



Designation: D 4625 – 04

An American National Standard



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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), by Test Methods D 93, and 90 % distilled points below 340°C (644°F), by Test Method D 86.

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 86 Test Method for Distillation of Petroleum Products at Atmospheric Pressure

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

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.

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

D 396 Specification for Fuel Oils

D 975 Specification for Diesel Fuel Oils

D 1193 Specification for Reagent Water

D 2880 Specification for Gas Turbine Fuel Oils

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D 4177 Practice for Automatic 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 that remain tightly attached to the walls of the vessel.

3.1.2 *filterable insolubles, n*—solids formed during storage that 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 with typical storage conditions. Test results have been shown to predict storage stability more

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

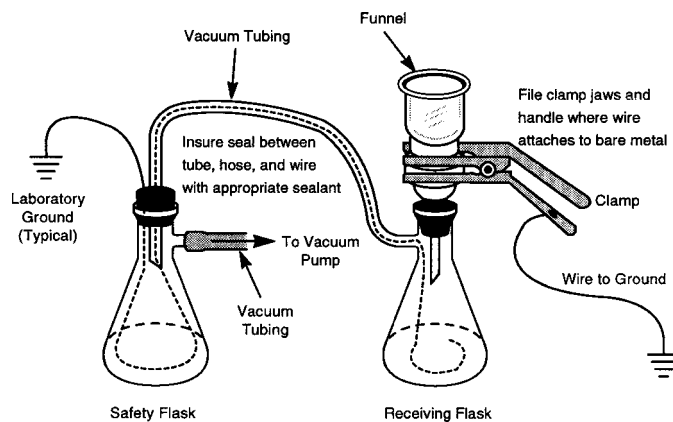


FIG. 1 Schematic of Filtration System

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.

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 up 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.).

7.5 *Purity of Water*—Unless otherwise indicated, references to water mean reagent water as defined by Type III of Specification [D 1193](#).

7.6 *Liquid or Powder Detergent*, water-soluble, for cleaning glassware.

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.

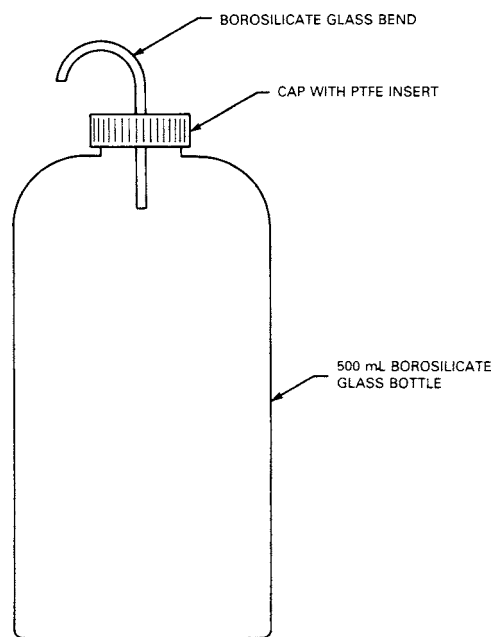


FIG. 2 Sample Storage Container

8. Sampling Procedure

8.1 Samples for testing shall be obtained by an appropriate method outlined in Practice D 4057 or D 4177. 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 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 Clean all components of the filtration apparatus as described in 9.2.1-9.2.7.

9.2.1 Remove any labels, tags, and so forth.

9.2.2 Wash with warm tap water containing detergent.

9.2.3 Rinse thoroughly with warm tap water.

9.2.4 Rinse thoroughly with deionized water. Container caps should be handled only externally with clean laboratory crucible tongs during this and subsequent washings.

9.2.5 Rinse thoroughly with propan-2-ol that has been filtered through a 0.45- μ m membrane filter.

9.2.6 Rinse thoroughly with filtered flushing fluid and dry.

9.2.7 Keep a clean protective cover (the cover may be rinsed with filtered flushing fluid) over the top of the sample container until the cap is installed. Similarly, protect the funnel opening

of the assembled filtration apparatus with a clean protective cover until ready for use.

9.3 Preparation of Membrane Filters:

9.3.1 Each set of test filters consists of one test membrane filter and one control membrane filter. For fuels containing little particulate materials, only one set of filters is required. If the fuel is highly contaminated, more than one set of filters may be required. The two membrane filters used for each individual test shall be identified by marking the petri dishes used to hold and transport the filters. Clean all glassware used in preparation of membrane filters as described in 9.2.

9.3.1.1 Using forceps, place the test and control membrane filters side by side in a clean petri dish. To facilitate handling, the membrane filters should rest on clean glass support rods, or watch glasses, in the petri dish.

9.3.1.2 Place the petri dish, with its lid slightly ajar, in a drying oven at $90 \pm 5^\circ\text{C}$ and leave it for 30 min.

9.3.1.3 Remove the petri dish from the drying oven, and place it near the balance. Keep the petri dish cover ajar, but keep it such that the membrane filters are still protected from contamination from the atmosphere. Allow 30 min for the membrane filters to come to equilibrium with room air temperature and humidity.

9.3.1.4 Remove the control membrane filter from the petri dish with forceps, handling by the edge only, and place it centrally on the weighing pan of the balance. Weigh it, record the initial mass to the nearest 0.0001 g, and return it to the petri dish.

9.3.1.5 Repeat 9.3.1.4 for the test membrane filter.

9.3.1.6 Using clean forceps, place the weighed control membrane filter centrally on the membrane filter support of the filtration apparatus (see Fig. 1). Place the weighed test membrane filter on top of the control membrane filter. Install the funnel and secure with locking ring or spring clip. Do not remove the plastic film from the funnel opening until ready to start filtration.

10. Preparation of Sample

10.1 If the fuel has been stored at reduced temperature, allow the sample to come to ambient temperature. To dissolve any separated wax, be certain that the entire fuel sample is at least 5°C above its cloud point before proceeding.

10.2 Assemble a filtration system, as shown in Fig. 1, to filter the fuel. Clean the receiving flask, separatory funnel, and glass funnel in the same manner as the storage bottles (9.1). Rinse these items with filtered *isooctane* and then air.

11. Procedure

11.1 Sample Aging:

11.1.1 Adjust the storage oven for sample storage to a temperature of $43 \pm 1^\circ\text{C}$ ($110 \pm 2^\circ\text{F}$).

11.1.2 Place 400 mL of filtered fuel into each bottle. Use two bottles for each sampling period. (Commonly used sampling periods are 0, or any of the following: 4, 8, 12, and 24 weeks). Fill three extra bottles with fuel to be used in case of accidents, for further tests at other times of storage, or to extend the overall test duration.

11.1.3 Label each storage bottle with the time and date the test is started, sample identification, and the time and date