



Designation: D 4625 – 03

An American National Standard



Designation: 378/87

Standard Test Method for Middle 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 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.*

2. Referenced Documents

2.1 ASTM Standards:²

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

D 396 Specification for Fuel Oils

D 975 Specification for Diesel Fuel Oils

D 2880 Specification for Gas Turbine Fuel Oils

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products

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 middle 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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This test method was adopted as a joint ASTM/IP standard in 1986.

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.

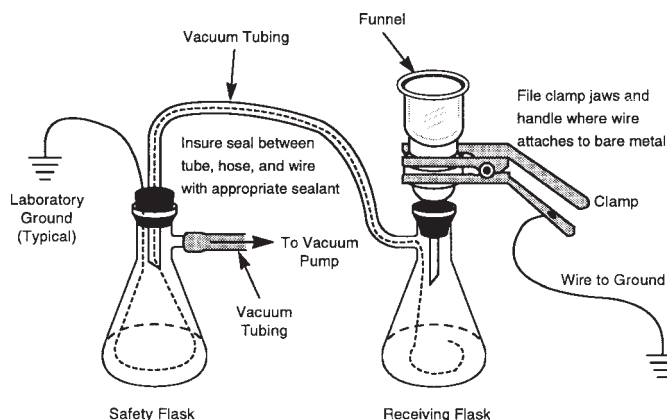


FIG. 1 Schematic of Filtration System

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 $43 \pm 1^\circ\text{C}$ ($110 \pm 2^\circ\text{F}$). It shall be as dark as possible to prevent degradation due to photolytic reactions and shall also be *explosion proof*.

6.3 *Filter Drying Oven*, shall be capable of safely evaporating the solvent at $90 \pm 5^\circ\text{C}$ for the drying of filter materials.

6.4 *Filtration System*—Arrange the following components as shown in Fig. 1.

6.4.1 *Funnel and Funnel Base*, with filter support for a 47-mm diameter membrane and a locking ring or spring action clip.

6.4.2 *Ground/Bond Wire*, 0.912–2.59 mm (No. 10–No. 19) bare-stranded, flexible stainless steel or copper installed in the flasks and grounded as shown in Fig. 1.

6.4.3 *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.

6.4.4 *Safety Flask*, 1.5 L or larger borosilicate glass vacuum filter flask equipped with a sidearm to connect the vacuum system. A fuel and solvent resistance rubber hose, through which the grounding wire passes, shall connect the sidearm of the receiving flask to the tube passing through the rubber stopper in the top of the safety flask.

6.4.5 *Vacuum System*, either a water-aspirated or a mechanical vacuum pump may be used if capable of producing a vacuum of 1 to 100 kPa below atmospheric pressure when measured at the receiving flask.

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.³ 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 *Nylon Test and Control Membrane Filters*—plain, 47-mm diameter, nominal pore size 0.8- μm . (Membrane filters with a grid imprinted on their surface may be used as control membrane filters for identification.)

7.3 *Hydrocarbon Solvent, 2,2,4-trimethylpentane (isooctane)*—ASTM knock test reference fuel grade, prefiltered through two glass-fiber filters. (**Warning**—Extremely flammable. Harmful if inhaled. Vapors may cause flash fire.)

7.4 *Adherent Insolubles Solvent* (**Warning**—Extremely flammable. Vapors harmful. May cause flash fire)—Mix equal volumes of reagent grade acetone (**Warning**—Extremely flammable. Vapors may cause flash fire), methyl alcohol (**Warning**—Flammable. Vapor harmful. May be fatal or cause blindness if swallowed or inhaled. Cannot be made nonpoisonous), and toluene (**Warning**—Flammable. Vapor harmful.).

8. Sampling Procedure

8.1 Samples for testing shall be obtained by an appropriate method outlined in Practice 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 of Apparatus and Sample Bottles

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

³ *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 *Annual 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.