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An American National Standard

Standard Test Method for High Temperature Stability of Middle 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 D2069~~. 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.

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1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3.1 *Exception*—The maximum vacuum includes inch-pound units in 6.5 and 11.2.

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 Cup 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) ~~D2069Specification 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 Cup Tester
D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D 4625 Test Method for Middle Distillate Fuel Storage Stability at 43C (110F)
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.

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[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.

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

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.

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.~~ Bath Thermometer, either glass or digital measuring temperature measuring device, whose accuracy in the 140 to 160°C range is certified or traceable to a certified thermometer.

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. ~~Note4—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

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[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.~~, Stavinoha, L. L. and Henry, C. P., Eds., ASTM International, 1981, pp. 22-33.

[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, 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, which you may attend.

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~~4—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~~ 5—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.

8. Sampling

8.1 When samples of a fuel batch are obtained to determine stability, obtain samples in accordance with Practice D 4057. Use only epoxy-lined cans or borosilicate glass bottles. Shield clear glass bottles from sunlight to prevent photochemical reactions.

8.2 When samples are from a fuel or component rundown line, exercise care to ensure that the sampling line and valving are thoroughly flushed with current mainstream sample.

8.3 Because stability of some fuels, as determined in this test method, changes over time, the sampling date shall be recorded; record time and date if sample is from a fuel or component rundown line. Samples should be stored at temperatures below 5°C. If storage for more than a few days is expected, oxygen should be removed from the fuel by subsurface purging with a stream of nitrogen; for example, by bubbling nitrogen for 1 min/L of sample.

8.3.1 ~~After taking the sample out of the cold storage, let it warm to ambient temperature before the analysis to prevent water condensation.~~ If samples are taken out of cold storage, warm them to ambient temperature and thoroughly mix prior to aliquot sampling.

NOTE~~7—If~~ 6—If multiple analyses are to be performed on a sample, it is not a good practice to warm the whole sample repeatedly for this purpose. One way of doing this would be to pour an aliquot of the cold sample into a graduated cylinder, cap, allow to warm, and then dispense into the aging tube for analysis. If the cloud point of the fuel is above 5ºC, warm to a temperature 5ºC higher than the cloud point before dispensing.

9. Preparation of Apparatus

9.1 *Cleaning Aging Tubes*~~—Clean new tubes using adherent insolubles solvent, then with a mildly alkaline or neutral laboratory detergent, followed by copious rinsing with deionized or distilled water to remove all traces of detergent. Then rinse with acetone and air dry. Rinse used tubes with adherent insolubles solvent, dry, then clean as above for new tubes. Visually inspect tubes before use, and reclean or reject if there is the slightest trace of contamination.~~

~~9.1.1Because of the small sample size and the high surface to volume ratio in this test method, carefully avoid carryover from past tests or from cleaning agents. There are especially strong effects from traces of copper, strong acids, and strong bases.~~ —Clean new tubes using adherent insolubles solvent, then with a mildly alkaline or neutral laboratory detergent, followed by copious rinsing with deionized or distilled water to remove all traces of detergent. Then rinse with acetone and air dry. Rinse used tubes with trisolvent, dry, then clean as above for new tubes. Visually inspect tubes before use, and reclean or reject if there is the slightest trace of contamination.

9.1.1 Because of the small sample size and the high surface to volume ratio in this test method, carefully avoid carryover from past tests or from cleaning agents. There are especially strong effects from traces of copper, strong acids, and strong bases.

NOTE 7—Clean test tubes carefully to avoid carryover from past tests or from cleaning agents. As a result of the small sample size evaluated and the high surface to volume ratio inherent to the test method, the results obtained can be greatly influenced by the presence of trace contaminants such as copper, strong acids, or strong bases.

9.2 *Cleaning Membrane Filter Holder* ~~—Rinse with adherent insolubles solvent, acetone, and air dry.~~ —Rinse with trisolvent, then with acetone, and air dry.

10. Calibration and Standardization

10.1 Turn on the reflection meter and allow at least 30 min for warm-up. ~~The~~ Set the gain ~~should be set in~~ to LO position for search unit *Y* with green filter.

[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.