Designation: D7683 – 11

Standard Test Method for Cloud Point of Petroleum Products (Small Test Jar Method)[1]

This standard is issued under the fixed designation D7683; 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 the determination of the cloud point of petroleum products, biodiesel, and biodiesel blends that are transparent in layers 40 mm in thickness, using an automatic instrument.

1.2 The measuring range of the apparatus is from -65 to 51°C, however the precision statements were derived only from samples with cloud point temperatures from -50 to +6°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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D2500 Test Method for Cloud Point of Petroleum Products

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

2.2 *Energy Institute Standards:*[3]

IP219 Test Method for Cloud Point of Petroleum Products

3. Terminology

3.1 *Definitions:*

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

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-esters and glycerin. The fuel typically may contain up to 14 different types of fatty acids that are chemically transformed into fatty acid methyl esters (FAME).

3.1.2 *biodiesel blend (BXX), n*—blend of biodiesel fuel with petroleum-based diesel fuel designated BXX, where XX is the volume percentage (as a whole number without the percentage sign) of biodiesel.

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

3.1.3.1 *Discussion*—The cloud point occurs when the temperature of the specimen is low enough to cause hydrocarbon 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 hydrocarbon crystals that precipitate at lower temperatures are typically, but not excluded to, straight chain hydrocarbons commonly called “wax crystals.”

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *small test jar method, n—in cloud point test methods*, automatic test procedure using a small sample size, prescribed cooling rate, specimen receptacle, and optical system for detection of crystal formation.

3.2.1.1 *Discussion*—The prescribed cooling rate is described in 11.4, the specimen receptacle is described in 6.3, and the optical system for detection of crystal formation is described in A1.2.3.

3.2.2 *D2500/IP219 equivalent cloud point, n*—temperature of a specimen, in integers, calculated by applying a bias and rounding the results of this test method to the next lower integer (see 12.2).

3.2.2.1 *Discussion*—This test method produces results with 0.1°C resolution. Should the user wish to provide results with

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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.

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 inserting the glass test jar containing the specimen into the automatic apparatus and initiating the test program, the specimen is heated, if necessary, to the designated temperature and then cooled by prescribed rates. (See 11.4.) The test specimen is continuously monitored for appearance of hydrocarbon crystals with a light emitter and a light receiver through coaxial type optical fibers. (See A1.2.3.) When the crystallization in the specimen is detected by the optical system, the temperature is recorded to 0.1°C resolution. The specimen is then heated to facilitate the start of the next test.

5. Significance and Use

5.1 The cloud point of petroleum products and biodiesel fuels 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 corrected for bias and rounded to the next lower integer (see 12.2), have been found equivalent to Test Method D2500.

5.5 This test method determines the cloud point in a shorter time period than required by Test Method D2500.

6. Apparatus

6.1 *Automatic Apparatus*—The automatic cloud point apparatus described in this test method is a microprocessor controlled apparatus that is capable of heating and cooling a test specimen at prescribed rates, optically observing the first appearance of hydrocarbon wax crystals, and recording the temperature of the test specimen. A detailed description of the apparatus is provided in Annex A1.

6.2 *Temperature Measuring Device*—The temperature measuring device in the specimen chamber shall be capable of measuring the temperature from -65 to 51°C at a resolution of 0.1°C.

6.3 *Test Jar*—Clear cylindrical borosilicate glass with a flat bottom with an approximate capacity of 12 mL. Approximately 4.5 mL of sample specimen is contained when filled to the scribed line. During the test, the test jar is fitted with a test jar cap assembly on its top. See A1.1.2 for more details on the test jar.

6.4 *Metallic Block Bath*—Metallic block with a cylindrical hole to fit the test jar. The metallic block assembly shall have a provision for cooling/heating. A temperature sensor is embedded in the metallic block to monitor its temperature.

7. Reagents and Materials

7.1 *Cleaning Agents*—Capable of cleaning and drying the test jar and test jar cap assembly, after each test. Chemical agents such as alcohol and petroleum-based solvents have been found suitable to use. (**Warning—**Flammable.) (**Warning—**May be harmful by itself or when evaporated.)

8. Sampling

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

8.2 A minimum volume of 4.5 mL of sample is required for each test.

8.3 Samples of very viscous materials may be warmed until they are reasonably fluid before they are tested. However, no sample should be heated more than is absolutely necessary to facilitate pouring the sample into the instrument test jar. (**Warning—**Exercise care when selecting the preheating temperature. Samples which are fluid at ambient room temperature can also have a low flash point. Use higher preheating temperatures only on samples known to be solid near ambient room temperature.)

8.4 The sample shall not be heated above 60°C. When the sample is heated above 60°C allow the sample to cool below 60°C before filtering or inserting into the apparatus.

8.5 When moisture is present, 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 1—Moisture will be noticed in the sample as a separate phase or as a haze throughout the entire sample. Generally, a slight haze will not interfere with the detection of the wax cloud.

9. Preparation of Apparatus

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

10. Calibration and Standardization

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

10.2 A sample with a well documented cloud point can be used to verify the performance of the automatic apparatus. Alternatively, a sample that has been extensively tested in a cloud point cross-check program can be used. Such verification materials can also be prepared from intra-company cross-checks.

11. Procedure

11.1 Pour the sample specimen into the test jar to the scribed mark. When necessary, heat the sample in a bath or oven until it is just sufficiently fluid to pour into the test jar. Samples with an expected cloud point above 36°C or samples which appear solid at room temperature can be heated above 45°C, but they shall not be heated above 60°C.

11.2 Insert the charged test jar into the metallic block bath, and install the test jar cap assembly snugly.