Designation: D2274 – 14

Designation: 388/97

Standard Test Method for Oxidation Stability of Distillate Fuel Oil (Accelerated Method)[1]

1. Scope*

1.1 This test method covers the measurement of the inherent stability of middle distillate petroleum fuels under specified oxidizing conditions at 95 °C.

NOTE 1—Fuels used in establishing the precision measures for this test method were described as gas oil, diesel fuel, No. 2 heating oil, and DFM, a Navy distillate fuel suitable for diesels, boilers, and gas turbines. (The term DFM is no longer used when referring to fuel meeting MIL-F-16884 requirements; rather it is called F76 as it conforms to NATO F76 requirements.) While the test method may be used for fuels outside the range of these fuels, the precision measures may not apply.

1.2 This test method is not applicable to fuels containing residual oil. This test method has not been validated for testing biodiesel, such as meeting Specification D6751 or blends of middle distillates and biodiesel, such as meeting Specification D7467, or both. Test Method D7462 has been determined to be suitable for testing B100 and all blends of middle distillates and biodiesel.

NOTE 2—No. 1 and No. 2 grades in Specifications D396 or D975 currently allow up to 5 % biodiesel meeting Specification D6751. Samples containing biodiesel can result in partial dissolution or compromise of the membrane filter and give erroneous results.

1.3 The values given in SI units are to be regarded as the standard. The values in parentheses are for information only.

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]

D381 Test Method for Gum Content in Fuels by Jet Evaporation
D396 Specification for Fuel Oils
D943 Test Method for Oxidation Characteristics of Inhibited Mineral Oils
D975 Specification for Diesel Fuel Oils
D1193 Specification for Reagent Water
D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
D4625 Test Method for Middle Distillate Fuel Storage Stability at 43°C (110°F)
D6751 Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels
D7462 Test Method for Oxidation Stability of Biodiesel (B100) and Blends of Biodiesel with Middle Distillate Petroleum Fuel (Accelerated Method)
D7467 Specification for Diesel Fuel Oil, Biodiesel Blend (B6 to B20)

2.2 *Military Specification:*[3]

MIL-F-16884 Fuel, Navy Distillate

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *adherent insolubles* (formerly adherent gum), *n*—material which is produced in the course of stressing

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[1] This test method is under the jurisdiction of Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.14 on Stability and Cleanliness of Liquid Fuels.

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[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.

[3] Available from Standardization Documents Order Desk, Bldg. 4, 700 Robbins Ave., Philadelphia, PA 19111-5098. Attn: NPODS

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

distillate fuel under the conditions of this test and which adheres to the glassware after fuel has been flushed from the system.

3.1.2 *filterable insolubles, n*—material, which is produced in the course of stressing distillate fuel under the conditions of this test, which is capable of being removed from the fuel by filtration. This includes both material suspended in the fuel and material easily removed from the oxidation cell and oxygen delivery tube with hydrocarbon solvent.

3.1.3 *inherent stability, n*—the resistance to change when the fuel is exposed to air, but in the absence of other environmental factors such as water, or reactive metals and dirt.

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

3.1.5 *zero time, n*—the time the first of a batch of oxidation cells is placed in the heating bath.

3.1.5.1 *Discussion*—This is the time taken as the start of the 16 h of residence in the heating bath.

4. Summary of Test Method

4.1 A 350 mL volume of filtered middle distillate fuel is aged at 95 °C (203 °F) for 16 h while oxygen is bubbled through the sample at a rate of 3 L/h. After aging, the sample is cooled to approximately room temperature before filtering to obtain the filterable insolubles quantity. Adherent insolubles are then removed from the oxidation cell and associated glassware with trisolvent. The trisolvent is evaporated to obtain the quantity of adherent insolubles. The sum of the filterable and adherent insolubles, expressed as milligrams per 100 mL, is reported as total insolubles.

5. Significance and Use

5.1 This test method provides a basis for the estimation of the oxidation stability of middle distillate fuels such as No. 2 fuel oil.

5.2 The test method may not provide a prediction of the quantity of insolubles that will form in field storage over any given period of time. The amount of insolubles formed in such field storage is subject to the specific conditions which are too variable for this test method to predict accurately.

5.3 Test Method D2274 yields results more rapidly than Test Method D4625, the 43 °C bottle test. However, as a result of the significantly elevated temperature and the pure oxygen atmosphere, the nature and amount of insolubles may deviate to a greater extent than Test Method D4625 from those formed in field storage.

6. Interferences

6.1 Oxidation is a major chemical process causing adherent and filterable insolubles to form. Any substance such as copper or chromium that catalyzes oxidation reactions will cause greater quantities of insolubles to form. Since the apparatus used in this test can also be used in Test Method D943, where coils of copper and steel are used, it is important that any residues that could contain these metals be eliminated from the apparatus by thorough cleaning prior to use. Similarly, to preclude the presence of chromium ions, as well as to protect laboratory personnel from potential harm, chromic acid shall not be used for cleaning glassware in the practice of this method.

6.2 It has been found that commercial grades of acetone, if used in the trisolvent, can have impurities which cause an apparently greater level of adherent insolubles to be measured. It is, therefore, critical that only reagent (or higher) grade materials be used in preparing the trisolvent mixture.

6.3 Ultraviolet light exposure has been found to increase the amount of total insolubles. Therefore, the fuel being tested shall be shielded from direct exposure to ultraviolet light (sunlight or fluorescent). Conduct all sampling, measuring, filtration, and weighing away from direct sunlight and in as dark an area as would be compatible with other laboratory operations. Storage before stress, the stress period and cool-down after stressing shall be in the dark.

7. Apparatus

NOTE 3—It is suggested that all measuring equipment be calibrated according to manufacturer's instructions on a periodic basis to assure consistency of results.

7.1 *Oxidation Cell,* of borosilicate glass, as shown in Fig. 1, shall consist of a test tube, condenser, and oxygen delivery tube. This cell is identical to that used in Test Method D943.

7.2 *Heating Bath,* with a thermostatically controlled liquid medium, shall be capable of maintaining the bath temperature at 95 °C ± 0.2 °C (203 °F ± 0.4 °F). It shall be fitted with a suitable stirring device to provide a uniform temperature throughout the bath. It shall be large enough to hold the desired number of oxidation cells immersed to a depth of approximately 350 mm. Further, the bath construction must permit shielding the fuel samples in the oxidation cells from light while they are undergoing oxidation.[4]

7.3 *Flowmeters,* shall have a capability of measuring 3 L⁄h ± 0.3 L⁄h of oxygen. One flowmeter shall be provided for each oxidation cell.

7.4 *Filter Drying Oven,* shall be capable of safely evaporating the solvent at 80 °C ± 2 °C (176 °F ± 4 °F) for the drying of filters.

7.5 *Glassware Drying Oven,* shall be capable of drying glassware at 105 °C ± 5 °C (221 °F ± 9 °F).

7.6 *Filtration Assembly,* see Fig. 2, shall be capable of holding the filters described in 7.7.

7.7 *Filter Media*[5], 47 mm diameter cellulose ester surfactant-free membrane filters with a nominal pore size of 0.8 µm.

7.7.1 Single filters are to be used for prefiltration.

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[4] This apparatus is available from suppliers of specialty petroleum testing equipment.

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1012. Filters may be qualified using the procedure in this research report.

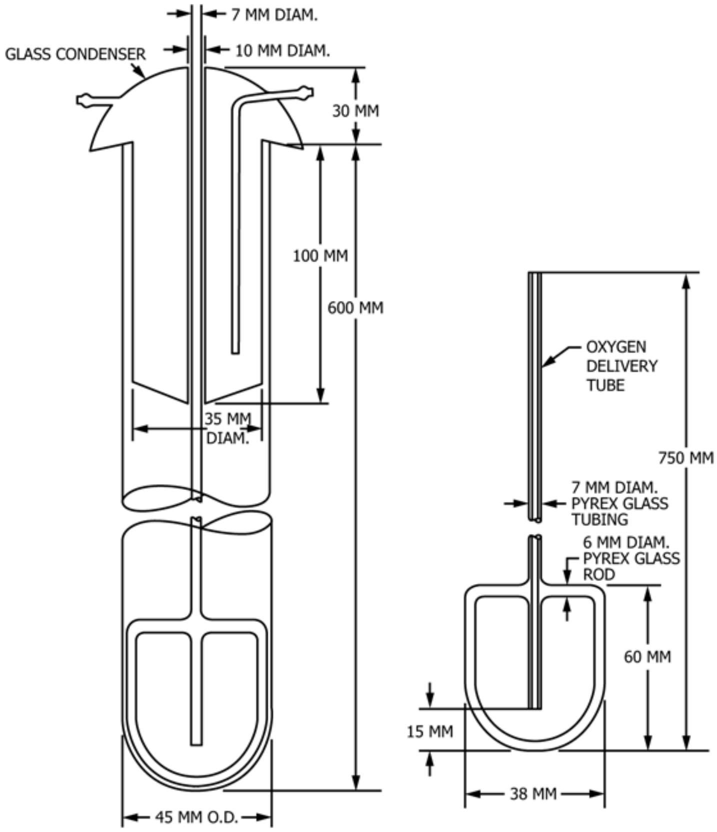

FIG. 1 Oxidation Cell

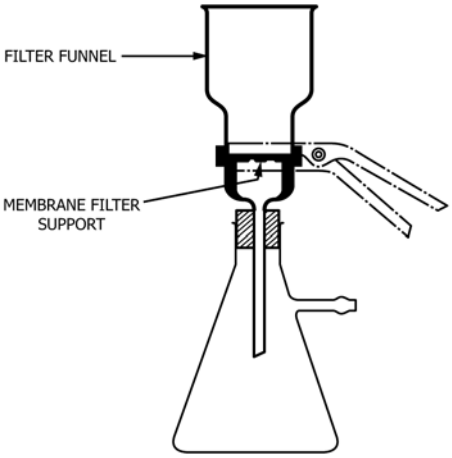

FIG. 2 Apparatus for Determining Filterable Insolubles

7.7.2 A matched weight pair of filters or alternatively, a preweighed control and sample, filters shall be used for determination of filterable insolubles

7.8 *Evaporating Vessel,* borosilicate glass beaker, 200 mL capacity, tall style.

7.9 *Hot Plate,* capable of heating a liquid in the evaporating vessel (7.8) to 135 °C (275 °F).

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall 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.

8.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean reagent water as defined by Type III of Specification D1193.

8.3 *2.2,4-trimethylpentanel (isooctane),* 99.75 % purity prefiltered through a filter medium of the type specified in 7.7.

8.4 *Oxygen,* 99.5 % purity or better. When the oxygen is delivered through a plant system of piping, a filter shall be provided adjacent to the constant temperature bath to prevent the introduction of line debris or moisture into the oxidation cells; a pressure regulator adequate to maintain a constant flow of gas through the apparatus shall also be used. A tank of oxygen of the specified purity can be used provided it is equipped with a two-stage pressure regulator. (**Warning—** Oxygen vigorously accelerates combustion. Do not use equipment having exposed surfaces containing oil or grease.)

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[6] *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.

8.5 *Trisolvent,* a mixture of equal volumes of acetone, methanol, and toluene. See 8.1. (**Warning**—It is particularly important that technical, commercial, practical, or industrial grades (however they are designated by the particular manufacturer) are not to be used, as their use may lead to apparently increased levels of adherent insolubles.) (**Warning**—Fire hazard, toxic.)

9. Samples and Sampling

9.1 When obtaining samples for the laboratory, follow Practices D4057 or D4177, or other standard practice capable of providing representative samples.

9.2 Analyze fuel samples as soon as possible after receipt. When a fuel cannot be tested within one day, blanket it with an inert gas such as oxygen-free nitrogen, argon, or helium and store at a temperature no higher than 10 °C (50 °F) but not lower than the cloud point. (**Warning**—Plastic containers are not acceptable for samples due to the potential for leaching of plasticizers. Samples should be taken preferably in metal cans previously cleaned according to Practice D4057. Borosilicate glass containers can be used if they are wrapped or boxed to exclude light. Do not use soft (soda) glass containers.)

9.3 *Test Samples*—Reduction of the laboratory sample to test sample size (about 400 mL for each determination) depends upon the size of sample received by the laboratory. If the laboratory sample is stored in a tank, drum, or 19 L (5 gal) or larger can, use the pertinent procedures of Practice D4057. Thoroughly mix smaller laboratory samples by shaking, rolling, or other techniques before taking an aliquot portion by pouring, pipetting, or other means. Clean any tube, thief, pipet, beaker, or other equipment that is to contact the laboratory sample with trisolvent and rinse with a portion of the sample prior to use. Prior to mixing thoroughly and taking an aliquot, allow samples that have been stored at temperatures much below 10 °C (50 °F) to warm to room temperature; thus allowing any separated wax to redissolve and to allow the viscosity to decrease to a point where mixing is effective.

10. Preparation of Apparatus

10.1 *Preparation of Glassware Other Than Oxidation Cells*—Rinse all glassware thoroughly with trisolvent followed by water, then wash with a mildly alkaline or neutral laboratory detergent. Rinse three times with deionized or distilled water followed by acetone to remove water.

10.2 *Preparation of Oxidation Cells and Accessories*—After completion of 10.1, fill oxidation cells with laboratory detergent in water. Place the oxygen delivery tube in the oxidation cell, place the condenser over the oxygen delivery tube and allow to soak at least two hours. Wash, drain, then rinse five times with tap water followed by three rinses with distilled or deionized water meeting Specification D1193 Type III requirements. Rinse with acetone; drain and allow the oxidation cell and oxygen delivery tube to dry.

10.3 *Preparation of Evaporating Beakers*—Dry the 200 mL cleaned beakers (10.1) for 1 h in an oven at 105 °C ± 5 °C (221°F ± 9 °F). Place the beakers in a desiccator (without desiccant) and allow them to cool for 1 h. Weigh beakers to the nearest 0.1 mg.

11. Procedure

11.1 *Preparing the Sample*—Place one filter (described in 7.7) on the filter support and clamp the filter funnel to the support as shown in Fig. 2. Apply suction (approximately 80 kPa (12 psi)). Pour 400 mL of the fuel through the filter (see 7.7) into a clean (10.1) 500 mL glass filtration flask. Repeat preparation for each sample to be run. After filtration is complete, discard the filter media. Never use the same filters for a second increment of fuel, because any material deposited on the filters by a previous increment of fuel can result in a greater removal of solids from the next increment.

11.2 *Assembling the Oxidation Apparatus:*

11.2.1 Place a clean oxygen delivery tube into a clean oxidation cell (Section 10) and pour 350 mL ± 5 mL of the filtered fuel into the cell. As soon thereafter as possible, but in no case more than 1 h after measuring the fuel, immerse the test cell in the 95 °C (203 °F) heating bath. *During any interim period store the cell in the dark.* The level of fuel in the oxidation cell shall be below the level of the liquid medium in the heating bath.

11.2.2 Place a condenser over the oxygen delivery tube and oxidation cell; connect the condenser to the cooling water. Connect the oxygen delivery tube to the oxygen supply through the flowmeter and adjust the oxygen flow to 3 L/h ± 0.3 L/h. Make sure samples are protected from light. When the number of test cells is less than the capacity of the heating bath, fill the empty positions with dummy oxidation cells containing 350 mL of a stable petroleum liquid with physical properties similar to those of the fuel being tested.

11.2.3 Record the time the first oxidation cell is placed in the bath as zero time.

NOTE 4—Military Specification MIL-F-16884 permits the use of a modified Test Method D2274 procedure in which the time in the heating bath is extended to 40 h. Periods other than 16 h may also be used in research or in other specifications. However, the precision values given in Test Method D2274 apply only to the 16 h period in the heating bath.

11.3 *Cooling the Sample:*

11.3.1 16 h± 0.25 h from zero time, remove the samples from the heating bath in the same sequence as they were placed therein. Cover the opening of each cell with a piece of aluminum foil or plastic to prevent entrance of dirt, dust, or moisture. Record the time the first cell is removed.

11.3.2 Place in a dark, ventilated site at room temperature, which shall be above the cloud point of the fuel, until the fuel attains room temperature but for no longer than 4 h.

11.4 *Determining Filterable Insolubles:*

11.4.1 Assemble the filter apparatus as illustrated in Fig. 2 using one set of matched pair filters (or preweighed control and sample filters). Apply vacuum as necessary to filter the sample in an adequate time (approximately 80 kPa (12 psi)); pour the cooled sample through the filter. *Filter all fuel through the filters before pouring isooctane rinses into the filter assembly.*

NOTE 5—If the filtration of the sample cannot be completed within 2 h as a result of severe filter plugging, filter the remaining fuel through a separate set of filters.

11.4.2 On completion of filtration, *completely* rinse the oxidation cell with three separate 50 mL ± 5 mL volumes of *iso*octane from a squeeze bottle or vessel capable of directing a fine stream of *iso*octane. Pass all rinsings through the filter assembly.

11.4.3 After filtration and rinsing of the oxidation apparatus is complete, wash down the sides of the funnel with an additional 50 mL ± 5 mL of *iso*octane directed as a fine stream. The stream of *iso*octane should be directed in a manner that, where possible, would push particles to the middle of the filter pad.

11.4.4 After filtration is complete, disconnect the top part of the filter assembly and wash down the rim of the filter media and adjacent parts of the filter assembly with a further 50 mL ± 5 mL of *iso*octane. Be certain the area of the filters previously under the filtration funnel is adequately rinsed to eliminate any presence of residual distillate fuel. Discontinue the vacuum and disconnect the filter assembly. Discard the filtrate.

11.4.5 Dry the two filters at 80 °C (176 °F) for 30 min, cool them for 30 min, and weigh the upper (sample) and lower (control) filters separately to the nearest 0.1 mg.

11.5 *Determining Adherent Insolubles:*

11.5.1 After the final rinsing of the oxidation cell and the oxygen delivery tube with *iso*octane, dissolve adherent insolubles from the surfaces of these pieces using three equal rinses totalling 75 ± 5 mL of trisolvent.

11.5.2 Examine the oxidation cell and the oxygen delivery tube for evidence of stain or color indicating incomplete removal. If such stain or color is noted, rinse with a fourth 25 mL volume of trisolvent.

11.5.3 Evaporate the trisolvent from the rinsings in either of two ways.

11.5.3.1 Collect the rinsings in a tared 200 mL tall beaker. Place the beaker and contents on a hot plate and evaporate the trisolvent at 135 °C under a hood. When all the solvent has evaporated, place the tared beaker in a desiccator (without desiccant) or similar container to cool for 1 h. The container should protect the beaker from airborne dust/dirt. After 1 h, weigh the beaker to the nearest 0.1 mg.

11.5.3.2 Optionally, collect the rinsings in one or two 100 mL beakers of the type specified for Test Method D381 and evaporate the trisolvent at 160 °C by the Test Method D381 air-jet method.

11.5.4 Run an adherent insolubles blank by evaporating a volume of trisolvent equal to that used in the test and correct for the presence of impurities in the solvent.

12. Calculation

12.1 Calculate the filterable insolubles mass (A) in milligrams per 100 mL. Subtract the mass of the blank (bottom) filter W_1, in milligrams, from that of the sample (top) filter W_2, in milligrams, (11.4) and divide by 3.5 to reduce the result to a 100 mL basis.

$$A = \frac{W_2 - W_1}{3.5} \quad (1)$$

12.2 Calculate the adherent insolubles mass (B) in milligrams per 100 mL. Subtract the tare mass of the blank (W_3) and sample (W_4) beakers from their final masses (W_5), (W_6) respectively. Subtract the mass of the blank from the corrected mass of the adherent insolubles (11.5) and divide by 3.5.

$$B = \frac{(W_6 - W_4) - (W_5 - W_3)}{3.5} \quad (2)$$

where:

W_3 = tare mass of the blank beaker, mg,
W_4 = tare mass of the sample beaker, mg,
W_5 = final mass of the blank beaker, mg, and
W_6 = final mass of the sample beaker, mg.

12.3 Calculate the total insolubles mass (C) in milligrams per 100 mL as the sum of the filterable insolubles (A) and the adherent insolubles (B).

$$C = A + B \quad (3)$$

where:

C = total insolubles, mg/100 mL,
A = filterable insolubles, mg/100 mL, and
B = adherent insolubles, mg/100 mL.

NOTE 6—Some specifications may require results in SI units of g/m^3 rather than as mg/100 mL. In such cases, the values in mg/100 mL can be converted to SI units by multiplying by ten.

13. Report

13.1 Report total insolubles (C) in mg/100 mL, to the nearest 0.1 mg/100 mL.

13.2 A report of filterable insolubles (A), and the adherent insolubles (B) is optional. Report units in milligrams per 100 mL.

14. Precision and Bias[7]

TABLE 1 Calculated Precision Data for Several Levels of Total Insolubles[A]

Total Insolubles, mg/ 100 mL	Repeatability	Reproducibility
0.1	0.30	0.60
0.5	0.45	0.89
1.0	0.54	1.06
1.5	0.60	1.17
2.0	0.64	1.26
2.5	0.68	1.33
3.0	0.71	1.40

[A] Same values apply if adherent insolubles are determined using optional drying method.

14.1 *Precision*—The precision (see Table 1) of this test method as determined by statistical examination of interlaboratory results is as follows:

14.1.1 *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 the normal and correct operation of the test method, exceed the following values only in one case in twenty:

$$0.54\left(\sqrt[4]{\text{total insolubles, mg/100 mL}}\right) \quad (4)$$

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[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1240.

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

$$1.06\left(\sqrt[4]{\text{total insolubles, mg/100 mL}}\right) \quad (5)$$

14.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for this test method no statement on bias is being made.

15. Keywords

15.1 diesel fuel; filterable insolubles; middle distillate fuel; oxidation stability

SUMMARY OF CHANGES

Subcommittee D02.14 has identified the location of selected changes to this standard since the last issue (D2274–10) that may impact the use of this standard. (Approved Dec. 1, 2014.)

(1) Numerous minor changes in Scope subsection 1.3, Terminology subsections 3.1.3 and 3.1.4, Significance and Use subsection 5.1, Apparatus Note 3 and subsections 7.4 and 7.6, Samples and Sampling subsection 9.3, Preparation of Apparatus subsection 10.3, and Procedure subsections 11.1, 11.2.1, 11.4.1, 11.4.5, and 11.5.3.1 to improve grammar, clarity, and conformance with ASTM Form and Style.

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