Designation: D5771 − 21

Designation: IP 444/09

Standard Test Method for Cloud Point of Petroleum Products and Liquid Fuels (Optical Detection Stepped Cooling Method)[1]

This standard is issued under the fixed designation D5771; 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.

INTRODUCTION

This test method describes an alternative procedure for the determination of cloud point of petroleum products Test Method D2500/IP 219 using an automatic apparatus. The temperature results from this test method have been found to be equivalent to Test Method D2500/IP 219. When specification requires Test Method D2500/IP 219, do not substitute this test method or any other method without obtaining comparative data and agreement from the specifier.

1. Scope*

1.1 This test method covers the description of the determination of the cloud point of petroleum products and biodiesel fuels that are transparent in layers 40 mm in thickness, by an automatic instrument using an optical device.

1.2 This test method covers the range of temperatures from −60 °C to +49 °C with temperature resolution of 0.1 °C, however, the range of temperatures included in the 1997 interlaboratory cooperative test program only covered the temperature range of −56 °C to +34 °C.

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

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

1.5 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D2500 Test Method for Cloud Point of Petroleum Products and Liquid Fuels
D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
D6708 Practice for Statistical Assessment and Improvement of Expected Agreement Between Two Test Methods that Purport to Measure the Same Property of a Material
D6751 Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels

2.2 *Energy Institute Standard:*[3]

IP 219 Test Method for Cloud Point of Petroleum Products[3]

2.3 *Other Standard:*[4]

IEC 751 Industrial Platinum Resistance Thermometer Sensors

3. Terminology

3.1 *Definitions:*

3.1.1 *biodiesel, n*—fuel comprised of mono-alkyl esters of long chain fatty acids derived from vegetable oils or animal fats, designated B100.

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[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 D02.07 on Flow Properties.

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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 Energy Institute, 61 New Cavendish St., London, WIG 7AR, U.K., http://www.energyinst.org.uk.

[4] Available from International Electrotechnical Commission (IEC), 3 rue de Varembé, Case postale 131, CH-1211, Geneva 20, Switzerland, http://www.iec.ch.

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

3.1.1.1 *Discussion*—Biodiesel is typically produced by a reaction of vegetable oil or animal fat with an alcohol such as methanol or ethanol in the presence of a catalyst to yield mono-alkyl esters and glycerin, which is removed. The finished biodiesel derives approximately 10 % of its mass from the reacted alcohol. The alcohol used in the reaction may or may not come from renewable resources.

3.1.2 *biodiesel blend (BXX), n*—a homogeneous mixture of hydrocarbon oils and mono-alkyl esters of long chain fatty acids.

3.1.2.1 *Discussion*—In the abbreviation, BXX, the XX represents the volume percentage of biodiesel fuel in the blend.

3.1.2.2 *Discussion*—The mono-alkyl esters of long chain fatty acids (that is, biodiesel) used in the mixture shall meet the requirements of Specification D6751.

3.1.2.3 *Discussion*—Diesel fuel, fuel oil, and non-aviation gas turbine oil are examples of hydrocarbon oils.

3.1.3 *biodiesel fuel, n*—synonym of biodiesel.

3.1.4 *cloud point, n—in petroleum products and biodiesel fuels*, the temperature of a liquid specimen when the smallest observable cluster of wax crystals first occurs upon cooling under prescribed conditions.

3.1.4.1 *Discussion*—The cloud point occurs when the temperature of the specimen is low enough to cause wax crystals to precipitate. In a homogeneous liquid, the cloud is always noted first at the location in the specimen where the specimen temperature is the lowest. The cloud point is the temperature at which the crystals first occur, regardless of their location in the specimen, and not after extensive crystallization has taken place. The wax crystals that precipitate at lower temperatures are typically, but not excluded to, straight-chain hydrocarbons and lipids.

3.1.4.2 *Discussion*—The purpose of the cloud point method is to detect the presence of the wax crystals in the specimen; however, trace amounts of water and inorganic compounds may be present. The intent of the cloud point method is to capture the temperature at which the liquids in the specimen begin to change from a single liquid phase to a two-phase system containing solid and liquid. It is not the intent of this test method to monitor the phase transition of the trace components, such as water.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *automatic cloud point, n*—the temperature of a specimen when the appearance of the cloud is determined under the conditions of this test method.

3.2.1.1 *Discussion*—The cloud point in this test method is determined by an automatic instrument using an optical device for detection of the crystal formation. The apparatus and the conditions are different from those established for Test Method D2500, although, according to interlaboratory examination the results have been determined to be equivalent to Test Method D2500.

3.2.2 *optical detection stepped cooling method, n—in cloud point test methods*, test procedure using prescribed cooling rate, specimen receptacle, and optical system for detection of crystal formation.

3.2.2.1 *Discussion*—The prescribed cooling rate is described in 4.1, the specimen receptacle is described in Section 6, and the optical system for the detection of crystal formation is described in Section 6.

3.2.3 *D2500/IP 219 equivalent cloud point, n*—the temperature of a specimen, in integers, calculated by rounding the results of this test method to the next lower integer.

3.2.3.1 *Discussion*—This test method produces results with 0.1 °C resolution. Should the user wish to provide results with a similar format to Test Method D2500, then this calculation can be performed. Some apparatus can perform this calculation automatically.

4. Summary of Test Method

4.1 After insertion of the prescribed test jar (6.3) containing the specimen into the apparatus, and the initiation of the program, the specimen is cooled incrementally according to the cooling profile listed in Table 1. The specimen is continuously monitored by a reflective optical system (6.1 and Fig. 1) for the formation of a crystalline structure. When the crystallization of the wax in the specimen is detected by the optical system, the temperature is recorded to within 0.1 °C resolution. The specimen is then heated to facilitate the start of the next test.

5. Significance and Use

5.1 For petroleum products and biodiesel fuels, the cloud point is an index of the lowest temperature of their utility for certain applications. Wax crystals of sufficient quantity can plug filters used in some fuel systems.

5.2 Petroleum blending operations require precise measurement of the cloud point.

5.3 This test method can determine the temperature of the test specimen at which wax crystals have formed sufficiently to be observed as a cloud with a resolution of 0.1 °C.

5.4 This test method provides results that, when rounded to the next lower integer, are equivalent to Test Method D2500. Refer to 12.2.

5.5 This test method is more precise than Test Method D2500.

NOTE 1—According to interlaboratory examination, the reproducibility of this test method has been found to be more precise than Test Method D2500.

6. Apparatus

6.1 *Optical Cloud Point Apparatus*[5]—The automated cloud point apparatus (see Fig. 2) described in this test method consists of a microprocessor controller that is capable of controlling one or more independent test cells. The apparatus shall include provisions for independently controlling the temperature of each cell according to the specified cooling profile, continuously monitoring the specimen temperature, detecting the appearance of the cloud point at the bottom of the

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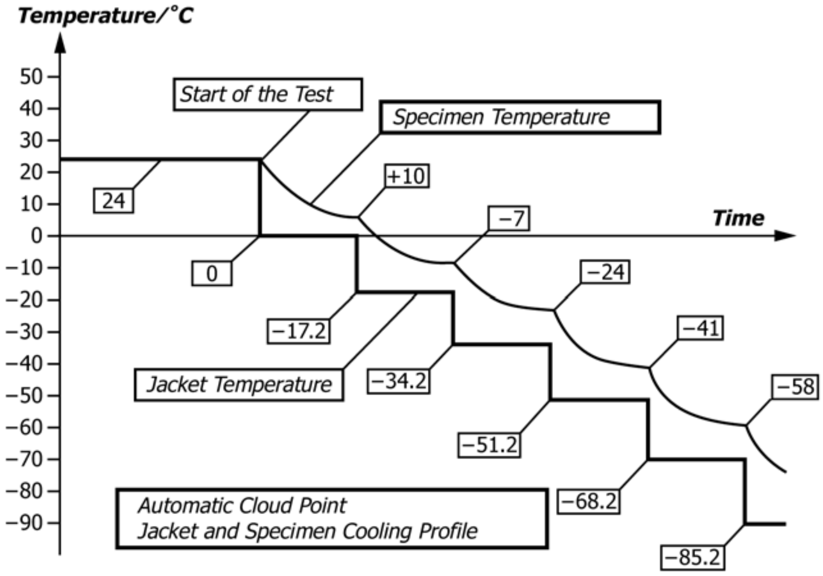
[5] The sole source of supply of the ISL Model CPP97-6 and CPP97-2 and CPP-5Gs known to the committee at this time is ISL SA, BP 40, 14790 Verson, France. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

TABLE 1 Jacket and Specimen Cooling Temperatures

Specimen Temperature, °C	Jacket Temperature, °C
ST > + 10	0 ± 0.5
+ 10 ≥ ST > – 7	–17.2 ± 0.5
–7 ≥ ST > – 24	–34.2 ± 0.5
–24 ≥ ST > – 41	–51.2 ± 0.5
–41 ≥ ST > – 58	–68.2 ± 0.5
–58 ≥ ST > – 75	–85.2 ± 0.5



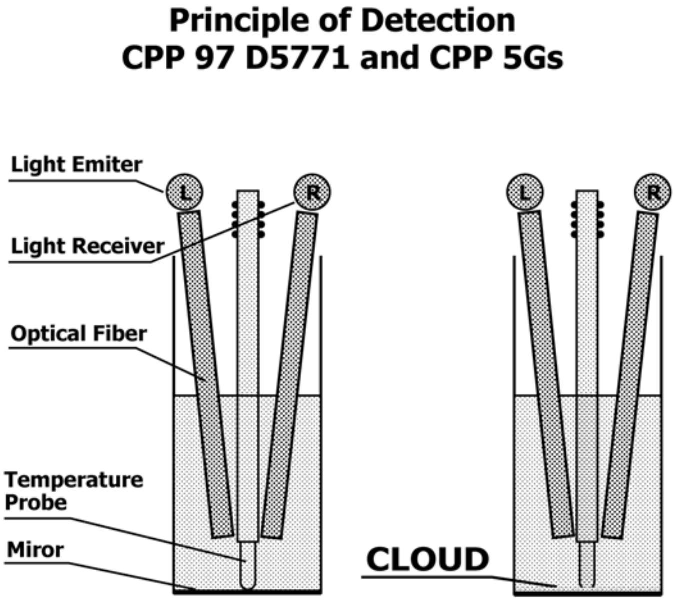


FIG. 1 Test Jar and Detection System

test jar without removing it from the jacket, and displaying the 0.1 °C or 1.0 °C result (see Fig. 1 and Fig. 3).

FIG. 2 Automated Cloud Point Apparatus

6.2 *Temperature Probe, IEC 751 Class A:* $\Delta T = \pm(0.15 + 0.002\,|T|)$, capable of measurement from −50 °C to +80 °C. The temperature probe shall be in contact with the bottom of the test jar.

6.3 *Test Jar,* clear cylindrical glass, mirrored flat bottom, 34 mm ± 0.5 mm outside diameter, 1.4 mm ± 0.15 mm wall thickness, 120 mm ± 0.5 mm height, thickness of the bottom

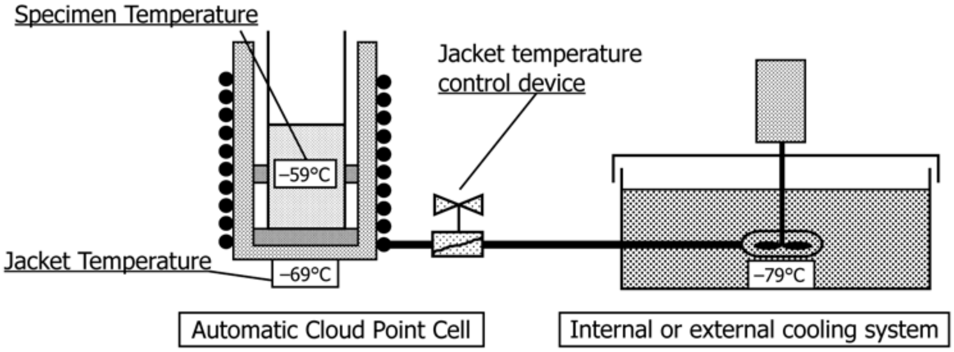


FIG. 3 Test Jar Cooling Chamber and Cooling System

2.0 mm ± 0.5 mm, marked with a line to indicate the sample height 54 mm ± 0.5 mm above the inside bottom.

6.4 *Jacket,* brass, cylindrical, flat bottom, 113 mm ± 0.2 mm in depth, 45 mm ± 0.1 mm inside diameter. It must be cooled according to the cooling profile specified.

6.5 *Cooling System,* either an external system equipped with a circulating pump and capable of maintaining a temperature at least 10 °C below the last required jacket temperature level (see Table 1 and Fig. 3), or an internal system capable of maintaining the required jacket temperatures (see Table 1 and Fig. 3).

6.6 *Cork Disk,* 6 mm ± 0.2 mm thick, to fit loosely inside the jacket. Felt may be used but special attention must be given to the potential for moisture in the felt disk. The felt must be dried before each test.

6.7 *Cork Ring,* to fit snugly around the outside of the test jar and loosely inside the sample cell. Its purpose is to prevent the test jar from touching the jacket.

7. Reagents and Materials

7.1 *Cleaning Solvents,* suitable for cleaning and drying the test cell, such as petroleum naphtha and hexane.

7.2 *Methyl Alcohol, Anhydrous,* for use as cooling medium in circulating bath cooling system, when used.

7.3 *Sodium Sulfate*—A reagent grade of anhydrous sodium sulfate should be used when required. (**Warning—**Flammable. Liquid causes eye burns. Vapor harmful. May be fatal or cause blindness if swallowed or inhaled.)

7.4 Lint-free filter paper may be used as a drying medium.

8. Sampling

8.1 Obtain a sample in accordance with Practice D4057 or D4177.

8.2 Samples of very viscous materials may be warmed until they are reasonably fluid before they are sampled. However, no sample should be heated more than is absolutely necessary.

8.3 The sample shall not be heated above 70 °C. If the sample is heated above 70 °C, allow the sample to cool below 70 °C before filtering or inserting into the apparatus.

8.4 When moisture is present in the sample, remove the moisture by a method such as filtration through dry, lint-free filter paper, until the oil is perfectly clear, but make such filtration at a temperature at least 14 °C above the expected cloud point.

NOTE 2—A wax cloud or haze is always noted at the bottom of the test jar where the temperature is lowest. A slight haze throughout the entire specimen, which slowly becomes more apparent as the temperature is lowered, is usually due to water in the specimen. Generally this water haze will not interfere with the determination of the wax cloud point. In most cases of interference, filtration through dry lint-free filter papers such as described in 11.2 is sufficient.

8.5 For diesel fuels, if the haze is very dense, a fresh portion of sample must be dried by shaking 100 mL with 5 g of anhydrous sodium sulfate for at least 5 min and then filtering through dry lint-free filter paper. Given sufficient contact time, this procedure will remove or sufficiently reduce the water haze so that the wax cloud can be readily discerned. Drying and filtering should always be done at a temperature at least 14 °C above the approximate cloud point, but otherwise not in excess of 49 °C.

9. Preparation of Apparatus

9.1 Prepare the instrument for operation in accordance with the manufacturer's instructions.

9.2 Clean and dry the test head and test jar using suitable solvents as prescribed by the manufacturer.

9.3 Adjust the set point of the cooling system, when necessary, to the appropriate temperature to cool the jacket to the required temperature (see Table 1).

10. Calibration and Standardization

10.1 Ensure that all of the manufacturer's instructions for calibrating, checking, and operating the apparatus are followed.

10.1.1 A test head simulator with a known resistance is used to calibrate the equipment. Follow the manufacturer's calibration instructions.

NOTE 3—A test head simulator may be obtained from the apparatus supplier which can assist in verification of temperature measurement.

10.2 A sample with a mutually agreed upon cloud point such as a sample from a interlaboratory test program can be used to verify performance of the apparatus.

11. Procedure

11.1 Set the temperature unit on the apparatus to 1 °C or to 0.1 °C.

NOTE 4—The temperature unit, in common laboratory practice, is 1 °C. If a higher resolution is desired, 0.1 °C can be selected.

11.2 Using a water bath or oven, bring the sample to be tested to a temperature at least 14 °C above the expected cloud point. When moisture is present in the sample, remove the moisture present by a method, such as filtration through dry lint-free filter paper, until the sample is perfectly clear, but make such filtration at a temperature of at least 14 °C above the approximate cloud point but otherwise not in excess of 70 °C.

11.3 Pour the clear sample into the test jar to the level mark.

11.4 Place a cork disk at the bottom of the jacket in the appropriate cell and fit a cork ring around the test jar. Use the cork placement guide, if necessary, for the final adjustment. The cork ring must be 25 mm ± 3.0 mm above the bottom of the test jar.

NOTE 5—A cork placement guide may be obtained from the apparatus supplier which can assist in proper placement of the cork ring.

11.5 Place the test jar in the appropriate test cell. Attach the detector head according to the manufacturer's instructions.

11.6 Start the test according to the manufacturer's instructions. The instrument shall then automatically adjust the jacket temperature in accordance with Table 1 and begin to optically monitor the specimen for the cloud point. The instrument shall monitor and display the jacket temperature and the specimen temperature during the test procedure.

11.7 The instrument shall continue to monitor and adjust the jacket temperature according to Table 1. The time to move from one jacket temperature level to the next jacket temperature level shall not exceed 200 s, for jacket temperatures down to −52 °C.

NOTE 6—For lower jacket temperatures, the time to move from one jacket temperature level to the next jacket temperature should not exceed 300 s. Maintain cooling system temperatures as low as possible to attain these jacket temperatures in the shortest time period possible and utilize cooling system with cooling capacity capable of achieving the lowest temperature required.

11.8 The instrument shall detect the cloud point at the bottom of the test jar and record and display the temperature as selected in 11.1. The instrument shall have an audible alarm, such that when a cloud point is detected, it immediately notifies the operator. This function allows the operator to remove the specimen from the instrument and visually inspect the cloud formation and verify the result, if desired.

11.9 After the determination, the instrument shall adjust the jacket temperature to 23 °C ± 2 °C.

12. Report

12.1 If 0.1 °C was selected in 11.1, report the temperature recorded in 11.9 as automated cloud point Test Method D5771.

12.2 If 1.0 °C was selected in 11.1, report the temperature recorded in 11.9 as Test Method D2500 equivalent cloud point Test Method D5771.

13. Precision and Bias

13.1 *Precision*—The precision of this test method as determined by the statistical examination of the interlaboratory test results[6,7] is as follows:

13.1.1 *Repeatability*—The difference between successive test results, obtained by the same operator using the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of this test method, exceed 2.2 °C only in one case in twenty.

13.1.2 *Reproducibility*—The difference between two single and 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 this test method, exceed 3.9 °C only in one case in twenty.

13.1.3 The precision statements were derived from a 1997 interlaboratory cooperative test program.[7] Participants analyzed eleven sample sets as blind duplicates, comprised of various distillate fuels and lubricating oils with a temperature range from +34 °C to −56 °C. Ten laboratories participated with the automatic machines and eight laboratories participated with the manual Test Method D2500/IP 219. Information on the type of samples and their average cloud points are in the research report.

13.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedure in this test method, bias has not been determined.

13.3 *Relative Bias*—The results of the interlaboratory program were examined for bias relative to Test Method D2500/IP 219. Although statistically significant bias was observed, the magnitude was determined small enough (−0.56 °C) to be of little practical significance.

13.4 *Precision for Biodiesel Products*—The precision of this test method as determined by the statistical examination of the interlaboratory test results is as follows:

13.4.1 *Repeatability for Blends of Biodiesel in Diesel*—The difference between successive test results, obtained by the same operator using the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of this test method, exceed 1.2 °C only in one case in twenty.

13.4.2 *Reproducibility for Blends of Biodiesel in Diesel*—The difference between two single and independent test 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 this test method, exceed 2.7 °C only in one case in twenty.

NOTE 7—The precision for blends of biodiesel in diesel samples comprised cloud points from about −2 °C to +10 °C.

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[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1373. Contact ASTM Customer Service at service@astm.org.

[7] Supporting data (the results of the 1997 interlaboratory cooperative test program) have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1508. Contact ASTM Customer Service at service@astm.org.

13.4.3 The precision statements were derived from a 2001 interlaboratory cooperative test program.[8] Participants analyzed eleven sample sets comprised of two petroleum distillate fuels, diesel and kerosine, with various biodiesel fuels with a temperature range from +10 °C to –45 °C. Nine laboratories participated with the automatic machines, and ten laboratories participated with the manual Test Method D2500/IP 219. Information on the type of samples and their average cloud points are in the research report.

NOTE 8—One of the outcomes of the interlaboratory study was the selection of the sample types used in the study contributed to a difficulty in determining the precision statement. Kerosine is a sufficiently different fuel type from biodiesel to cause some slight separation of phases upon cooling when in B20 blends. Also, the particular kerosine sample used was atypical, which complicated the study further. Therefore data from the blends of kerosine in biodiesel were not used in the precision statement. In addition, the diesel fuel used in the research report was high cloud point material. Due to the cloud point of the base diesel material, the temperature range of the precision statement was limited.

NOTE 9—A future interlaboratory cloud study will be done including a wider range of biodiesel fuels with various distillate blend stocks.

13.5 *Bias for Biodiesel Products*—Since there is no accepted reference material suitable for determining the bias for the procedure in this test method, bias has not been determined.

13.6 *Relative Bias*—The statistical analysis for the interlaboratory test program for bias relative to Test Method D2500/IP 219 has not been determined for blends of biodiesel in diesel samples.

13.7 *Relative Bias between Models CPP97-6(2) and CPP-5Gs*[9]—The statistical analysis of the between method bias by Practice D6708 indicates that there is no statistical bias between the models of instruments.

14. Keywords

14.1 automatic cloud point; cloud point; petroleum products

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[8] Supporting data (the results of the 2001 interlaboratory cooperative test program) have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1524. Contact ASTM Customer Service at service@astm.org.

[9] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1740. Contact ASTM Customer Service at service@astm.org.

SUMMARY OF CHANGES

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D5771 – 20) that may impact the use of this standard. (Approved Jan. 1, 2021.)

(1) Revised definitions for biodiesel blend and added biodiesel fuel in Section 3.

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D5771 – 17) that may impact the use of this standard. (Approved June 1, 2020.)

(1) Revised definitions for biodiesel and biodiesel blend in Section 3.