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.

Designation: D7397 – 21

# Standard Test Method for Cloud Point of Petroleum Products and Liquid Fuels (Miniaturized Optical Method)[1]

This standard is issued under the fixed designation D7397; 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 and biodiesel fuels that are transparent in layers 40 mm in thickness by an automatic instrument.

1.2 This test method covers the range of temperatures from –60 °C to +20 °C with temperature resolution of 0.1 °C; however, the range of temperatures included in the 2006 interlaboratory cooperative test program only covered the temperature range of –35 °C to +12 °C. See Section 13.

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

D5773 Test Method for Cloud Point of Petroleum Products and Liquid Fuels (Constant Cooling Rate Method)

D6751 Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels

2.2 *Energy Institute Standards:*[3]

IP 219 Test Method for Cloud Point of Petroleum Products

IP 446 Test Method for Cloud Point of Petroleum Products

## 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 (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 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

[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

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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.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *D2500/IP 219 equivalent cloud point, n*—temperature of a specimen, in integers, calculated by applying a bias and rounding the result to the next lower integer (see 12.2).

3.2.1.1 *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 (see 12.2). Some apparatus can perform this calculation automatically.

3.2.2 *automatic cloud point, n*—temperature of a specimen, when the appearance of the cloud is determined under the conditions of this test method.

3.2.2.1 *Discussion*—The cloud point in this test method is determined by an automatic instrument using a miniaturized test receptacle equipped with two optical fibers, one to bring light into the test receptacle and the other to receive light scattered from the specimen.

3.2.3 *miniaturized optical method, n—in cloud point test methods*, test procedure using prescribed cooling rate, specimen receptacle, and optical system for detection of crystal formation.

3.2.3.1 *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.

3.2.4 *Peltier device, n*—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.[4]

## 4. Summary of Test Method

4.1 A specimen is cooled by a Peltier device in a miniaturized specimen receptacle (A1.1.1) at a rate of 30 °C ± 5 °C⁄min, while continuously being illuminated by a light source (A1.1.4). The specimen is continuously monitored by an optical detector (A1.1.5) for the first appearance of a cloud of wax crystals. Once crystals are first detected, as manifested by an increase in scattered light level received by the optical detector, the specimen is warmed at a rate of 15 °C ± 5 °C⁄min. As soon as all the crystals have re-dissolved into the liquid specimen, warming is halted and the specimen is cooled again; but this time at a slower rate of 6 °C ± 3 °C⁄min. When crystals first appear under this slower cooling rate, the temperature of the specimen is recorded to 0.1 °C resolution as cloud point.

## 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 D5773/IP 446. The temperature results of this test method have been found to be warmer than those of Test Method D2500/IP 219 by an average of 0.49 °C; however, no sample specific bias was observed.

5.5 Similar to Test Method D5773/IP 446, this test method determines cloud point in a shorter period of time than Test Method D2500/IP 219.

NOTE 1—In cases of samples with cloud points near ambient temperatures, time savings may not be realized.

NOTE 2—This test method eliminates most of the operator time required of Test Method D2500/IP 219.

NOTE 3—The only utility required by the apparatus described in this test method is electricity with power consumption of approximately 20 W. The electric power can come from an alternating current source (wall receptacle) or direct current source such as a battery or a cigarette lighter plug in a vehicle.

NOTE 4—The apparatus described by this test method can be made much smaller and lighter than that of Test Methods D5773/IP 446 and D2500/IP 219, allowing full portability.

NOTE 5—The apparatus used in the 2006 interlaboratory study weighed approximately 1 kg and occupied the space of a small lunch box. See Section 13.

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## 6. Apparatus

6.1 *Automatic Apparatus*[4,5]—The automatic cloud point apparatus described in this test method consists of a test chamber 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 receptacle, optical detector, light source, optical fibers, 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 rate of 3 °C to 35 °C⁄min.

[4] The Peltier device is covered by a patent. Interested parties are invited to submit information regarding the identification of an alternative(s) to this patented item to the ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[5] The sole source of supply of the apparatus known to the committee at this time is Phase Technology Miniature Cloud Point Analyzer available from Phase Technology, 11168 Hammersmith Gate, Richmond, B.C., Canada V7A-5H8. 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.

6.4 The temperature-measuring device in the specimen receptacle shall be capable of measuring the temperature of the test specimen from –60 °C to +20 °C at a resolution of 0.1 °C.[4]

NOTE 6—The apparatus described above is covered by patents. If you are aware of an alternative(s) to the patented items, please attach to your ballot return a description of the alternative(s). All suggestions will be considered by the committee.

## 7. Reagents and Materials

7.1 Disposable syringe that is capable of dispensing at least 10 mL ± 0.5 mL per full discharge of sample into the specimen receptacle.

NOTE 7—The apparatus can also be connected to a sample supply line to receive new sample. The amount of sample required per analysis is the same as that for the syringe injection procedure (that is, 20 mL ± 1.0 mL per analysis). In such cases, a disposable syringe would not be needed.

## 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 specimen is perfectly clear, but make such filtration at a temperature at least 14 °C above the expected cloud point.

NOTE 8—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.

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## 9. Preparation of Apparatus

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

9.2 Turn on the main power switch of the analyzer.

## 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 Draw 10 mL ± 0.5 mL of bubble-free sample into a new disposable syringe. Connect the syringe to the inlet port, and inject the full charge of sample into the test receptacle. The specimen excess will flow into a waste-receiving container.

11.2 Draw another 10 mL ± 0.5 mL of bubble-free sample into the syringe. Connect the syringe to the inlet port, and inject the full charge of sample into the test receptacle. The specimen excess will flow into a waste-receiving container. The total amount of sample (that is, 20 mL ± 1.0 mL) is sufficient in quantity to flush out any previous sample in the specimen receptacle and fill it with the fresh sample.

**TABLE 1 Typical Repeatability at Different Cloud Points**

| Cloud Point °C | Repeatability °C |
|---|---|
| 15 | 0.4 |
| 10 | 0.5 |
| 5 | 0.7 |
| 0 | 0.8 |
| -5 | 0.9 |
| -10 | 1.1 |
| -15 | 1.2 |
| -20 | 1.3 |
| -25 | 1.5 |
| -30 | 1.6 |
| -35 | 1.7 |

NOTE 9—Follow manufacturer's instructions for sample injection if the specimen receptacle is connected to a sample supply line as described in Note 7.

11.3 Start the operation of the apparatus according to the manufacturer's instructions. From this point on, the apparatus automatically controls the procedure.

11.4 Cool the sample at a rate of 30 °C ± 5 °C/min, while continuously illuminating the sample with the light source. Monitor the specimen continuously with the optical detector. Once crystals are first detected, as manifested by an increase in light level received by the optical detector, warm the specimen at a rate of 15 °C ± 5 °C/min. As soon as all the crystals have re-dissolved into the liquid specimen in accordance with this test method, stop the warming and cool the specimen again at a slower rate of 6 °C ± 3 °C/min. When crystals first appear under this slower cooling rate, record the temperature of the specimen as the cloud point.

11.5 The measurement is automatically terminated once the cloud point is detected.

11.6 When the measurement is complete, the cloud point value per Test Method D7397 will be displayed by the apparatus.

## 12. Calculation or Interpretation of Results

12.1 Report the temperature recorded in 11.6 as the automatic cloud point Test Method D7397.

12.2 When specified, correct the temperature recorded in 11.6 with the relative bias in accordance with 13.3, round the result to the next lower integer (a colder temperature), and report as the Test Method D2500/IP 219 equivalent cloud point per Test Method D7397.

## 13. Precision and Bias[6]

13.1 *Precision*—The precision of this test method as determined by the statistical examination of the interlaboratory test results is as follows:

[6] Supporting data (the results of the 2006 Interlaboratory Cooperative Test Program) have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1627. Contact ASTM Customer Service at service@astm.org.

TABLE 2 Typical Reproducibility at Different Cloud Points

| Cloud Point °C | Reproducibility °C |
|---|---|
| 15 | 0.5 |
| 10 | 0.7 |
| 5 | 0.9 |
| 0 | 1.1 |
| -5 | 1.2 |
| -10 | 1.4 |
| -15 | 1.6 |
| -20 | 1.7 |
| -25 | 1.9 |
| -30 | 2.1 |
| -35 | 2.2 |

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:

$$0.26E - 01 \times (31.0 - X)°C$$

where $X$ = cloud point in °C only in one case in twenty. See Table 1.

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 normal and correct operation of this test method, exceed:

$$0.34E - 01 \times (31.0 - X)°C$$

where $X$ = cloud point in °C) only in one case in twenty. See Table 2.

13.1.3 The precision statements were derived from a 2006 interlaboratory cooperative test program.[6] Participants analyzed blind replicates of 13 sample sets comprised of 4 petroleum distillates, 3 biodiesels (derived from soy, tallow and yellow grease) and 6 blends of biodiesels in petroleum distillates representing B5, B10, and B20 blends. The cloud point ranges from –35 °C to +12 °C. A total of 20 laboratories participated in this study with seven laboratories each for the miniaturized cloud point method and D5773/IP 446. Six laboratories participated with Test Method D2500/IP 219 apparatus. Information on the type of sample and their average cloud points are in the research report.

13.2 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*—There was a small but statistically significant bias relative to Test Method D2500/IP 219 of –0.49°C:

$$Y(D2500) = X(D7397) - 0.49°C \quad (1)$$

13.3.1 There is no bias relative to Test Method D5773/IP 446.

## 14. Keywords

14.1 automatic could point; cloud point; miniaturized cloud point apparatus; Peltier; petroleum products; thermoelectric wax crystals

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## ANNEX

(Mandatory Information)

### A1. DETAILED DESCRIPTION OF APPARATUS

A1.1 *Test Chamber*, comprised of light source, optical fibers, detector, specimen receptacle, temperature sensor, Peltier device, and heat sink arranged in a configuration as shown in Fig. A1.1. The working principle is illustrated in Fig. A1.2. The test chamber is self-cleaning with the introduction of new specimen.

A1.1.1 *Specimen receptacle,* comprising a disc (6.0 mm ± 0.1 mm diameter and 2.0 mm ± 0.2 mm height of highly reflective material. The surfaces serve to reflect light. The transfer of heat to and away from the specimen is controlled by the Peltier device.

A1.1.2 *Temperature Sensor,* reading to 0.1 °C, permanently embedded into the bottom of the specimen receptacle. This temperature sensor, which is made of a single strand of platinum, provides accurate measurement of the specimen temperature.

A1.1.3 *Peltier Device,* capable of controlling the specimen temperature over a wide range. During specimen cooling, heat is transferred from the top of the device to the bottom. Since the top is in thermal contact with the specimen cup, the specimen will be chilled. The bottom of the Peltier device is in thermal contact with the heat sink where heat is dissipated. During specimen warming, the reverse process will take place.

A1.1.4 *Light Source,* to provide a beam of light with a wavelength to which the optical detector is sensitive. An optical fiber is used to transmit light from the light source to the specimen receptacle.

A1.1.5 *Optical Detector,* to receive light from an optical fiber that is positioned in the specimen receptacle in such a way that it can capture light scattered from the crystals inside the specimen. As shown in Fig. A1.2, this fiber is placed at a 90° angle with respect to the light emitting fiber described in A1.1.4. This spatial relationship was found to be sensitive in