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Designation: 444/99

Standard Test Method for Cloud Point of Petroleum Products (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 to $+49^{\circ}\text{C}$ 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 to $+34^{\circ}\text{C}$.

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

2.2 *Energy Institute Standard:*³

IP 219 Test Method for Cloud Point of Petroleum Products³

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 B 100.

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, n*—a blend of biodiesel fuel with petroleum-based diesel fuel designated BXX, where XX is the volume % of biodiesel.

3.1.3 *cloud point, n—in petroleum products and biodiesel fuels*, the temperature of a liquid specimen when the smallest

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products 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.

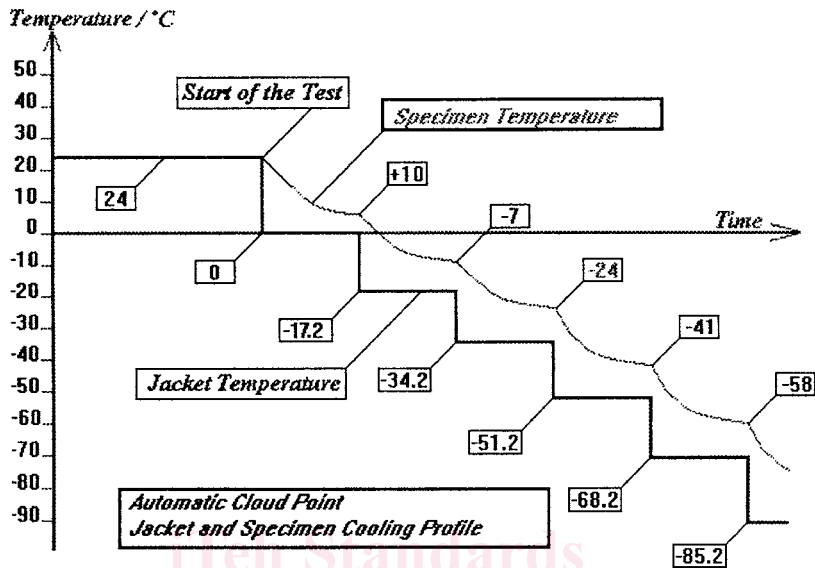
³ Available from Energy Institute, 61 New Cavendish St., London, WIG 7AR, U.K., <http://www.energyinst.org.uk>.

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

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

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



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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.”

3.1.3.2 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

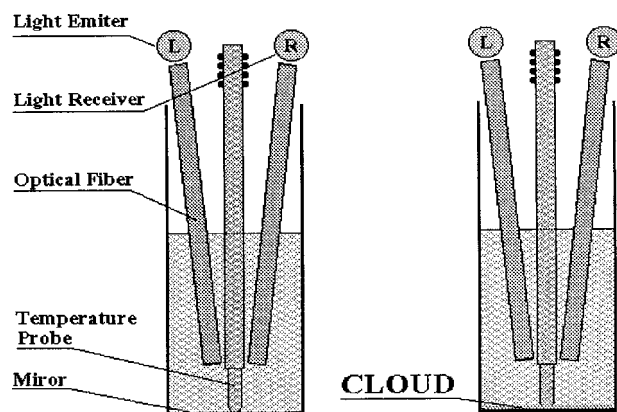


FIG. 1 Test Jar and Detection System

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

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

⁵ The sole source of supply of the ISL Model CPP97-6 and CPP97-2 known to the committee at this time is ISL North America, Stevensville, MD or 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 committee¹, which you may attend.

6.3 *Test Jar*, clear cylindrical glass, mirrored flat bottom, 34 ± 0.1-mm outside diameter, 31 ± 0.3-mm inside diameter, 120 ± 0.5-mm height, thickness of the bottom 2.0 ± 0.5 mm, marked with a line to indicate the sample height 54 ± 0.5 mm above the inside bottom.

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

6.5 *Cooling Circulating Bath*, 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).

6.6 *Cork Disk*, 6 ± 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.

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.)

8. Sampling

8.1 Obtain a sample in accordance with Practices D4057 or by 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.