Designation: D7462 – 11

Standard Test Method for Oxidation Stability of Biodiesel (B100) and Blends of Biodiesel with Middle Distillate Petroleum Fuel (Accelerated Method)[1]

This standard is issued under the fixed designation D7462; 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.

1. Scope*

1.1 This test method covers a measurement of the oxidation stability of biodiesel (B100) blendstock as specified in Specification D6751 and blends of biodiesel with middle distillate petroleum fuels, including B6 to B20 blends as specified in Specification D7467 under specified oxidizing conditions at 95°C. Specifically, the oxidation stability is assessed by the formation and measurement of insoluble degradation materials.

NOTE 1—Biodiesel B100, composed of alkyl esters, can have good solubility for some products of oxidative degradation. Thus some B100 samples could undergo significant degradation, but show little or no sediment formation. By contrast, many petroleum diesel fuels have relatively poor solubility for products of oxidative degradation, so Bxx blends, such as B20, could show higher sediment levels. Refer to Appendix X1 for a suggested paraffinic dilution procedure to evaluate oxidative degradation of B100 for degradation materials that are soluble in B100, but insoluble in iso-octane.

NOTE 2—No. 1 and No. 2 grades in Specifications D396 or D975 currently allow up to 5% biodiesel meeting Specification D6751. This method is preferred for testing these fuels that may contain biodiesel blendstock rather than Test Method D2274 due to the incompatibility of biodiesel blendstock with the membrane filters specified in Test Method D2274.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *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
D2274 Test Method for Oxidation Stability of Distillate Fuel Oil (Accelerated Method)
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
D7467 Specification for Diesel Fuel Oil, Biodiesel Blend (B6 to B20)

3. Terminology

3.1 *Definitions:*

3.1.1 *adherent insolubles (formerly adherent gum), n*—material that is produced in the course of stressing 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 that is produced in the course of stressing the sample fuel under the conditions of this test and can be removed from the fuel by filtration. This

[1] 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 and Cleanliness of Liquid Fuels.

Current edition approved Jan. 1, 2011. Published March 2011. Originally approved in 2008. Last previous edition approved in 2008 as D7462–08. DOI: 10.1520/D7462-11.

[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.

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

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

3.2 *Definitions of Terms Specific to This Standard:*

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

3.2.1.1 *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 sample is aged at 95°C 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. The interior of the oxidation cell is also washed with hydrocarbon solvent to remove any filterable insolubles and liquid that can be removed by the solvent. These washings are also filtered and included as filterable insolubles.

4.2 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.

4.3 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 measurement of the oxidation stability of biodiesel and biodiesel blends.

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 storage conditions, which are too variable for this test method to predict accurately.

5.3 This test method 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 substances, such as copper or chromium, that catalyze oxidation reactions will cause more rapid or greater quantities of insoluble material, or both, 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 test method.

6.2 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

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 D2274.

7.2 *Heating Bath/Block,* shall be capable of maintaining a uniform temperature at 95 ± 1°C. It shall be large enough to hold the desired number of oxidation cells immersed to a depth of approximately 350 mm. Further, the construction must permit shielding the fuel samples in the oxidation cells from light while they are undergoing oxidation.[3]

7.3 *Flow Meters,* shall have a capability of measuring 3 ± 0.3 L/h of oxygen. One flow meter shall be provided for each oxidation cell.

7.4 *Filter Drying Oven,* shall be capable of safely evaporating the solvent at 90 ± 2°C for the drying of filter materials.

7.5 *Glassware Drying Oven,* shall be capable of drying glassware at 105 ± 5°C.

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

7.7 *Filter Media,* 47-mm diameter glass fiber membrane filters with a nominal pore size of 0.7 µm.

7.8 *Evaporating Beaker,* borosilicate glass beaker, of 100 to 200-mL capacity.

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

7.10 ALL equipment should be calibrated according to the manufacturer's instructions on a periodic basis to ensure consistency of results.

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.[4] 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.

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

[4] *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.