Standard Test Method for
High Temperature Stability of Middle Distillate Fuels[1]

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 D396, D975, D2880, and D3699. 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 D56, D93, or D3828, is less than 38°C. This test method is not suitable for fuels containing residual oil.

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]

D56 Test Method for Flash Point by Tag Closed Cup Tester

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

D396 Specification for Fuel Oils

D975 Specification for Diesel Fuel Oils

D1500 Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)

D2274 Test Method for Oxidation Stability of Distillate Fuel Oil (Accelerated Method)

D2880 Specification for Gas Turbine Fuel Oils

D3699 Specification for Kerosine

D3828 Test Methods for Flash Point by Small Scale Closed Cup Tester

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4625 Test Method for Middle Distillate Fuel Storage Stability at 43°C (110°F)

D5452 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.

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

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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*, Stavinoha, L. L. and Henry, C. P., Eds., ASTM International, 1981, pp. 22–33.

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 D4625, 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 *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 D5452. 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.

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.[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,* (**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 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 D2274 and D4625.

7.5 *Filter Paper (Filter Pad),* Whatman No. 1, 47-mm diameter, or equivalent.

NOTE 5—Filter papers of 42.5 or 55-mm diameter are technically satisfactory. Filters with a diameter of 47 mm permit a small unused

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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 D4057. 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 If samples are taken out of cold storage, warm them to ambient temperature and thoroughly mix prior to aliquot sampling.

Note 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 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 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. Set the gain to LO position for search unit *Y* with green filter.

10.2 Follow the manufacturer's instructions to carry out a two point calibration of the reflection meter, using the black standard and standard plaque supplied with the search unit.

Note 8—The calibration procedure ensures that the instrument is working properly. It also indirectly sets a nominal 100 % reflectance setting that is subsequently reset in 10.3.

10.3 Place a new filter paper on top of a stack of at least ten unused Whatman No. 1 filters of the same size. Place the search unit on the center of the filter, and adjust the meter reading to 100 %, using the sensitivity control.

Note 9—Such adjustment, which sets the test method 100 % reflectance point, alters the reflectance scale; as a result, the reflection meter will no longer read the recited reflectance of the standard plaque.

10.4 Place the search unit on the center of the black glass standard, and adjust the meter reading to 0 %, using the ZERO control. Recheck the 100 % adjustment against the new filter paper, and continue adjustment until the meter reads both 0 % with the black glass standard and 100 % with the new filter pad.

11. Procedure

11.1 Adjust the heating bath to a temperature of 150± 1.5°C (see 6.3).

11.2 Assemble the filter apparatus with a new filter paper. Filter at least 100 mL of the fuel sample through the paper. Use the inline vacuum regulator so that the maximum vacuum is 27 kPa (200 mm Hg). Measure two 50 ± 2-mL volumes of filtered fuel and decant into each of two aging tubes (see Appendix X3).

Note 10—Where practical, filter a fuel sample larger than 100 mL. This will reduce the possibility that the paper may absorb trace materials that affect stability.

11.3 Place the uncapped sample tubes in the heating bath, making sure the level of the fluid in the test tube is below the surface of the oil in the heating bath. Age the fuel samples for 90 ± 3 min, or 180 ± 5 min. Place the tubes in the bath in the same order in which they are to be removed. (**Warning**—Fuels will be heated above their flash points. There is a possibility that certain light fuels such as kerosine will boil under conditions of the test. Both situations raise the risk of fire when an ignition source is present. Fuel samples that may contain gasoline or other volatile components should not be tested.)

Note 11—The severity of the test is increased by aging for longer times at 150°C. The selection of the aging time depends on application and should be established by correlation with other tests or with application requirements.

11.4 Remove the samples from the heating bath and allow to cool gradually in air to 20 to 25°C over a period of 90 min to 4 h. Cool in the dark to prevent photochemical reactions. Do not accelerate cooling by immersing in a cooling bath as this can result in small particle size and a lower pad rating. If samples are allowed to stand more than 4 h before filtering, insoluble gum may adhere to the aging tubes, resulting in erratic data. (**Warning**—The hot samples can cause severe burns. Use protective equipment.)

11.5 Prepare a filtration assembly with a new filter paper, and attach all grounding clips to ground. Filter one of the two aged fuel samples.

11.6 While maintaining vacuum, wash the aging tube with three small portions, about 15 mL each, of *iso*-octane and filter

through the filter paper. Wash the inside of the filter assembly with *iso*-octane, and remove the funnel portion of the assembly. While still maintaining vacuum, use a gentle stream of *iso*-octane from a squeeze bottle to wash the filter clean of any traces of fuel oil, and allow to dry under vacuum for 1 or 2 min (see Note 13). Shut off vacuum, and remove the filter from the assembly with forceps. If the deposit on the filter pad is not evenly distributed, reject the pad and rerun the test after cleaning the membrane filter holder (see 9.2).

NOTE 12—When multiple tests are carried out, the margin of the pad may be used for sample identification.

NOTE 13—Some laboratories may contain sufficient airborne contamination to change results if the air drying period is prolonged.

11.7 If the deposit on the filter pad is not evenly distributed, reject the pad and rerun the test (see 9.2).

11.8 Place the test filter on the stack of at least ten unused filter pads, center the search unit on the filter, and record the meter value as percent reflectance *filter pad rating* to one decimal place.

11.9 Repeat 11.5 – 11.8, using the second of two aged fuel samples.

12. Report

12.1 Report the following information:

12.1.1 Sampling date and date of test (see 8.3).

12.1.2 Aging time at 150°C.

12.1.3 Percent reflectance *filter pad rating* as the average of duplicate determinations, to the nearest whole percent.

13. Precision and Bias[7]

13.1 *Precision*—The precision statement is based on analysis of five D975 S5000 and five samples of D975 S500 fuels in

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[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1463.

15 laboratories. A separate program also verified that properly calibrated reflection meters in different laboratories were in excellent agreement. See the interlaboratory report (RR:D02-1463).[7] The precision of this test method as determined by statistical examination of interlaboratory results is as follows:

13.1.1 *Repeatability*— The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in normal and correct operation of the test method, exceed the following values in only one case in twenty:

Aging Time	Repeatability
90 min	22.42 - 0.2130x
180 min	22.55 - 0.2145x

where:

x = the average of two results in percent reflectance.

13.1.2 *Reproducibility*— The difference between two independent test results, obtained by different operators working in different laboratories on identical test material, would in the long run, in normal and correct operation of the test method, exceed the following values in only one case in twenty:

Aging Time	Reproducibility
90 min	44.04 - 0.4281x
180 min	34.11 - 0.3034x

where:

x = the average of two results in percent reflectance.

13.2 *Bias*—Since there is no accepted reference material for determining the bias for this test method, bias cannot be determined.

14. Keywords

14.1 fuel insolubles; high temperature stability; middle distillate fuels; storage stability; thermal stability

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APPENDIXES

(Nonmandatory Information)

X1. INTERPRETATION OF RESULTS

X1.1 Test results should be correlated to actual storage or use conditions, or equipment operation, to have maximum utility. In the case of storage stability, results should be checked against longer term, less accelerated test methods.

X1.2 For internal, contractual, or industry specification purposes, satisfactory results are partly defined by age of the sample at time of testing, by anticipated storage needs, or by equipment needs; all of these vary and may reflect more or less conservative approaches.

X1.3 Table X1.1 shows an interpretation of test results, which appears to be generally accepted. Rankings are with respect to relative performance in the test; relevance to use applications requires interpretation and judgment.

TABLE X1.1 Interpretation of Results

Relative Stability	Y Search Unit with Green Filter % Reflectance	W Search Unit % Reflectance
	100-90	100-85
	89-80	84-71
⏐	79-70	70-62
Decreasing	69-60	61-53
Stability	59-50	52-32
↓	49-0	31-0

X1.4 The *Y* search unit with green filter has a different spectral response than the *W* search unit, and on average, gives a higher result. Because the sediments on filter pads vary in color, the difference between results varies.

X1.4.1 During the precision program on this test method, some laboratories used the *W* search unit, and a precision statement for its use was developed (see RR:D02-1463[7]). However, because the result and difference between results over the percent reflectance range varies when obtained with the two search units, it was agreed that only one should be specified in the test method, to enable generation of comparable results, especially comparable laboratory-to-laboratory results. Further, the data suggest that the bias between the two search units is not uniform, so a bias correction cannot be recommended. Other reflection meters can be used, but in that case, the use of a different instrument should be reported, and it should also be reported that a modified test method was used. It would also be appropriate to generate a new table, such as Table X1.1, which establishes the relevance of the results.

X1.5 When the fuel is very dark, the filter pad may be stained a dark color, yet microscopic examination shows little or no particulate matter is present. Fuel color does not necessarily correlate with performance; however, dark-colored fuels frequently have oxidation sediment associated with them and may form sediment if commingled with fuels having poor solubilizing properties (for example, when a dark light cycle oil is commingled with a paraffinic fuel). Staining alone will typically not give filter pad ratings with less than 50 % reflectance.

X1.6 The test method can be used to determine the relative performance of additives under conditions of the test. Because results depend on the history of the fuel sample, relative performance is best determined by carrying out comparison tests in a single batch of samples, prepared and aged together.

X1.7 The test temperature may simulate conditions that exist in some equipment where fuel is exposed to temperatures from 100 to 150°C, or higher, then recycled back to a storage tank.

X1.8 This test method is effective to monitor the relative thermal stability and possible change in storage stability at a production site where crude source and process conditions vary over time. Substantial changes can signal a fuel quality excursion. The test method can also be used to verify that stabilizing additives have been appropriately added to distillate fuels with marginal stability.

X2. FACTORS WHICH AFFECT THE TEST METHOD

X2.1 Due to the high test temperature and the lack of oxygen feed to the test specimen during aging, there is a tendency of the test method to give better results with fresh fuel than with the same fuel after it has aged at ambient temperature with oxygen exposure. When tests are carried out on freshly produced fuel, the aging period can be increased to 180 min, or more stringent standards can be imposed on the pad rating to better reflect change in properties that may result from distribution and storage.

X2.1.1 The choice of fuel stressing (90 min or 180 min) is directed by the specification for which the fuel is evaluated or by the specific requirements of the evaluator. Generally, stressing for 180 min is a more stringent evaluation of fuel thermal stability.

X2.2 Additives can have positive or negative effects in this test, which may or may not be observed in actual storage or use.

X2.2.1 The test method tends to give results that amplify the benefits of some stabilizer additives. The benefit of metal deactivator additive, when fuel contains dissolved copper, correlates well with less accelerated tests. Effects of dispersant additives are also well correlated.

X2.2.2 Some additives exhibit effects on samples aged in accordance with this test method, which are not found in other tests where samples are aged at 100°C or lower. For example, many fuels will give worse results if alkyl nitrate cetane number improver is present. The relevance of these findings is not entirely clear.

X3. FUEL COLOR

X3.1 Diesel fuel natural color ranges from water white (colorless) to dark amber, reflecting the crude oil or the refinery process streams from which the fuel is produced, or both. Color per se, as determined by Test Method D1500, does not necessarily reflect suitability of the fuel for its intended use. There is also no agreement on how to measure fuel color when dye is present.

X3.2 Change in fuel color (darkening) alone, during storage at ambient temperatures or upon accelerated aging at elevated temperatures, is not a reliable measure of fuel quality. Nonetheless, some fuel technologists believe that measurement of fuel color before and after aging provides useful information.

X3.3 Practitioners of this test method who insist on measuring Test Method D1500 color before and after aging should measure unaged fuel color (initial color) after filtration in 11.2 and carry out aged fuel color measurement on the filtrate generated in 11.5 that has been decanted from the vacuum flask before use of any *iso*-octane.