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.

[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.

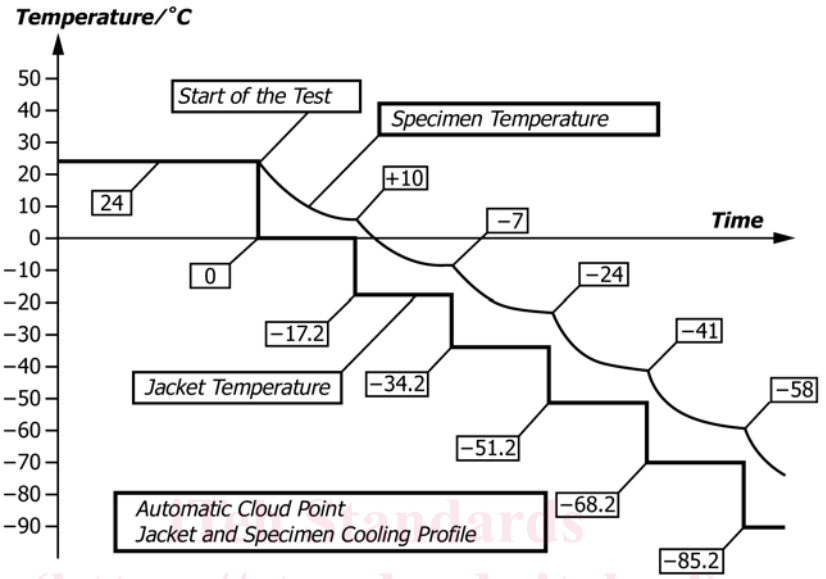
6. Apparatus

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

[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



Principle of Detection
CPP 97 D5771 and CPP 5Gs

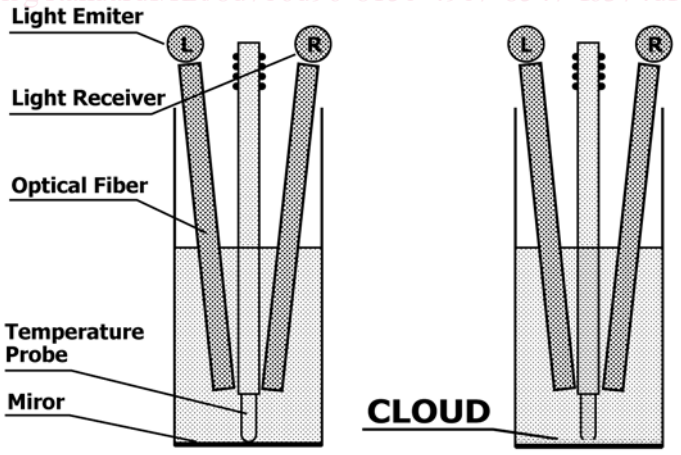
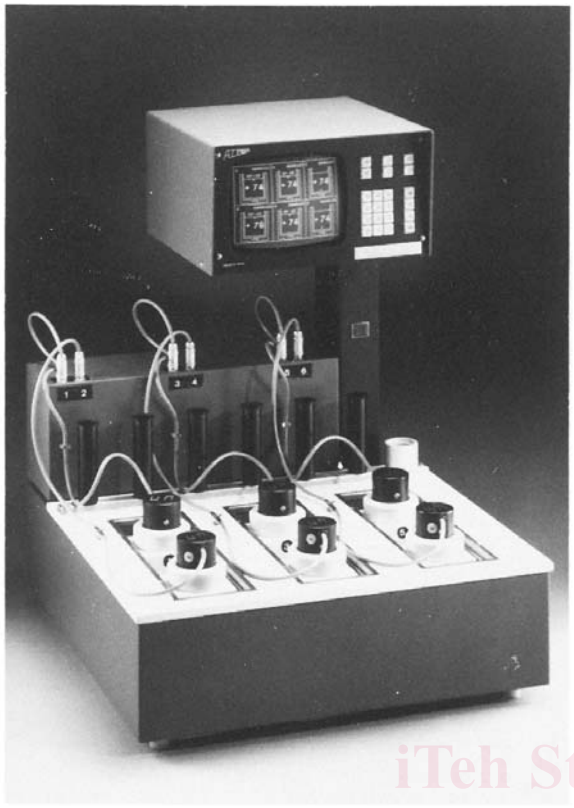


FIG. 1 Test Jar and Detection System

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).



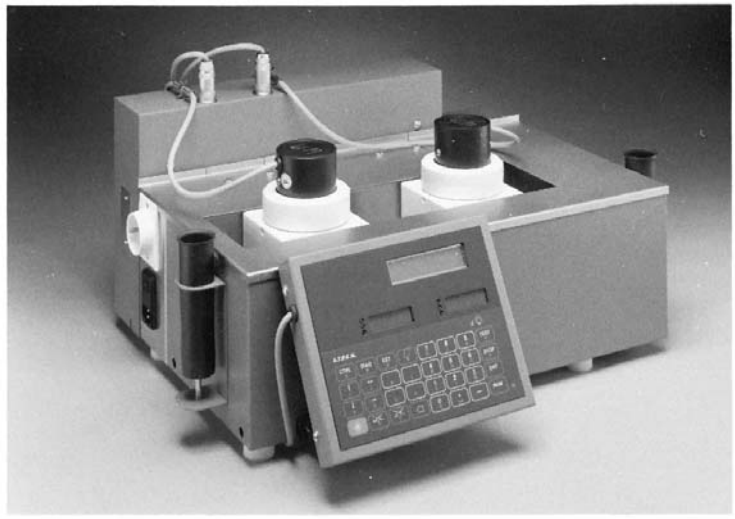


FIG. 2 Automated Cloud Point Apparatus

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