

Designation: D 2500 – 02<sup>€1</sup>

An American National Standard British Standard 4458

THE INSTITUTE OF PETROLEUM

Designation: 219/82

# Standard Test Method for Cloud Point of Petroleum Products<sup>1</sup>

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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

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

 $\epsilon^1$  Note—Table 1 was editorially corrected in August 2004.

# 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 Section 7.

#### 2. Referenced Documents

2.1 ASTM Standards:

- D 1500 Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)<sup>2</sup>
- E 1 Specification for ASTM Thermometers<sup>3</sup>
- 2.2 IP Standard:

Specifications for IP Standard Thermometers<sup>4</sup>

#### 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).

Copyright © ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States.

<sup>&</sup>lt;sup>1</sup> 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 on Flow Properties.

Current edition approved April 10, 2002. Published July 2002. Originally published as D 2500–66. Last previous edition D 2500–99.

<sup>&</sup>lt;sup>2</sup> Annual Book of ASTM Standards, Vol 05.01.

<sup>&</sup>lt;sup>3</sup> Annual Book of ASTM Standards, Vol 14.03.

<sup>&</sup>lt;sup>4</sup> Available from Institute for Petroleum, 61 New Cavendish St., London, England WIM 8AR.

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.

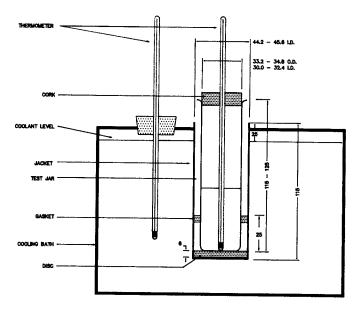
## 5. Significance and Use

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

## 6. Apparatus (see 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  $54 \pm 3$  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.



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

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

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	–57°C
chilled in a covered metal beaker with an ice-salt mix-	
ture to -12°C, then with enough solid carbon dioxide to	
give the desired temperature	

## 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