Designation: D 6468 – 99 (Reapproved 2004)

An American National Standard

Standard Test Method for High Temperature Stability of Distillate Fuels [1]

This standard is issued under the fixed designation D 6468; 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.

1. Scope

1.1 This test method covers relative stability of middle distillate fuels under high temperature aging conditions with limited air exposure. This test method is suitable for all No. 1 and No. 2 grades in Specifications D 396, D 975, D 2880, and D 3699 and for grades DMX and DMA in Specification D 2069. It is also suitable for similar fuels meeting other specifications.

1.2 This test method is not suitable for fuels whose flash point, as determined by Test Methods D 56, D 93, or D 3828, is less than 38°C. This test method is not suitable for fuels containing residual oil.

1.3 The values stated in SI units are to be regarded as the standard.

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]

D 56 Test Method for Flash Point by Tag Closed Tester

D 93 Test Methods for Flash Point by Pensky-Martens Closed Cup Tester

D 396 Specification for Fuel Oils

D 975 Specification for Diesel Fuel Oils

D 1500 Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)

D 2069 Specification for Marine Fuels

D 2274 Test Method for Oxidation Stability of Distillate Fuel Oil (Accelerated Method)

D 2880 Specification for Gas Turbine Fuel Oils

D 3699 Specification for Kerosine

D 3828 Test Methods for Flash Point by Small Scale Closed Tester

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D 4625 Test Method for Distillate Fuel Storage Stability at 43°C

D 5452 Test Method for Particulate Contamination in Aviation Fuels by Laboratory Filtration

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *adherent insolubles*—material that is produced in the course of stressing distillate fuel and that adheres to the glassware after fuel has been flushed from the system.

3.1.2 *filterable insolubles*—material that is produced in the course of stressing distillate fuel and that is capable of being removed from the fuel by filtration.

3.1.3 *inherent stability*—the resistance to change when exposed to air, but in the absence of other environmental factors such as water, reactive metal surfaces, and dirt.

3.1.4 *storage stability*—the resistance of fuel to formation of degradation products when stored at ambient temperatures.

3.1.5 *thermal stability*—the resistance of fuel to formation of degradation products when thermally stressed.

4. Summary of Test Method

4.1 Two 50-mL volumes of filtered middle distillate fuel are aged for 90 or 180 min at 150°C in open tubes with air exposure. After aging and cooling, the fuel samples are filtered and the average amount of filterable insolubles is estimated by measuring the light reflectance of the filter pads. The 100 and 0 % extremes of the reflectance rating range are defined by an unused filter pad and a commercial black standard, respectively.

5. Significance and Use [3]

5.1 This test method provides an indication of thermal oxidative stability of distillate fuels when heated to high temperatures that simulate those that may occur in some types of recirculating engine or burner fuel delivery systems. Results have not been substantially correlated to engine or burner operation. The test method can be useful for investigation of operational problems related to fuel thermal stability.

[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.

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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] Henry, C. P., "The du Pont F21 149°C (300°F) Accelerated Stability Test," *Distillate Fuel Stability and Cleanliness, ASTM STP 751*, L. L. Stavinoha and C. P. Henry, Eds., ASTM, 1981, pp. 22-33.

5.2 When the test method is used to monitor manufacture or storage of fuels, changes in filter rating values can indicate a relative change in inherent stability. Storage stability predictions are more reliable when correlated to longer-term storage tests, for example, Test Method D 4625, or other lower temperature, long-term tests. When fuel samples are freshly produced, aging for 180 min, instead of the traditional 90-min interval, tends to give a result correlating more satisfactorily with the above methods (see Appendix X2).

5.3 The test method uses a filter paper with a nominal porosity of 11 µm, which will not capture all of the sediment formed during aging but allows differentiation over a broad range. Reflectance ratings are also affected by the color of filterable insolubles, which may not correlate to the mass of the material filtered from the aged fuel sample. Therefore, no quantitative relationship exists between the pad rating and the gravimetric mass of filterable insolubles.

6. Apparatus

6.1 *Aging Tubes*, 25× 200 mm, heavy wall test tubes made of borosilicate glass.

6.2 *Heating Bath*, with liquid heating medium, thermostatically controlled to maintain the sample in the aging tube within 1.5°C of 150°C. It must be large enough to hold aging tubes immersed in the heating liquid to a depth above the level of samples in the tubes. The bath and its location shall be such to enable shielding of the samples from direct light during aging. The volume of bath and its heat recovery rate shall be such that the temperature of the medium does not drop more than 5°C when the maximum number of aging tubes are inserted, and recovery to 150°C shall not require more than 15 min. (**Warning**—The flash point of the liquid heating medium must be at least 180°C. Bath vapors and oil sample vapors shall be properly vented. Exposed hot surfaces on the apparatus and hot heating medium can cause severe burns.)

6.3 *Thermometer*, either glass or digital, whose accuracy in the 140 to 160°C range is certified or traceable to a certified thermometer. Use to monitor the temperature of the heating bath in 6.2.

6.4 *Membrane Filter Holder*, to fit 47-mm membrane filters, fitted to a heavy-walled 500-mL or 1-L vacuum flask.

NOTE 1—Several types of membrane filter holders are available. To reduce electrostatic hazards, an all metal filter holder equipped with grounding cables is recommended.[4] Such an apparatus and correct grounding practices are described in Test Method D 5452. A fritted glass filter holder is less preferred because of a tendency to become partially clogged during use so that filter pads that do not have uniform deposits are obtained. Glass filter holders that use a 75-µm (200-mesh) screen to support the filter are available; however, since the screen can be an unbonded electrostatic charge collector, these are not recommended for use with flammable liquids.

6.5 *Vacuum Source*, that limits the maximum vacuum to 27 kPa (200 mm Hg) below atmospheric pressure. The vacuum should rise to 27 kPa within 10 to 15 s after the sample is added to the filtration funnel.

NOTE 2—Use of reduced vacuum improves retention of particulate on the relatively porous filter media.

6.6 *Reflection meter*, Photovolt Model 577 Digital Reflection Meter, complete with search unit *Y* with a green filter and polished black glass standard.[5]

NOTE 3—Other reflection meters or search units, or both, can be used, but they are likely to provide only similar (not identical) results. For example, Photovolt Model 577 digital reflection meter equipped with search unit *W* usually gives somewhat lower percent reflectance values. Correlation of these values is discussed in Appendix X1.

NOTE 4—Older reflection meters, including but not limited to Photovolt Model 670 analog meter, are satisfactory.

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 of the determination.

7.2 *Acetone*, reagent grade. (**Warning**—Extremely flammable.)

7.3 *Adherent Insolubles Solvent (Trisolvent or TAM)*, a mixture of equal parts by volume of reagent grade toluene (**Warning**—Flammable. Vapor harmful.), acetone (**Warning**—see 7.2), and methanol (**Warning**—Flammable. Vapor Harmful. May be fatal or cause blindness if swallowed or inhaled. Cannot be made nonpoisonous.).

7.4 *Hydrocarbon Solvent*, 2,2,4-trimethylpentane (*iso*-octane), 99.75 % purity minimum (**Warning**—see 7.2).

NOTE 5—Heptane is a satisfactory alternative hydrocarbon solvent. However, small differences may be seen due to slightly different solubility characteristics. *Iso*-octane is specified to be in agreement with the hydrocarbon solvent used in other middle distillate stability test methods such as Test Methods D 2274 and D 4625.

7.5 *Filter Paper (Filter Pad)*, Whatman No. 1, 47-mm diameter, or equivalent.

NOTE 6—Filter papers of 42.5 or 55-mm diameter are technically satisfactory. Filters with a diameter of 47 mm permit a small unused margin for identifying the sample and fit all filtration apparatuses.

[4] The sole source of supply of the apparatus known to the committee at this time is a suitable filter holder available from Millipore Corporation, 80 Ashby Rd., Bedford, MA 01730; Catalog No. XX20 047 20. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee [1], which you may attend.

[5] The sole source of supply of the apparatus known to the committee at this time is available from UMM Electronics Inc., Photovolt Instruments, 6911 Hillsdale Court, Indianapolis, IN 46250-2062. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee [1], which you may attend.

[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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.