Designation: ~~D5772 – 20~~ D5772 – 21

Designation: IP 445/09

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

This standard is issued under the fixed designation D5772; 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 of 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 a linear 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

[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.

Current edition approved ~~June 1, 2020~~Jan. 1, 2021. Published ~~June 2020~~January 2021. Originally approved in 1995. Last previous edition approved in ~~2017~~2020 as ~~D5772 – 17~~.D5772 – 20. DOI: ~~10.1520/D5772-20.~~10.1520/D5772-21.

[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.

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

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
D6751 Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels

2.2 *Energy Institute Standard:*
IP 219 Test Method for Cloud Point of Petroleum Products[3]

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.

3.1.2 *biodiesel ~~blend,~~ blend (BXX), n*—~~blend of biodiesel fuel with diesel fuels and fuel oils.~~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 for 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 also be present. The intent of the cloud point method 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 *linear 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 11.6; the specimen receptacle is described in Annex A1, and the optical system for the detection of crystal formation is described in Annex A1.

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

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 specimen into the apparatus and initiation of the program, the prescribed specimen test cell (Annex A1) is heated and then linearly cooled at a specified rate (11.6). The specimen is continuously monitored by an opposing optical light barrier (Annex A1 and Fig. A1.3) for the crystal structure formation. The temperature, when the crystallization of the wax in the specimen is detected by the optical barrier, is recorded with a resolution of 0.1 °C. The specimen is then heated to the original starting temperature.

5. Significance and Use

5.1 For petroleum products and diesel fuels, the cloud point is an index of the lowest temperature of its 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.

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

6. Apparatus

6.1 *Automatic Apparatus*[4]—The automatic cloud point apparatus described in Annex A1 consists of a microprocessor-controlled measuring unit that is capable of heating, cooling, optically observing the appearance of the automatic cloud point, and recording the temperature of the specimen. The apparatus shall be equipped with a digital display, cooling/heating block assembly, optical light barrier assembly, and a test cell of the approximate dimensions listed in Annex A1, and contain a temperature measuring device.

6.2 *Beaker,* glass or plastic (disposable) (50 mL), for containing the sample prior to introduction into the test cell.

6.3 *Circulating Bath,* a refrigeration unit equipped with a circulating pump capable of maintaining a temperature at least 20 °C lower than the lowest expected cloud point to be measured.

7. Reagents and Materials

7.1 *Cleaning Solvents,* suitable for cleaning and drying the test cell, such as petroleum naphtha and acetone. (**Warning—** Flammable. Liquid causes eye burns. Vapor is harmful. May be fatal or cause blindness if swallowed or inhaled.)

7.2 *Methyl Alcohol,* anhydrous, for use as cooling medium in circulating bath.

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

[4] The sole source of supply of the Herzog Model SC 815 and SC 819 known to the committee at this time is Walter Herzog, Lauda, Germany. 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.

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 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. 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.

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

9.3 Prepare the refrigerated circulating bath for operation in accordance with the manufacturer's instructions and allow it to attain a temperature at least 20 °C lower than the expected cloud point of the sample.

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 Pour at least 20 mL of the sample into the beaker. When necessary, heat the sample in a water bath or oven until it is just sufficiently fluid to pour into the beaker.

11.2 Place the beaker under the entrance of the test cell and start the test in accordance with the manufacturer's instructions.

11.3 When the expected cloud point is known to be above 25 °C, preselect a starting temperature which is at least 14 °C above the expected cloud point. The highest starting temperature that can be programmed is 70 °C.

11.4 When the expected cloud point is known to be at or below 25 °C, the test duration can be shortened by preselecting a starting temperature which is at least 14 °C above the expected cloud point. The lowest starting temperature that can be programmed is 20 °C.

11.5 When the expected cloud point is not known, preselect a starting temperature of 40 °C. The apparatus automatically heats the test specimen to approximately 40 °C when a starting temperature is not selected. When the expected cloud point is not known and the sample needs to be heated before transferring into the test specimen beaker, preselect a starting temperature of 70 °C.

11.6 A portion of the sample is drawn into the test cell. Initially, the specimen is heated to 40 °C or to a starting temperature preselected by the operator between 20 °C and 70 °C. The flow of the cooling fluid from the circulating bath is then regulated to maintain the rate of cooling of the specimen at 1 °C ± 0.2 °C per min (see Note 2). The specimen is continuously monitored by the optical light barrier and the specimen temperature is continuously displayed. At the detection of the automatic cloud point, the temperature the specimen attained, to within 0.1 °C resolution, is held on the digital display until cleared by the operator. The specimen is then heated to 40 °C or to the preselected starting temperature.

NOTE 2—Although the apparatus is capable of faster cooling rates of 2 °C or 3 °C/min, the interlaboratory program does not address the equivalency and precision of results using these cooling rates.

11.7 Clean the test cell by flushing solvents through the cell and then dry with clean air according to the manufacturer's instructions.

NOTE 3—Some apparatus is capable of automatic cleaning of the test cell and automatic sample changing.

12. Report

12.1 Report the temperature recorded in 11.6 as the automatic cloud point Test Method D5772.

12.2 When specified, round the temperature recorded in 11.6 to the next lower integer and report as the Test Method D2500 equivalent cloud point in accordance with Test Method D5772.

13. Precision and Bias

13.1 *Precision*—The precision of this test method as determined by the statistical examination of the interlaboratory test results[5,6] 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 1.3 °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 the normal and correct operation of this test method, exceed 3.3 °C only in one case in twenty.

13.1.3 The precision statements were derived from a 1997 interlaboratory cooperative test program.[6] 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. Five laboratories participated with the automatic apparatus and eight laboratories participated with the manual Test Method D2500/IP 219 test method. Information on the type of samples and their average cloud points are in the research report.[5]

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 to be small enough (−0.67 °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 Biodiesel in Diesel Blends*—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 0.7 °C only in one case in twenty.

13.4.2 *Reproducibility for Biodiesel in Diesel Blends*—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 2.2 °C only in one case in twenty.

[5] Supporting data (the results of the 1990 interlaboratory cooperative test program) 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.

[6] 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:D02-1509. Contact ASTM Customer Service at service@astm.org.