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An American National Standard

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Standard Test Method for Distillate Fuel Storage Stability at 43°C (110°F)[1]

This standard is issued under the fixed designation D 4625; 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 test method was adopted as a joint ASTM/IP standard in 1986.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers a method for evaluating the inherent storage stability of distillate fuels having flash points above 38°C (100°F) and 90 % distilled points below 340°C (644°F).

NOTE 1—ASTM specification fuels falling within the scope of this test method are Specification D 396 grade Nos. 1 and 2, Specification D 975 grades 1-D and 2-D, and Specification D 2880 grades 1-GT and 2-GT.

1.2 This test method is not suitable for quality control testing but, rather it is intended for research use to shorten storage time relative to that required at ambient storage temperatures.

1.3 Appendix X1 presents additional information about storage stability and the correlation of Test Method D 4625 results with sediment formation in actual field storage.

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.* For specific hazard information, see Notes 2-6.

2. Referenced Documents

2.1 *ASTM Standards:*

D 381 Test Method for Existent Gum in Fuels by Jet Evaporation[2]

D 396 Specification for Fuel Oils[2]

D 975 Specification for Diesel Fuel Oils[2]

D 2880 Specification for Gas Turbine Fuel Oils[2]

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

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *adherent insolubles, n*—gums formed during storage which remain tightly attached to the walls of the vessel.

3.1.2 *filterable insolubles, n*—solids formed during storage which can be removed from the fuel by filtration.

3.1.3 *inherent storage stability, n—of mid-distillate fuel*—the resistance to change in storage in contact with air, but in the absence of other environmental factors such as water, or reactive metallic surfaces and dirt.

3.1.4 *total insolubles, n*—sum of the filterable insolubles plus the adherent insolubles.

4. Summary of Test Method

4.1 Four-hundred millilitre volumes of filtered fuel are aged by storage in borosilicate glass containers at 43°C (110°F) for periods of 0, 4, 8, 12, 18, and 24 weeks. After aging for a selected time period, a sample is removed from storage, cooled to room temperature, and analyzed for filterable insolubles and for adherent insolubles.

5. Significance and Use

5.1 Fuel oxidation and other degradative reactions leading to formation of sediment (and color) are mildly accelerated by the test conditions, compared to typical storage conditions. Test results have been shown to predict storage stability more reliably than other more accelerated tests. See Appendix X1 for information on the correlation of test results with actual field storage.

5.2 Because the storage periods are long (4 to 24 weeks), the test method is not suitable for quality control testing, but does provide a tool for research on storage properties of fuels.

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

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

5.3 Because environmental effects and the materials and nature of tank construction affect storage stability, the results obtained by this test are not necessarily the same as those obtained during storage in a specific field storage situation.

6. Apparatus

6.1 *Sample Containers*, borosilicate glass bottles. The containers must have a lid or cover, preferably with a polytetrafluoroethylene (PTFE) insert and a hole for a borosilicate glass vent. The total capacity of the containers is 500 mL.

6.2 *Storage Oven*, large enough to contain all of the sample bottles. The oven shall be thermostatically controlled to maintain a temperature of 43 ± 1°C (110 ± 2°F). It shall be as dark as possible to prevent degradation due to photolytic reactions and shall also be *explosion proof*.

6.3 *Drying Oven*, maintained at 99 ± 1°C (210 ± 2°F).

6.4 *Gooch Crucible*, porcelain, No. 4 and Walter crucible holder.

6.5 *Filter Flask* assembly, as shown in Fig. 1.

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.[4] 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 *Glass Fiber Filter*, 2.4-cm circle, 1.5-µm nominal pore size.[5]

7.3 *Hydrocarbon Solvent, iso*—octane, ASTM knock test reference fuel grade, prefiltered through two glass-fiber filters.

NOTE 2—**Warning:** Extremely flammable. Harmful if inhaled. Vapors may cause flash fire.

7.4 *Adherent Insolubles Solvent* (Note 3)—Mix equal parts of reagent grade acetone (Note 4), methanol (Note 5), and toluene (Note 6).

NOTE 3—**Warning:** Extremely flammable. Vapors harmful. May cause flash fire.

NOTE 4—**Warning:** Extremely flammable. Vapors may cause flash fire.

NOTE 5—**Warning:** Flammable. Vapor harmful. May be fatal or cause blindness if swallowed or inhaled. Cannot be made nonpoisonous.

NOTE 6—**Warning:** Flammable. Vapor harmful.

8. Sampling Procedure

8.1 Samples for testing shall be obtained by an appropriate method outlined in Test Method D 4057. Sample containers should be 1 gal (3.78 L) or larger, epoxy-lined cans. Fill sample cans almost to the top to avoid a significant air space. Purge the void space with nitrogen. Store the samples at reduced temperature, − 7 to 4°C (20 to 40°F), prior to use, where possible.

9. Preparation and Apparatus

9.1 *Sample Storage Bottles*—Scrub each bottle with a detergent solution and rinse it with water. Soak the bottle overnight in a mildly alkaline laboratory glassware cleaning solution. Rinse the bottle with tap-water, then invert it and flush it with a stream of distilled water. Allow the bottles to dry and rinse the bottles with 50 mL of the fuel sample. Vent the bottles during storage, using a glass tube bent in an upside down "U," (see Fig. 2), to prevent contamination of the sample from airborne particulates. Insert the glass tube through a cover, preferably equipped with a polytetrafluoroethylene (PTFE) insert (see Fig. 2).

9.2 *Filter Preparation*—Insert two glass-fiber filter disks into the clean Gooch crucible (Note 7). Wash the filters by pouring 200 mL of knock-grade *iso*octane through the Gooch crucible. After removing the excess solvent with suction, dry the crucible and filters for 1 h in an oven maintained at 99 ± 1°C (210 ± 2°F).

9.3 *Cooling and Weighing*—After drying, place the crucible and filters in a desiccator without desiccant for at least 30 min to protect the crucible and filters from airborne particulates and to allow the crucible to cool in an environment similar to the surrounding environment and thereby prevent possible errors due to sudden absorption of moisture from the atmosphere when the crucible is removed from the desiccator. Use the weight of the *moisture blank* crucible to correct for atmospheric moisture. Weigh the crucibles with the filters to the nearest 0.1 mg and retain for sample analysis. This must be

[4] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., 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.

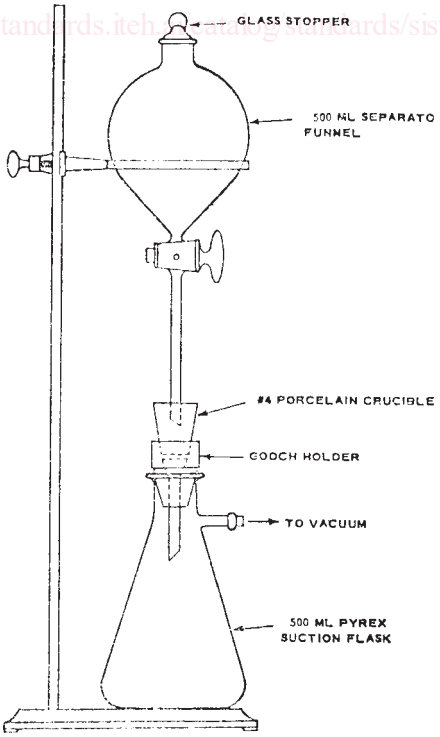

FIG. 1 Self-Feeding Filtering Assembly

[5] The glass fiber filter paper available from Whatman as Catalog No. 1827 024 (934-AH), has been found satisfactory for this purpose.