

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

Current edition approved June 10, 2003. Published July 2003. Originally approved in 1992. Last previous edition approved in 2002 as D 5304 – 02a.

[2] *Annual Book of ASTM Standards*, Vol 05.01.

[3] *Annual Book of ASTM Standards*, Vol 05.02.

[4] *Annual Book of ASTM Standards*, Vol 14.03.

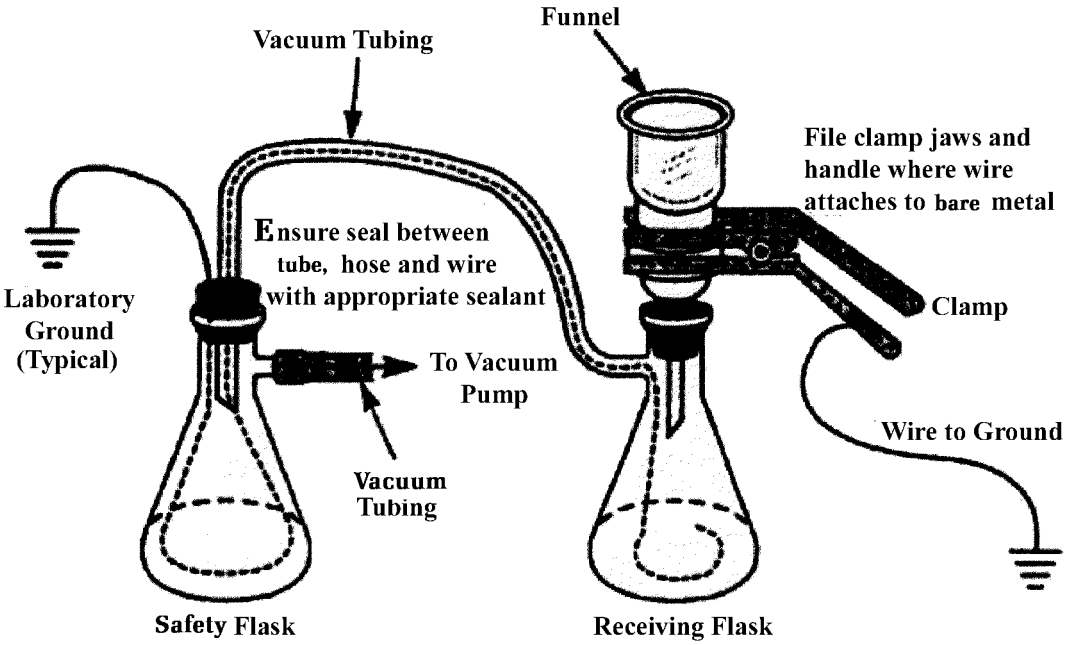

FIG. 1 Schematic of Filtration System

standards with or without stabilizer additives when tested under identical conditions. 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 D 525 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))*[5], designed for safe operating pressures of 800 kPa (100 psig) in oxygen service (**Warning**—See 4.1), equipped with a pressure gage (**Warning**—The pressure for the procedure in this test method is 800 kPa (absolute) (100 psig). Many pressure gages are calibrated in kPa (gage). For such gages, the test pressure would be 700 kPa (gage). Maximum gage gradations should be 20 kPa (5 psig)). The gage should be calibrated against standards, and capable of holding the four sample containers (**Warning**—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 D 525 oxidation bombs 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 ± 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. (**Warning**—Static (non-forced air) ovens and unstirred liquid medium baths, such as the Test Method D 525 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 ± 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 – 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.

[5] Pressure vessels available from the following sources have been found to be satisfactory for use with this test method: Koehler Instrument Company, Inc., 1595 Sycamore Ave., Bohemia, NY 11716-1796; Parr Instrument Company, 211 53rd Street, Moline, IL 61265; and Stanhope-Seta Limited, Park Close, Englefield Green Egham, Surrey TW20 Oxd, England.