



Designation: ~~D97-08~~ Designation: D 97 – 09

An American National Standard



Designation: 15/95

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

This standard is issued under the fixed designation D 97; 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 reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

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

### 1. Scope\*

1.1 This test method covers and is intended for use on any petroleum product.<sup>2</sup> A procedure suitable for black specimens, cylinder stock, and nondistillate fuel oil is described in 8.8. ~~A procedure for testing the fluidity of a residual fuel oil at a specified temperature is described in Appendix X1.~~ The cloud point procedure formerly part of this test method now appears as Test Method D 2500.

1.2 Currently there is no ASTM test method for automated Test Method D 97 pour point measurements.

1.3 Several ASTM test methods offering alternative procedures for determining pour points using automatic apparatus are available. None of them share the same designation number as Test Method D 97. When an automatic instrument is used, the ASTM test method designation number specific to the technique shall be reported with the results. A procedure for testing the pour point of crude oils is described in Test Method D 5853.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *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:<sup>3</sup>

D 117 [Guide for Sampling, Test Methods, and Specifications for Electrical Insulating Oils of Petroleum Origin](#)

D 396 [Specification for Fuel Oils](#) ~~D 1659 Test Method for Maximum Fluidity Temperature of Residual Fuel Oil~~

D 2500 [Test Method for Cloud Point of Petroleum Products](#) ~~D 3245 Test Method for Pumpability of Industrial Fuel Oils~~

D 5853 [Test Method for Pour Point of Crude Oils](#)

D 6300 [Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants](#)

E 1 [Specification for ASTM Liquid-in-Glass Thermometers](#)

#### 2.2 Energy Institute Standards:

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

### 3. Terminology

#### 3.1 Definitions:

3.1.1 *black oil, n*—lubricant containing asphaltic materials. Black oils are used in heavy-duty equipment applications, such as mining and quarrying, where extra adhesiveness is desired.

3.1.2 *cylinder stock, n*—lubricant for independently lubricated engine cylinders, such as those of steam engines and air compressors. Cylinder stock are also used for lubrication of valves and other elements in the cylinder area.

<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 Sept. 1, 2008; April 15, 2009. Published September 2008; April 2009. Originally approved in 1927, replacing D 47. Last previous edition approved in 2007/2008 as ~~D 97-07~~-D 97-08.

In the IP, this test method is under the jurisdiction of the Standardization Committee. This test method was adopted as a joint ASTM-IP Standard in 1965.

<sup>2</sup> Statements defining this test and its significance when applied to electrical insulating oils of mineral origin will be found in Guide D 117.

<sup>3</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>4</sup> Withdrawn.

<sup>4</sup> Methods for Analysis and Testing, *IP Standards for Petroleum and its Products*, Part I, Vol 2.

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

3.1.3 *pour point, n*—in petroleum products, the lowest temperature at which movement of the test specimen is observed under prescribed conditions of test.

3.1.4 *residual fuel, n*—a liquid fuel containing bottoms remaining from crude distillation or thermal cracking; sometimes referred to as heavy fuel oil.

3.1.4.1 *Discussion*—Residual fuels comprise Grades 4, 5, and 6 fuel oils, as defined in Specification D 396.

**4. Summary of Test Method**

4.1 After preliminary heating, the sample is cooled at a specified rate and examined at intervals of 3°C for flow characteristics. The lowest temperature at which movement of the specimen is observed is recorded as the pour point.

**5. Significance and Use**

5.1 The pour point of a petroleum specimen is an index of the lowest temperature of its utility for certain applications.

**6. Apparatus**

6.1 *Test Jar*, cylindrical, of clear glass, flat bottom, 33.2 to 34.8-mm outside diameter, and 115 to 125 mm in height. The inside diameter of the jar can range from 30.0 to 32.4 mm, within the constraint that the wall thickness be no greater than 1.6 mm. The jar shall have a line to indicate a sample height  $54 \pm 3$  mm above the inside bottom. See Fig. 1.

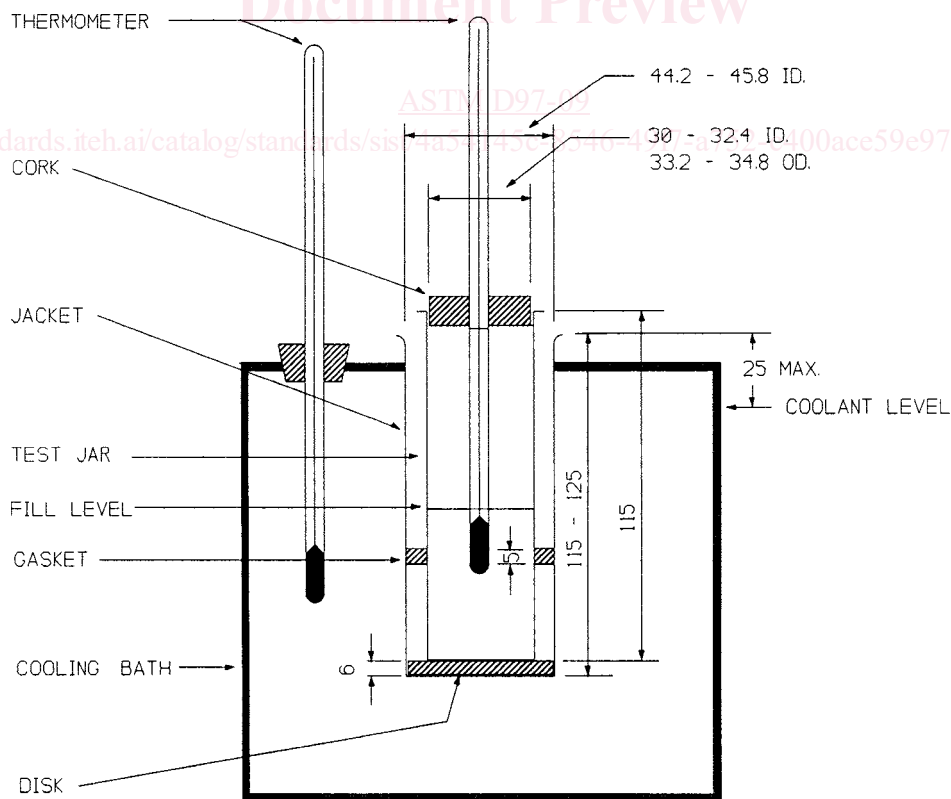
6.2 *Thermometers*, having the following ranges and conforming to the requirements prescribed in Specification E 1 for thermometers:

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
Melting point	+32 to +127°C	61C		63C

6.2.1 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  $\pm 1^\circ\text{C}$  (for example ice point).

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

6.4 *Jacket*, watertight, cylindrical, metal, flat-bottomed,  $115 \pm 3$ -mm depth, with inside diameter of 44.2 to 45.8 mm. It shall be supported in a vertical position in the cooling bath (see 6.7) so that not more than 25 mm projects out of the cooling medium, and shall be capable of being cleaned.



NOTE—Dimensions are in millimetres (not to scale).

**FIG. 1 Apparatus for Pour Point Test**

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

6.6 *Gasket*, 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 obtained by refrigeration if available, otherwise by suitable cooling mixtures. Cooling mixtures commonly used for bath temperatures down to those shown are as follows:

	Bath Temperature
Ice and water	0 ± 1.5°C
Crushed ice and sodium chloride crystals, or acetone or petroleum naphtha (see Section 7) with solid carbon dioxide added to give the desired temperature	-18 ± 1.5°C
Acetone or petroleum naphtha (see Section 7) with solid carbon dioxide added to give the desired temperature	-33 ± 1.5°C
Acetone or petroleum naphtha (see Section 7) with solid carbon dioxide added to give the desired temperature	-51 ± 1.5°C
Acetone or petroleum naphtha (see Section 7) with solid carbon dioxide added to give the desired temperature	-69 ± 1.5°C

## 7. Reagents and Materials

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

7.1.1 *Acetone*, (**Warning**—Extremely flammable).

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

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

7.1.4 *Petroleum Naphtha*, (**Warning**—Combustible. Vapor harmful).

7.1.5 *Solid Carbon Dioxide*, (**Warning**—Extremely cold -78.5°C).

## 8. Procedure

8.1 Pour the specimen into the test jar to the level mark. When necessary, heat the specimen in a bath until it is just sufficiently fluid to pour into the test jar.

NOTE 1—It is known that some materials, when heated to a temperature higher than 45°C during the preceding 24 h, do not yield the same pour point results as when they are kept at room temperature for 24 h prior to testing. Examples of materials which are known to show sensitivity to thermal history are residual fuels, black oils, and cylinder stocks.

8.1.1 Samples of residual fuels, black oils, and cylinder stocks which have been heated to a temperature higher than 45°C during the preceding 24 h, or when the thermal history of these sample types is not known, shall be kept at room temperature for 24 h before testing. Samples which are known by the operator not to be sensitive to thermal history need not be kept at room temperature for 24 h before testing.

8.1.2 Experimental evidence supporting elimination of the 24-h waiting period for some sample types is contained in a research report.<sup>5</sup>

8.2 Close the test jar with the cork carrying the high-pour thermometer (5.2). In the case of pour points above 36°C, use a higher range thermometer such as IP 63C or ASTM 61C. Adjust the position of the cork and thermometer so the cork fits tightly, the thermometer and the jar are coaxial, and the thermometer bulb is immersed so the beginning of the capillary is 3 mm below the surface of the specimen.

8.3 For the measurement of pour point, subject the specimen in the test jar to the following preliminary treatment:

8.3.1 *Specimens Having Pour Points Above -33°C*—Heat the specimen without stirring to 9°C above the expected pour point, but to at least 45°C, in a bath maintained at 12°C above the expected pour point, but at least 48°C. Transfer the test jar to a bath maintained at 24 ± 1.5°C and commence observations for pour point. When using a liquid bath, ensure that the liquid level is between the fill mark on the test jar and the top of the test jar.

8.3.2 *Specimens Having Pour Points of -33°C and Below*—Heat the specimen without stirring to at least 45°C in a bath maintained at 48 ± 1.5°C. Transfer the test jar to a bath maintained at 24 ± 1.5°C. When using a liquid bath, ensure that the liquid level is between the fill mark on the test jar and the top of the test jar. When the specimen temperature reaches 27°C, remove the high cloud and pour thermometer, and place the low cloud and pour thermometer in position. Transfer the test jar to the cooling bath (see 8.6.1).

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. 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.

<sup>5</sup> Methods for Analysis and Testing, *IP Standards for Petroleum and its Products*, Part 1, Vol 2.

<sup>5</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1377.