Designation: D 5304 – 00

An American National Standard

Standard Test Method for Assessing 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 and are only approximate equivalents.

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 precautionary statements see 4.1, 6.2, 6.3, 7.4 and 10.1.

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

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

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

3.1.4 *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 pre-heated 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 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

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

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[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.

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 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 (or 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 accomodate 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. Heater temperature uniformity must be assured. Heater must be capable of holding the pressure vessel(s) (reactor(s)) described in 6.2 (**Warning**—Static (nonforced 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.).

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 *Filter Funnel*, capable of holding 47 mm diameter filters.

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 to 25 min to evaporate.

6.10 *Thermometer*, should conform to the requirements prescribed in Specification E 1. Thermometer 61C (IP No. 63C) is suitable. Use to monitor the temperature of the heater in 6.3.

[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.

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 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 or precision of the determination.

7.2 *Glass Fiber Filter*[7], 47 mm diameter, with nominal pore size between 0.8 µm and 1.0 µm.

7.3 *Hydrocarbon Solvent*—Hexanes, heptane, iso-octane, or petroleum ether with residue upon evaporation of less than 0.001 % and boiling range between 35 and 100°C are satisfactory. Filter before use with the filter specified in 7.2.

7.4 *Oxygen*—Use 99.5 % minimum oxygen from cylinders with two stage regulators capable of delivering up to 1600 kPa (200 psig). The secondary regulator should be calibrated against standards to deliver 800 kPa ± 10 kPa (100 psig ± 1 psig). (**Warning**—Oxygen at elevated temperature and pressure is capable of causing explosion or fire.)

7.5 *Adherent Insolubles Solvent (TAM)*— An equal volume mixture of toluene, acetone, and methanol (TAM). Filter before use with the filter specified in 7.2.

8. Sampling

8.1 *Field Sampling*—Field sampling should be in accordance with Practices D 4057 or D 4177. Bulk fuel to be sampled must be above its cloud point and thoroughly mixed prior to aliquot sampling. For field sampling and shipping, use only epoxy-lined steel cans that have been cleaned according to Practice D 4306.

8.2 *Laboratory Subsampling*—Fuel to be sampled must be above its cloud point and thoroughly mixed prior to aliquot sampling. Use clean amber or clean borosilicate glass containers for laboratory handling. Fuel in clear bottles must be protected from light, for example, by wrapping in aluminum foil.

9. Preparation of Apparatus

9.1 Rinse the sample containers thoroughly with the TAM solvent followed by water. Then wash with a mildly alkaline or neutral pH laboratory detergent. Rinse with deionized or distilled water. Dry in a drying oven at 110 to 120°C.

9.2 Soak the aluminum weighing dishes in fresh, clean TAM solvent for several minutes followed by drying in a drying oven at 110 to 120°C. Two hours after removal from the oven, firmly nest one dish inside of another for each sample replicate and for each blank replicate to be run. This is both the

[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 *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

[7] Glass fiber filters, 47 mm in diameter, Type A/E, 1 µm nominal pore size, available from Gelman Sciences, Inc., or Type AP40, 0.8 µm nominal pore size, available from Millipore Corp., have been found to be satisfactory for use with this test method.