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Standard Test Method for Cloud Point of Petroleum Products and Liquid Fuels (Constant Cooling Rate Method)[1]

This standard is issued under the fixed designation D5773; 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 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 a constant cooling rate.

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

2.2 *Energy Institute Standard:*[3]

IP 219 Test Method for Cloud Point of Petroleum Products

IP 446 Determination of the Cloud Point of Petroleum Products—Automatic Constant Cooling Rate Method

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.

3.1.1.1 *Discussion*—Biodiesel is typically produced by a reaction of a 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.

[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.

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

3.1.2 *biodiesel blend, n*—blend of biodiesel fuel with diesel fuels and fuel oils.

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

3.1.3 *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.

3.1.3.1 *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.

3.1.3.2 *Discussion*—The purpose of the cloud point is to measure the wax crystals in the specimen; however, trace amounts of water and inorganic compounds may also be present. The intent of the cloud point measurement is to capture the temperature at which the liquid fuel in the specimen begins 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 *constant cooling rate 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 Annex A1, and the optical system for the detection of crystal formation is described in Annex A1.

3.2.3 *Peltier device, n*—a solid-state thermoelectric device constructed with dissimilar semiconductor materials and configured in such a way that it will transfer heat to or away from a test specimen dependent on the direction of electric current applied to the device.

3.2.4 *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.

3.2.4.1 *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 A prescribed specimen (11.5) is cooled by a Peltier device (A1.1) at a constant rate of 1.5 °C⁄min ± 0.1 °C⁄min while continuously being illuminated by a light source (A1.1.4). The specimen is continuously monitored by an array of optical detectors (A1.1.5, Fig. A1.1) for the first appearance of a cloud of wax crystals. The detectors are sufficient in number to ensure that any solid-phase hydrocarbon crystals that may form are detected. The temperature at which the appearance of a cloud of wax crystals is first detected in the specimen is recorded to 0.1 °C resolution. When the recorded temperature is rounded to the next lower integer temperature, it is designated as the D2500/IP 219 equivalent cloud point per Test Method D5773.

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 a 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 are equivalent to Test Method D2500.

NOTE 1—This is based on the Test Method D2500 equivalent cloud point in which the 0.1 °C result is rounded to the next lower integer.

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

NOTE 2—In cases of samples with cloud points near ambient temperatures, time savings may not be realized.

5.6 This test method eliminates most of the operator time required of Test Method D2500.

5.7 This test method does not require the use of a mechanical refrigeration apparatus.

NOTE 3—In certain cases of high ambient temperature, a source of cooling water may be required to measure low-temperature cloud points (see 7.1).

6. Apparatus

6.1 *Automatic Apparatus*[4]—The automatic cloud point apparatus described in this test method consists of a test chamber

[4] The sole source of supply of the Phase Technology Cloud Point Analyzer model series 10, 30, 70, 70V, and 70X known to the committee at this time is Phase Technology, 11168 Hammersmith Gate, Richmond, B.C. Canada V7A 5H8. The various model series mentioned above are differentiated by their cooling capacities and user interfaces; however, all of them are capable of covering the entire temperature range specified in the scope. 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.

controlled by a microprocessor that is capable of controlling the heating and cooling of the test specimen, optically observing the first appearance of a cloud of wax crystals and recording the temperature of the specimen described in detail in Annex A1.

6.2 The apparatus shall be equipped with a specimen cup, optical detector array, light source, digital display, Peltier device, and a specimen temperature measuring device.

6.3 The Peltier device shall be capable of heating or cooling the test specimen at a constant rate of 1.5 °C/min ± 0.1 °C/min.

6.4 The temperature-measuring device in the specimen cup shall be capable of measuring the temperature of the test specimen from −40 °C to +70 °C at a resolution of 0.1 °C.

6.5 The apparatus shall be equipped with fittings to permit the circulation of a liquid cooling medium, if required, to remove heat generated by the Peltier device and other electronic components of the apparatus.

NOTE 4—Some apparatus are designed to use ambient air as a cooling medium. In such cases, a built-in fan is available to provide circulation of air and there is no need for fittings as described for a liquid cooling medium. The function of the cooling medium is to remove heat from the electronic components. The choice of the cooling medium has no impact whatsoever on the test results.

6.6 The apparatus shall be equipped with fittings to permit the circulation of purge gas to purge the test chamber containing the specimen cup of any atmospheric moisture.

7. Reagents and Materials

7.1 *Cooling Medium*—Air, tap water, or other liquid heat exchange medium sufficient to remove heat generated by the Peltier device and other electronic components from the apparatus. To achieve specimen cooling to −40 °C, supply circulation of liquid cooling medium at +25 °C or lower to the apparatus. For an apparatus which relies on air as cooling medium, the ambient air temperature has to be below +30 °C to achieve specimen cooling to −40 °C.

7.2 *Purge Gas*—A gas such as air, nitrogen, helium, or argon with a dew point below the lowest operating temperature of the analyzer. (**Warning—**Compressed gas under high pressure.) (**Warning—**Inert gas can be an asphyxiant when inhaled.)

7.3 *Precision Volume Dispensing Device,* capable of dispensing 0.15 mL ± 0.01 mL of sample.

7.4 *Cotton Swabs*—Plastic or paper shaft cotton swabs used to clean the sample cup. (**Warning—**The use of swabs with wooden shafts may damage the mirrored surface of the specimen cup.)

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 tested. However, no sample should be heated more than absolutely necessary.

8.3 The sample shall not be heated above 70 °C. When 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 5—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.

9.2 Make liquid cooling medium connections if required (see Note 4) and ensure that they do not leak.

9.3 Make purge gas connections and ensure that they do not leak.

9.4 Turn on the liquid cooling medium if required (see Note 4).

9.5 Turn on the purge gas.

9.6 Turn on the main power switch of the analyzer. After the automatic self diagnostics startup sequence is completed, the instrument will display a **READY** message.

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 mutually agreed upon cloud point can be used to verify performance of the apparatus.

11. Procedure

11.1 Inspect the specimen cup to ensure it is clean and dry. If not, clean the cup (see 11.3).

11.2 Deliver 0.15 mL ± 0.01 mL of specimen into the specimen cup. Pipette, syringe, or precision positive-displacement devices are suitable for use in delivering the specimen.

11.3 Clean the specimen out of the cup. The cup must be cleaned to the point where no visible droplets of specimen remain in the cup. Non-abrasive absorbent materials such as cotton swabs are suitable for use in cleaning the specimen cup. Cleaning solvents able to clean the specimen and compatible with the components of the apparatus can also be used. Naphtha, hexane, heptane, and toluene are suitable as cleaning solvents.

11.4 Repeat steps 11.2 and 11.3.

11.5 Carefully measure 0.15 mL ± 0.01 mL of specimen into the specimen cup.

11.6 Close and lock the test chamber lid.

11.7 Select the **PRE-HEAT** menu on the apparatus if the expected cloud point is less than 14 °C below the specimen