



Designation: D4625 – 21

## Standard Test Method for Middle Distillate Fuel Storage Stability at 43 °C (110 °F)<sup>1</sup>

This standard is issued under the fixed designation D4625; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the U.S. 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 D93, and 90 % distilled points below 340 °C (644 °F), by Test Method D86.

NOTE 1—ASTM specification fuels falling within the scope of this test method are Specification D396, Grade Nos. 1 and 2; Specification D975, Grades 1-D and 2-D; and Specification D2880, 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 D4625 results with sediment formation in actual field storage.

1.4 The values given in SI units are to be regarded as the standard.

1.4.1 *Exception*—The values in parentheses are for information only.

1.5 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.14 on Stability, Cleanliness and Compatibility of Liquid Fuels.

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### 2. Referenced Documents

2.1 *ASTM Standards*:<sup>2</sup>

D86 Test Method for Distillation of Petroleum Products and Liquid Fuels at Atmospheric Pressure

D93 Test Methods for Flash Point by Pensky-Martens Closed Cup Tester

D381 Test Method for Gum Content in Fuels by Jet Evaporation

D396 Specification for Fuel Oils

D975 Specification for Diesel Fuel

D1193 Specification for Reagent Water

D2880 Specification for Gas Turbine Fuel Oils

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

### 3. Terminology

3.1 *Definitions*:

3.1.1 For definitions of terms used in this test method, refer to Terminology D4175.

3.2 *Definitions of Terms Specific to This Standard*:

3.2.1 *adherent insolubles, n*—gums formed during storage that remain tightly attached to the walls of the vessel after fuel has been flushed from the container.

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

3.2.3 *inherent storage stability, n*—of middle distillate fuel—the resistance of the fuel to change during storage in contact with air, but in the absence of other environmental factors such as water, or reactive metals and dirt.

<sup>2</sup> 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

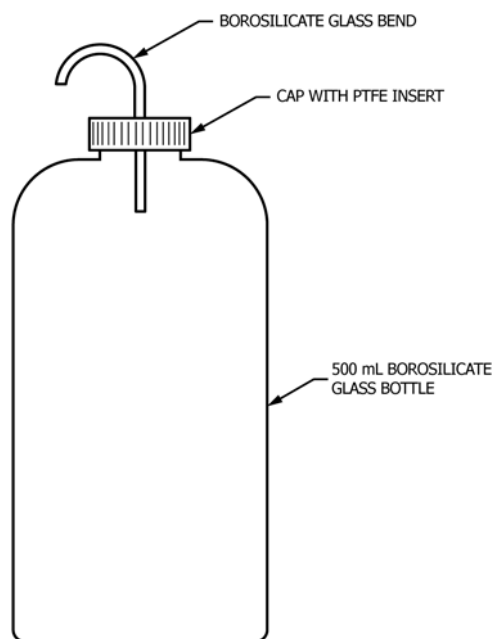


FIG. 1 Sample Storage Container

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

#### 4. Summary of Test Method

4.1 Four-hundred (400) mL volumes of filtered fuel are aged by storage in borosilicate glass containers at 43 °C (110 °F) for periods of 4, 8, 12, 18, and 24 weeks. If desired, perform zero-week analyses on the same day as the other samples are placed in storage. Zero-week data are used to provide base data and ensure satisfactory technique. 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 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 weeks 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, nominal capacity 500 mL ([Fig. 1](#)). The containers shall have a cap, lid,

or cover, preferably with a polytetrafluoroethylene (PTFE) insert and a hole for a borosilicate glass vent.

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 °C ± 1 °C (110 °F ± 2 °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 °C ± 5 °C for the drying of filters.

6.4 *Filtration System*—Arrange the following components as shown in [Fig. 2](#).

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 mm to 2.59 mm (No. 10 to No. 19) bare-stranded, flexible stainless steel or copper installed in the flasks and grounded as shown in [Fig. 2](#).

6.4.3 *Receiving Flask*, 1.5 L, or larger, borosilicate glass vacuum filter flask, into which the filtration apparatus fits, 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 resistant 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 80 kPa 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.<sup>3</sup> 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 or equivalent, prefiltered through two glass-fiber or nylon membrane filters, nominal pore size 0.8 μm. (**Warning**—Extremely flammable. Harmful if inhaled. Vapors may cause flash fire.)

<sup>3</sup> *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, 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.

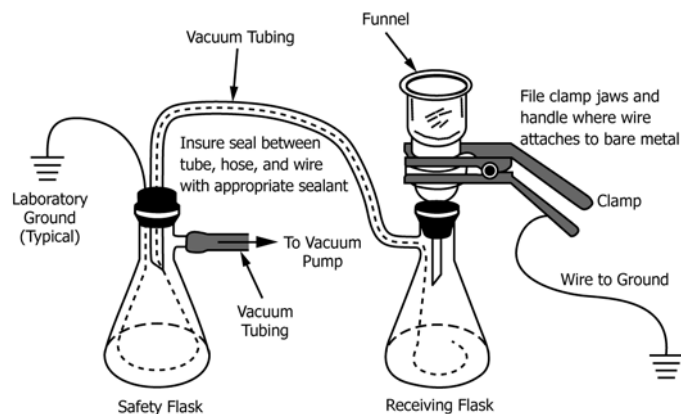


FIG. 2 Schematic of Filtration System

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 D1193.

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

## 8. Sampling Procedure

8.1 Samples for testing shall be obtained by an appropriate method outlined in Practice D4057 or D4177. 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,  $-7\text{ }^{\circ}\text{C}$  to  $4\text{ }^{\circ}\text{C}$  ( $20\text{ }^{\circ}\text{F}$  to  $40\text{ }^{\circ}\text{F}$ ), prior to use, where possible.

## 9. Preparation of Apparatus and Sample Bottles

9.1 *Sample Storage Bottles*—Scrub each bottle and cap with a detergent solution and rinse it with water. Soak the bottle and cap overnight in an alkaline laboratory glassware cleaning solution. Rinse the bottle and cap with tap water, then invert them and flush them with a stream of distilled water. Allow the bottles and caps to dry. Prior to introducing the sample, 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. 1), 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. 1).

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.

9.2.5 Rinse thoroughly with propan-2-ol that has been filtered through a  $0.45\text{ }\mu\text{m}$  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  $90\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$  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 0.1 mg, 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. 2). 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 The test fuel shall be filtered prior to placing it in storage. Assemble a filtration system, as shown in Fig. 2, to filter the fuel. Use a single membrane filter for this filtration step. This membrane filter need not be preweighed.

10.3 Using this filtration assembly, filter sufficient fuel to put 400 mL of fuel in each storage bottle. For a typical test, this is 4 L of fuel. It is prudent to filter a small amount of extra fuel. It may be necessary to replace the filter membrane (7.2) throughout this step, depending on the cleanliness of the test fuel.

## 11. Procedure

### 11.1 Sample Aging:

11.1.1 Adjust the storage oven for sample storage to a temperature of 43 °C ± 1 °C (110 °F ± 2 °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 4, 8, 12, 18, and 24 weeks). Extra bottles may be placed in storage to be used in case of accidents, for further tests at other times of storage, or to extend the overall test duration.

11.1.2.1 Some operators have found it useful to conduct a zero-week test as a demonstration of proper technique. Since the fuel is prefiltered, zero-week results should be nil. A zero-week analysis is not a mandatory part of the analysis.

11.1.3 Label each storage bottle with the time and date the test is started, sample identification, and the time and date when the bottle is to be removed from storage. Place the bottles in the oven in random order as a means to reduce the possible effects of hot zones in the oven.

### 11.2 Determination of Filterable Insolubles:

11.2.1 At the end of each prescribed period of time, remove two bottles from the storage oven and allow them to cool to 21 °C to 27 °C (70 °F to 80 °F) in a dark environment. This may take from 4 h to 24 h.

11.2.2 Assemble the filtration apparatus with a preweighed sample and control filter. After the fuel has cooled, pour fuel from the sample container to the graduated cylinder, start the vacuum, and then transfer 100 mL of fuel to the filter funnel.

11.2.2.1 Continue transferring 100 mL increments of fuel to the filter funnel. When all the fuel from the sample container has been filtered, or if filtration slows so that 100 mL of sample requires greater than 10 min for complete filtration, then remove the filter support/filter funnel from the receiving flask, pour the filtered fuel into a clean graduated cylinder, and record the volume of fuel that was filtered in millilitres. Keep the fuel sample filtrate separate from the solvent washings filtrate. This allows the fuel to be used for additional analyses (if desired). If all the fuel has been filtered, thoroughly rinse the sample

container and the graduated cylinder with one or more portions of filtered flushing fluid, pour the rinses into the funnel, and proceed to 11.2.2.2. If all the fuel has not been filtered, proceed to 11.2.2.2 and 11.2.2.3, and then repeat from 11.2.2.1.

11.2.2.2 Wash down the inside of the funnel and the outside of the joint between the funnel and filter base filtered with flushing fluid. With the vacuum applied, carefully separate the funnel from the filter base. Wash the periphery of the membrane filter by directing a gentle stream of filtered flushing fluid from the edge to the center, exercising care not to wash any of the particulate from the surface of the membrane filter. Maintain vacuum after the final washing for 10 s to 15 s to remove excess filtered flushing fluid from the membrane filter.

11.2.2.3 Using clean forceps, carefully remove the test and control membrane filters from the filter base and place them side by side on clean glass support rods or watch glasses in a clean, covered petri dish. Dry and reweigh the membrane filters as described in 9.3, taking care not to disturb the particulate on the surface of the test membrane filter. Record the final control membrane filter mass and the final test membrane filter mass to the nearest 0.1 mg for each filtration.

NOTE 2—Do not mix rinsings and filtrate if filtrate color changes are being measured.

NOTE 3—If severe filter plugging is encountered so that filtration is not complete in 3 h, discontinue the test with the notation that filter plugging occurred.

11.2.3 Repeat the procedure in 11.2.2 – 11.2.2.3 for the second bottle.

11.3 *Determination of Adherent Insolubles on Sample Bottle*—After the final washing with the hydrocarbon solvent, dissolve any adherent gum on the sample container walls with two washings of 30 mL to 35 mL of the adherent insolubles solvent. Pour each washing into previously weighed 100 mL beakers. Evaporate the solvent at 160 °C (320 °F) by the air jet method in accordance with Test Method D381. After the solvent is completely evaporated, place the beakers in a desiccator without desiccant and allow to cool to room temperature. Weigh the beakers to the nearest 0.1 mg. Use a tare beaker (moisture blank) in accordance with Test Method D381.

## 12. Calculation

12.1 Calculate the total insolubles ( $T_i$ ) in milligrams per 100 mL of fuel after aging, as follows:

$$T_i = \frac{(F_i + A_i)}{4} \quad (1)$$

where:

$T_i$  = total insolubles, mg/100 mL,

$F_i$  = mass of filterable insolubles, mg, as determined in 11.2, and

$A_i$  = moisture corrected mass of adherent insolubles, mg, as determined in 11.3.

## 13. Report

13.1 Report  $T_i$ ,  $F_i$ , and  $A_i$  from 12.1 for both bottles to show repeatability of tests at each aging period.