Designation: D 5551 – 95 (Reapproved 2001)

Standard Test Method for Determination of the Cloud Point of Oil[1]

This standard is issued under the fixed designation D 5551; 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 oils used in the softening and stuffing of leather and in the manufacture of fat liquors and other softening and stuffing compounds. This test method was derived from Test Methods D 97 and D 2500.

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

1.3 *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:*

D 97 Test Methods for Pour Point of Petroleum Oils[2]

D 2500 Test Method for Cloud Point of Petroleum Oils[2]

E 1 Specification for ASTM Thermometers[3]

3. Terminology

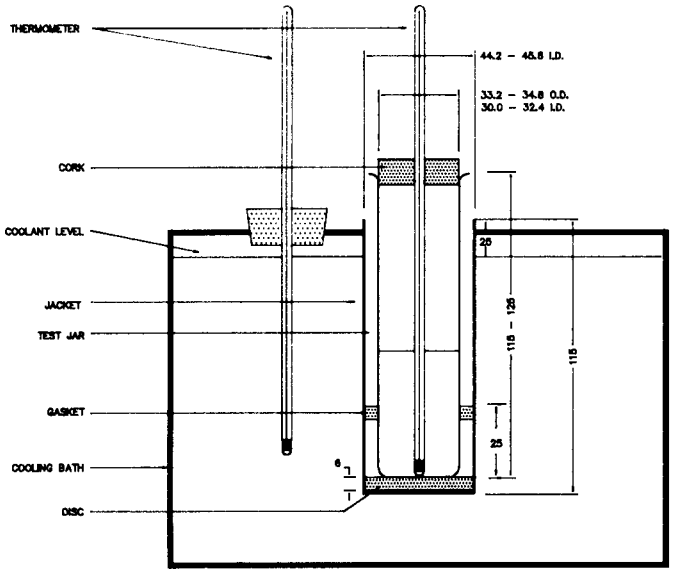
3.1 *cloud point*—that temperature, expressed as a multiple of 1°C (2°F), at which a cloud or haze of wax crystals appears at the bottom of the test jar when the oil is cooled under prescribed conditions.

4. Significance and Use

4.1 This test method is intended to determine the cloud point of oils used in the softening and stuffing of leather, as well as those used in the manufacture of products for such purpose. The cloud point of oils is measured for the purpose of quality assurance.

5. Apparatus

5.1 *Test Jar*, clear cylindrical glass, flat bottom, 30- to 33.5-mm inside diameter, and 115- to 125-mm height. To indicate sample height the jar should be marked with a line 54 ± 3 mm above the inside bottom.



NOTE 1—All dimensions in millimetres.

FIG. 1 Apparatus for Cloud Point Test

5.2 *Thermometers*, having ranges shown below and conforming to the requirements prescribed in Specification E 1 for thermometers:

Thermometer	Temperature Range	Thermometer Number ASTM	Thermometer Number IP
High cloud and pour	−38 to +50°C	5C	1C
Low cloud and pour	−80 to +20°C	6C	2C
Melting point	+32 to +127°C	61C	63C

5.2.1 Since separation of liquid column thermometers occasionally occurs and may escape detection, thermometers should be checked immediately prior to the test and used only if they prove accurate within ± 1°C (for example, ice point).

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

5.4 *Jacket*, metal or glass, watertight, cylindrical, flat bottom, 115 mm in depth, 42- to 50-mm inside diameter. It must be supported firmly in a vertical position in the cooling bath of 5.7 so that not more than 25 mm projects out of the cooling medium.

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

5.6 *Gasket*, to fit snugly around the outside of the test jar and loosely inside the jacket. The gasket may be made of

[1] This test method is under the jurisdiction of ASTM Committee D31 on Leather and is the direct responsibility of Subcommittee D31.08 on Fats and Oils. This test method was developed in cooperation with the American Leather Chemists Assn. (Method H 18-1957).

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[2] *Annual Book of ASTM Standards*, Vol 05.01.

[3] *Annual Book of ASTM Standards*, Vol 14.03.

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.

5.7 *Bath or Baths*, maintained at prescribed temperatures with a firm support to hold the jacket vertical. The required bath temperatures may be obtained by refrigeration if available, otherwise by suitable freezing mixtures. Freezing mixtures commonly used for temperatures down to those shown are as follows:

	For Temperatures Down
Ice and water	9°C
Crushed ice and sodium chloride crystals	−12°C
Crushed ice and calcium chloride crystals	−27°C
Acetone or petroleum naphtha (see Section 6) 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

NOTE 1—There are automatic pour point testers available and in use that may be advantageous in the saving of test time, permit the use of smaller samples, and have other factors that may merit their use. If automatic testers are used, the user must ensure that all of the manufacturer's instructions for calibration, adjustment, and operation of the instrument are followed. It must be reported that the pour point was determined by an automatic instrument. Precision of automatic pour point testers has not been determined. In any case of dispute, the pour point, as determined by the manual method described herein, shall be considered the reference test.

6. Reagents and Materials

6.1 The following solvents of technical grade are appropriate for low-temperature bath media.

6.2 *Acetone*. (**Warning—**Extremely flammable.)

6.3 *Alcohol, Ethanol*. (**Warning—**Flammable.)

6.4 *Alcohol, Methanol*. (**Warning—**Flammable. Vapor harmful.)

6.5 *Petroleum Naphtha*. (**Warning—**Combustible. Vapor harmful.)

6.6 *Solid Carbon Dioxide*. (**Warning—**Extremely cold − 78.5°C.)

7. Procedure

7.1 Bring the oil to be tested to a temperature at least 14°C (25°F) above the approximate cloud point. Remove any moisture present by any suitable method, such as by filtration through dry lintless filter paper until the oil is perfectly clear, but make such filtration at a temperature of at least 14°C (25°F) above the approximate cloud point.

7.2 Pour the clear oil into the test jar to the level mark.

7.3 Close the test jar tightly by the cork carrying the test thermometer (Note 2). Use the high cloud and pour thermometer if the expected cloud point is above −38°C (−36°F) and the low cloud and pour thermometer if the expected cloud point is below −38°C (−36°F). 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 2—Since separation of the mercury or toluene thread of cloud and pour thermometers occasionally occurs, and since such separation may otherwise escape immediate detection, it is suggested that the ice points of the thermometers be checked immediately prior to the test. Any thermometer that shows as ice point differing from 0°C (32°F) by more than 1°C (2°F) should be further examined or recalibrated, or both, before use.

7.4 Place the disk in the bottom of the jacket. Place the ring gasket around the test jar, 25 mm (1 in.) from the bottom. The disk, gasket, and inside of the jacket shall be clean and dry. Insert the test jar in the jacket.

7.5 Maintain the temperature of the cooling bath at −1 to +2°C (30 to 35°F). Support the jacket containing the test jar firmly in a vertical position in the cooling bath so that not more than 25 mm (1 in.) of the jacket projects out of the cooling medium.

7.6 At each test thermometer reading that is a multiple of 1°C (2°F), remove the test jar from the jacket quickly but without disturbing the oil, 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 10°C (50°F), transfer the test jar to another jacket in a second bath maintained at a temperature of −18 to −15°C (0 to +5°F). (Do not transfer the jacket.) If the oil does not show a cloud when it has been cooled to −7°C (20°F), transfer the test jar to another jacket in a third bath maintained at a temperature of −34 to −32°C (−30 to −25°F). For the determination of very low cloud points, additional baths are required, each bath to be maintained at 17°C (30°F) below the temperature of the preceding bath. In each case transfer the test jar when the temperature of the oil reaches a point 28°C (50°F) above the temperature of the new bath. At no time place the cold test jar directly in the cooling medium.

7.7 When such inspection first reveals a distinct cloudiness or haze in the oil at the bottom of the test jar, record the reading of the test thermometer as the cloud point.

NOTE 3—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 7.1 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 (25°F) above the approximate cloud point but otherwise not in excess of 49°C (120°F).

8. Report

8.1 Report the temperature recorded in 7.7 as the cloud point.

9. Precision and Bias

9.1 *Precision*:

9.1.1 *Oils*—The following criteria should be used for judging the acceptability of results (95 % confidence):

9.1.1.1 *Repeatability*—Duplicate results by the same operator should be considered suspect if they differ by more than 2°C (4°F), in the case of distillate oils, and by more than 6°C (10°F) in the case of other oils.

9.1.1.2 *Reproducibility*—The results submitted by each of two laboratories should be considered suspect if the two results differ by more than 4°C (8°F), in the case of distillate oils, and by more than 6°C (10°F) in the case of other oils.

9.2 *Bias*—There being no criteria for measuring bias in these test-product combinations, no statement of bias can be made.

10. Keywords

10.1 cloud point; fat liquors; leather; oil; softening and stuffing compounds