Designation: D 7397 – 07

An American National Standard

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

This standard is issued under the fixed designation D 7397; 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 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 to +12°C. See Section 13.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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]

D 2500 Test Method for Cloud Point of Petroleum Products

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

D 5773 Test Method for Cloud Point of Petroleum Products (Constant Cooling Rate Method)

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 comprising 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 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 (BXX)*, *n*—blend of biodiesel fuel with petroleum-based diesel fuel designated BXX, where XX is the volume percentage (as a whole number without the percentage sign) of biodiesel.

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

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *miniaturized optical method*, *n*—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 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.

3.2.2 *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

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.07.0D on Wax-Related Viscometric Properties of Fuels and Oils.

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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 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 at a rate of 30 ± 5°C/min, while continuously being illuminated by a light source. The specimen is continuously monitored by an optical detector for the first appearance of a cloud of wax crystals. Once crystals are first detected, as manifested by an increase in light level received by the optical detector, the specimen is warmed at a rate of 15 ± 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 ± 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 D 5773/IP 446. The temperature results of this test method have been found to be warmer than those of Test Method D 2500/IP 219 by an average of 0.49°C; however, no sample specific bias was observed.

5.5 Similar to Test Method D 5773/IP 446, this test method determines cloud point in a shorter period of time than Test Method D 2500/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 D 2500/IP 219.

NOTE 3—The only utility required by the apparatus described in this test method is electricity with power consumption of approximately 20 watts. 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 D 5773/IP 446 and D 2500/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.

[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.

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 to 35°C/min.

6.4 The temperature measuring device in the specimen receptacle shall be capable of measuring the temperature of the test specimen from –60 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 ± 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 ± 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 Practices D 4057 or D 4177.

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.

9. Preparation of Apparatus

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

[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.

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

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

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 ± 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 ± 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 ± 1.0 mL) is sufficient in quantity to flush out any previous sample in the specimen receptacle and fill it with the fresh sample.

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 ± 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 ± 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 ± 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 D 7397 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 D 7397.

12.2 When specified, round the temperature recorded in 11.6 to the next lower integer, correct the results with the relative bias per 13.3 and report as the Test Method D 2500/IP 219 equivalent cloud point per Test Method D 7397.

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:

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 to + 12°C. A total of 20 laboratories participated in this study with 7 laboratories each for the miniaturized cloud point method and D 5773/IP 446. Six laboratories participated with Test Method D 2500/IP 219 apparatus. Information on the type of sample and their average cloud points are in the research report.

[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.