



Designation: **D 2500 – 9902**

An American National Standard
British Standard 4458

Designation: ~~219/94~~ **219/82**

Standard Test Method for Cloud Point of Petroleum Products¹

This standard is issued under the fixed designation D 2500; 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. ~~This test method was adopted as an ASTM-IP Standard.~~

This standard has been approved for use by agencies of the Department of Defense.

1. Scope*

1.1 This test method covers only petroleum products and biodiesel fuels that are transparent in layers 40 mm in thickness, and with a cloud point below 49°C.

NOTE 1—The interlaboratory program consisted of petroleum products of Test Method D 1500 color of 3.5 and lower. The precisions stated in this test method may not apply to samples with ASTM color higher than 3.5.

1.2 *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.* For specific hazard statements, see ~~Notes 3–6.~~ Section 7.

2. Referenced Documents

2.1 ASTM Standards:

D 1500 Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)²

¹ This test method is under the jurisdiction of ASTM Committee ~~D-2~~ D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.07 on Flow Properties.

Current edition approved ~~Dec. April 10, 1999; 2002.~~ Published ~~January 2000; July 2002.~~ Originally published as D 2500–66. Last previous edition ~~D 2500–98a; D 2500–99.~~

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

E 1 Specification for ASTM Thermometers³

2.2 *IP Standard:*

Specifications for IP Standard Thermometers⁴

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *cloud point, n—in petroleum products and biodiesel fuels*, the temperature of a liquid specimen when the smallest observable cluster of wax crystals first appears upon cooling under prescribed conditions.

3.1.1.1 *Discussion*—To many observers, the cluster of wax crystals looks like a patch of whitish or milky cloud, hence the name of the test method. The cloud appears when the temperature of the specimen is low enough to cause wax crystals to form. For many specimens, the crystals first form at the lower circumferential wall of the test jar where the temperature is lowest. The size and position of the cloud or cluster at the cloud point varies depending on the nature of the specimen. Some samples will form large, easily observable, clusters, while others are barely perceptible.

3.1.1.2 *Discussion*—Upon cooling to temperatures lower than the cloud point, clusters of crystals will grow in multiple directions; for example, around the lower circumference of the test jar, towards the center of the jar, or vertically upwards. The crystals can develop into a ring of cloud along the bottom circumference, followed by extensive crystallization across the bottom of the test jar as temperature decreases. Nevertheless, the cloud point is defined as the temperature at which the crystals first appear, not when an entire ring or full layer of wax has been formed at the bottom of the test jar.

3.1.1.3 *Discussion*—In general, it is easier to detect the cloud point of samples with large clusters that form quickly, such as paraffinic samples. The contrast between the opacity of the cluster and the liquid is also sharper. In addition, small brightly-reflective spots can sometimes be observed inside the cluster when the specimen is well illuminated. For other more difficult samples, such as naphthenic, hydrocracked, and those samples whose cold flow behavior have been chemically altered, the appearance of the first cloud can be less distinct. The rate of crystal growth is slow, the opacity contrast is weak, and the boundary of the cluster is more diffuse. As the temperature of these specimens decrease below the cloud point, the diffuse cluster will increase in size and can form a general haze throughout. A slight haze throughout the entire sample, which slowly becomes more apparent as the temperature of the specimen decreases, can also be caused by traces of water in the specimen instead of crystal formation (see Note 4). With these difficult samples, drying the sample prior to testing can eliminate this type of interference.

3.1.2 *biodiesel, n—a fuel comprised of mono-alkyl esters of long chain fatty acids derived from vegetable oils or animal fats, designated B100.*

3.1.2.1 *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).

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

4. Summary of Test Method

4.1 The specimen is cooled at a specified rate and examined periodically. The temperature at which a cloud is first observed at the bottom of the test jar is recorded as the cloud point.

5. Significance and Use

5.1 For petroleum products and biodiesel fuels, cloud point of a petroleum product is an index of the lowest temperature of its their utility for certain applications.

6. Apparatus (Ssee Fig. 1)

6.1 *Test Jar*, clear, cylindrical glass, flat bottom, 33.2 to 34.8-mm outside diameter and 115 and 125-mm height. The inside diameter of the jar may range from 30 to 32.4 mm within the constraint that the wall thickness be no greater than 1.6 mm. The jar should be marked with a line to indicate sample height 54 ± 3 mm above the inside bottom.

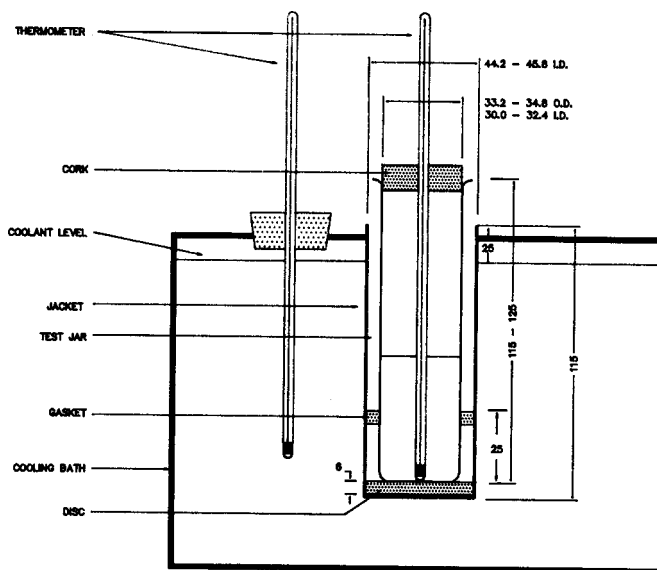
6.2 *Thermometers*, having ranges shown below and conforming to the requirements as prescribed in Specification E 1 or Specifications for IP Standard Thermometers.

Thermometer	Temperature Range	ASTM	Thermometer Number	IP
High cloud and pour	-38 to +50°C	5C		1C
Low cloud and pour	-80 to +20°C	6C		2C

² Annual Book of ASTM Standards, Vol 05.01.

³ Annual Book of ASTM Standards, Vol 14.03.

⁴ Available from Institute for Petroleum, 61 New Cavendish St., London, England W1M 8AR.



NOTE 1—All dimensions in millimetres.
FIG. 1 Apparatus for Cloud Point Test

6.3 *Cork*, to fit the test jar, bored centrally for the test thermometer.

6.4 *Jacket*, metal or glass, watertight, cylindrical, flat bottom, about 115 mm in depth, with an inside diameter of 44.2 to 45.8 mm. It shall be supported free of excessive vibration and firmly in a vertical position in the cooling bath of 6.7 so that not more than 25 mm projects out of the cooling medium.

6.5 *Disk*, cork or felt, 6-mm thick to fit loosely inside the jacket.

6.6 *Gasket*, ring form, about 5 mm in thickness, to fit snugly around the outside of the test jar and loosely inside the jacket. The gasket may be made of rubber, leather, or other material that is elastic enough to cling to the test jar and hard enough to hold its shape. Its purpose is to prevent the test jar from touching the jacket.

6.7 *Bath or Baths*, maintained at prescribed temperatures with a firm support to hold the jacket vertical. The required bath temperatures may be maintained by refrigeration if available, otherwise by suitable freezing mixtures.

NOTE 2—The mixtures commonly used for temperatures down to those shown are as follows:

Ice and water	10°C
Crushed ice and sodium chloride crystals	-12°C
Crushed ice and calcium chloride crystals	-26°C
Acetone, methyl or ethyl alcohol, or petroleum naphtha—chilled in a covered metal beaker with an ice-salt mixture to -12°C, then with enough solid carbon dioxide to give the desired temperature	-57°C
Acetone, methyl or ethyl alcohol, or petroleum naphtha chilled in a covered metal beaker with an ice-salt mixture to -12°C, then with enough solid carbon dioxide to give the desired temperature	-57°C

7. Reagents and Materials

7.1 *Acetone*—Technical grade acetone is suitable for the cooling bath, provided it does not leave a residue on drying. (**Warning**—Extremely flammable.)

7.2 *Calcium Chloride*—Commercial or technical grade calcium chloride is suitable.

7.3 *Carbon Dioxide (Solid) or Dry Ice*—A commercial grade of dry ice is suitable for use in the cooling bath.

7.4 *Ethanol or Ethyl Alcohol*—A commercial or technical grade of dry ethanol is suitable for the cooling bath. (**Warning**—Flammable. Denatured, cannot be made nontoxic.)

7.5 *Methanol or Methyl Alcohol*—A commercial or technical grade of dry methanol is suitable for the cooling bath. (**Warning**—Flammable. Vapor harmful.)

7.6 *Petroleum Naphtha*—A commercial or technical grade of petroleum naphtha is suitable for the cooling bath. (**Warning**—Combustible. Vapor harmful.)

7.7 *Sodium Chloride Crystals*—Commercial or technical grade sodium chloride is suitable.

7.8 *Sodium Sulfate*—A reagent grade of anhydrous sodium sulfate should be used when required (see Note 4).

8. Procedure

8.1 Bring the sample to be tested to a temperature at least 14°C above the expected cloud point. Remove any moisture present

by a method such as filtration through dry lintless filter paper until the oil is perfectly clear, but make such filtration at a temperature of at least 14°C above the approximate cloud point.

8.2 Pour the sample into the test jar to the level mark.

8.3 Close the test jar tightly by the cork carrying the test thermometer. Use the **H** high **C** cloud and **P** pour **T** thermometer if the expected cloud point is above –36°C and the **L** low **C** cloud and **P** pour **T** thermometer if the expected cloud point is below –36°C. Adjust the position of the cork and the thermometer so that the cork fits tightly, the thermometer and the jar are coaxial, and the thermometer bulb is resting on the bottom of the jar.

NOTE 3—Liquid column separation of thermometers occasionally occurs and may escape detection. Thermometers should be checked ~~immediately prior to the test~~ periodically and used only if their ice points are $0 \pm 1^\circ\text{C}$, when the thermometer is immersed to the immersion line in an ice bath, and when the emergent column temperature does not differ significantly from 21°C. Alternatively, immerse the thermometer to a reading and correct for the resultant cooler stem temperature.

8.4 See that the disk, gasket, and the inside of the jacket are clean and dry. Place the disk in the bottom of the jacket. The disk and jacket shall have been placed in the cooling medium a minimum of 10 min before the test jar is inserted. The use of a jacket cover while the empty jacket is cooling is permitted. Place the gasket around the test jar, 25 mm from the bottom. Insert the test jar in the jacket. Never place a jar directly into the cooling medium.

NOTE 4—Failure to keep the disk, gasket, and the inside of the jacket clean and dry may lead to frost formation, which may cause erroneous results.

8.5 Maintain the temperature of the cooling bath at $0 \pm 1.5^\circ\text{C}$.

8.6 At each test thermometer reading that is a multiple of 1°C, remove the test jar from the jacket quickly but without disturbing the specimen, inspect for cloud, and replace in the jacket. This complete operation shall require not more than 3 s. If the oil does not show a cloud when it has been cooled to 9°C, transfer the test jar to a jacket in a second bath maintained at a temperature of $-18 \pm 1.5^\circ\text{C}$ (see Table 1). Do not transfer the jacket. If the specimen does not show a cloud when it has been cooled to –6°C, transfer the test jar to a jacket in a third bath maintained at a temperature of $-33 \pm 1.5^\circ\text{C}$. For the determination of very low cloud points, additional baths are required, each bath to be maintained in accordance with Table 1. In each case, transfer the jar to the next bath, if the specimen does not exhibit cloud point and the temperature of the specimen reaches the lowest specimen temperature in the range identified for the current bath in use, based on the ranges stated in Table 1.

8.7 Report the cloud point, to the nearest 1°C, at which any cloud is observed at the bottom of the test jar, which is confirmed by continued cooling.

NOTE 5—A wax cloud or haze is always noted first at the bottom of the test jar where the temperature is lowest. A slight haze throughout the entire sample, which slowly becomes more apparent as the temperature is lowered, is usually due to traces of water in the oil. Generally this water haze will not interfere with the determination of the wax cloud point. In most cases of interference, filtration through dry lintless filter papers, such as described in 8.1, is sufficient. In the case of diesel fuels, however, if the haze is very dense, a fresh portion of the sample should be dried by shaking 100 mL with 5 g of anhydrous sodium sulfate for at least 5 min and then filtering through dry lintless 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 be done always at a temperature at least 14°C above the approximate cloud point but otherwise not in excess of 49°C.

9. Report

9.1 Report the temperature recorded in 8.7 as the **C** cloud **P** point, Test Method D 2500.

10. Precision and Bias

10.1 The precision of this test method as determined by statistical examination of interlaboratory results is as follows:

10.1.1 Repeatability—The difference between two test results, obtained by the same operator with 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 2°C only in ~~one~~ 1 case in ~~twenty~~.

~~10.3–20.~~

10.1.2 Reproducibility—The difference between two single and independent 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 4°C only in ~~one~~ 1 case in 20.

TABLE 1 Bath and Sample Temperature Ranges

Bath	Bath Temperature Setting, °C	Sample Temperature Range, °C
1	0 ± 1.5	Start to 10
2	-18 ± 1.5	9 to –6
3	-33 ± 1.5	–6 to –24
4	-51 ± 1.5	–24 to –42
5	-69 ± 1.5	–42 to –60

10.1.3 The precision statements were derived from a 1990 interlaboratory cooperative test program.⁵ Participants analyzed 13 sample sets comprised of various distillate fuels and lubricating oils with temperature range from -1 to -37°C. Eight laboratories participated with the manual D 2500/IP219 test method. Information on the type of samples and their average cloud points are in the research report.

10.42 *Bias*—The procedure in this test method has no bias, because the value of cloud point can be defined only in terms of a test method.

10.53 *Precision for Biodiesel Products*⁶—The precision of this test method as determined by statistical examination of interlaboratory results is as follows:

10.3.1 *Repeatability for Blends of Biodiesel in Diesel*—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 2°C only in 1 case in 20.

10.3.2 *Reproducibility for Blends of Biodiesel in Diesel*—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°C only in 1 case in 20.

NOTE 6—The precision for blends of biodiesel in diesel samples comprised cloud points from about -2 to +10°C.

10.3.3 The precision statements were derived from a 1990 2001 interlaboratory cooperative test program.⁶ Participants analyzed 13 eleven sample sets comprised of various different blends of two petroleum distillate fuels, diesel and lubricating oils kerosene, with various biodiesel fuels with temperature range from -1 +10 to -37°C. Eight -45°C. Ten laboratories participated with the manual D 2500/IP219 test method. Information on the type of samples and their average cloud points are in the research report.

NOTE 7—One of the outcomes of the interlaboratory study was the selection of the sample types, since the ones used in the study contributed to a difficulty in determining the precision statement. Kerosene is a sufficiently different fuel type from biodiesel to cause some slight separation of phases upon cooling when in B20 blends. Also, the particular kerosene sample used was atypical, which complicated the study further. Therefore, data from the blends of kerosene in biodiesel were not used in the precision statement. In addition, the diesel fuel used in the research report was high cloud point material. Due to the cloud point of the base diesel material, this temperature range in the precision statement was limited.

NOTE 8—A future interlaboratory cloud study will be done to include a wider range of base biodiesel fuels with various distillate blend stocks.

10.4 *Bias for Biodiesel Products*⁶—The procedure in this test method has no bias, because the value of cloud point can be defined only in terms of a test method.

11. Keywords

11.1 cloud point; petroleum products; wax crystals

⁶ Available from

⁵ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1444.

⁶ Supporting data (the results of the 2001 interlaboratory cooperative test program) have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1524.

SUMMARY OF CHANGES

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D 2500-99) that may impact the use of this standard.

- (1) Added biodiesel fuels to Scope.
- (2) Added biodiesel fuels to the definition for cloud point.
- (3) Added new definitions for biodiesel and biodiesel blends to Terminology.
- (4) Added biodiesel fuels to Significance and Use.
- (5) Changed Note 3 to indicate that “thermometers should be checked periodically” instead of “immediately prior to the test.”
- (6) Added precision and bias for biodiesel fuels.

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).