Designation: D97 – 09

Designation: 15/95

Standard Test Method for Pour Point of Petroleum Products[1]

This standard is issued under the fixed designation D97; 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 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.[2] A procedure suitable for black specimens, cylinder stock, and nondistillate fuel oil is described in 8.8. The cloud point procedure formerly part of this test method now appears as Test Method D2500.

1.2 Currently there is no ASTM test method for automated Test Method D97 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 D97. 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 D5853.

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:*[3]

D117 Guide for Sampling, Test Methods, and Specifications for Electrical Insulating Oils of Petroleum Origin

D396 Specification for Fuel Oils

D2500 Test Method for Cloud Point of Petroleum Products

D5853 Test Method for Pour Point of Crude Oils

D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants

E1 Specification for ASTM Liquid-in-Glass Thermometers

2.2 *Energy Institute Standards:*

Specifications for IP Standard Thermometers [4]

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.

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 D396.

[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 on Flow Properties.

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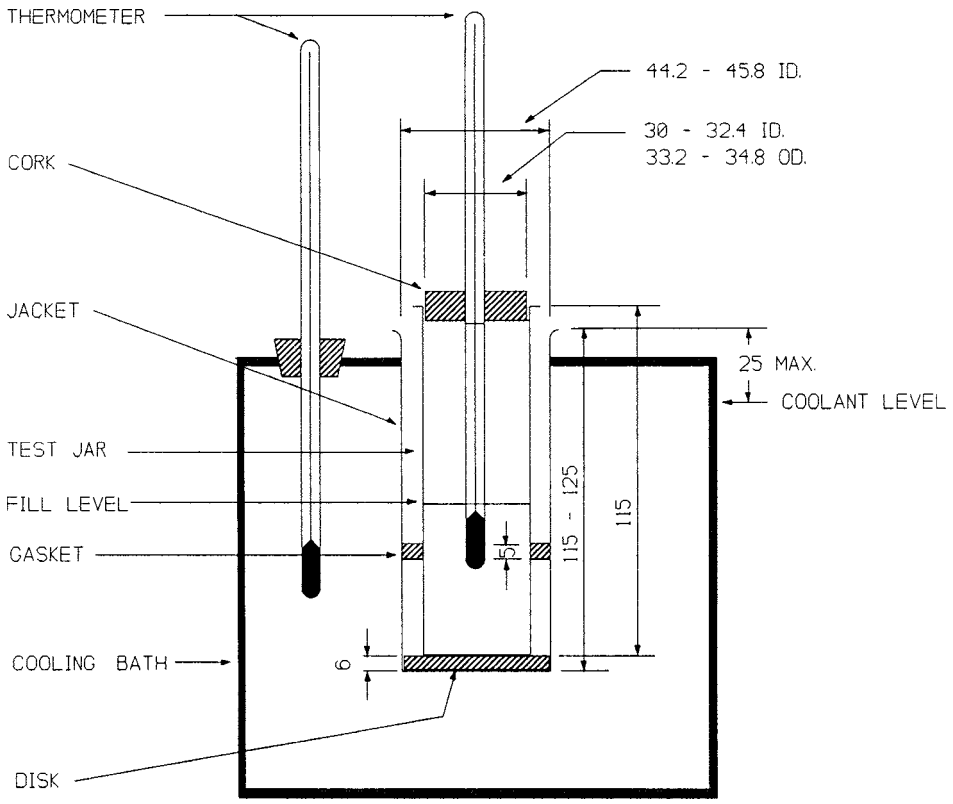
In the IP, this test method is under the jurisdiction of the Standardization Committee. This test method was adopted as a joint ASTM-IP Standard in 1965. DOI: 10.1520/D0097-09.

[2] Statements defining this test and its significance when applied to electrical insulating oils of mineral origin will be found in Guide D117.

[3] 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.

[4] 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.**



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

FIG. 1 Apparatus for Pour Point Test

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 ± 3 mm above the inside bottom. See Fig. 1.

6.2 *Thermometers*, having the following ranges and conforming to the requirements prescribed in Specification E1 for thermometers:

	Temperature	Thermometer Number	
Thermometer	Range	ASTM	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 ±1°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 ± 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.

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.[5]

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).

TABLE 1 Bath and Sample Temperature Ranges

Bath	Bath Temperature Setting, °C	Sample Temperature Range, °C
1	0 ± 1.5	Start to 9
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

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.

8.5 After the specimen has cooled to allow the formation of paraffin wax crystals, take great care not to disturb the mass of specimen nor permit the thermometer to shift in the specimen; any disturbance of the spongy network of wax crystals will lead to low and erroneous results.

8.6 Pour points are expressed in integers that are positive or negative multiples of 3°C. Begin to examine the appearance of the specimen when the temperature of the specimen is 9°C above the expected pour point (estimated as a multiple of 3°C). At each test thermometer reading that is a multiple of 3°C below the starting temperature remove the test jar from the jacket. To remove condensed moisture that limits visibility wipe the surface with a clean cloth moistened in alcohol (ethanol or methanol). Tilt the jar just enough to ascertain whether there is a movement of the specimen in the test jar. If movement of specimen in the test jar is noted, then replace the test jar immediately in the jacket and repeat a test for flow at the next temperature, 3°C lower. Typically, the complete operation of removal, wiping, and replacement shall require not more than 3 s.

8.6.1 If the specimen has not ceased to flow when its temperature has reached 27°C, transfer the test jar to a jacket in a cooling bath maintained at 0 ± 1.5°C. As the specimen continues to get colder, transfer the test jar to a jacket in the next lower temperature cooling bath in accordance with Table 1.

8.6.2 If the specimen in the jar does not show movement when tilted, hold the jar in a horizontal position for 5 s, as noted by an accurate timing device, and observe the specimen carefully. If the specimen shows any signs of movement before 5 s has passed, replace the test jar immediately in the jacket and repeat a test for flow at the next temperature, 3°C lower.

8.7 Continue in this manner until a point is reached at which the specimen shows no movement when the test jar is held in a horizontal position for 5 s. Record the observed reading of the test thermometer.

8.8 For black specimen, cylinder stock, and nondistillate fuel specimen, the result obtained by the procedure described in 8.1 through 8.7 is the upper (maximum) pour point. If

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report D02-1377.

required, determine the lower (minimum) pour point by heating the sample while stirring, to 105°C, pouring it into the jar, and determining the pour point as described in 8.4 through 8.7.

8.9 Some specifications allow for a pass/fail test or have pour point limits at temperatures not divisible by 3°C. In these cases, it is acceptable practice to conduct the pour point measurement according to the following schedule: Begin to examine the appearance of the specimen when the temperature of the specimen is 9°C above the specification pour point. Continue observations at 3°C intervals as described in 8.6 and 8.7 until the specification temperature is reached. Report the sample as passing or failing the specification limit.

9. Calculation and Report

9.1 Add 3°C to the temperature recorded in 8.7 and report the result as the Pour Point, ASTM D97. For black oil, and so forth, add 3°C to the temperature recorded in 8.7 and report the result as Upper Pour Point, ASTM D97, or Lower Pour Point, ASTM D97, as required.

10. Precision and Bias

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

10.1.1 *Lubricating Oil*[6]:

10.1.1.1 *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 6°C only in one case in twenty. Differences greater than this should be considered suspect.

10.1.1.2 *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 the normal and correct operation of this test method, exceed 9°C only in one case in twenty. Differences greater than this should be considered suspect.

10.1.1.3 The precision statements[6] were derived from a 1998 interlaboratory test program using Practice D6300. Participants analyzed five sets of duplicate base oils, three sets of duplicate multigrade lubricating oils, and one set each of duplicate hydraulic oils and automatic transmission fluid in the temperature range of -51 to -11°C. Seven laboratories participated with the manual Test Method D97. Information on the type of samples and their average pour points are in Research Report D02-1499.[6]

NOTE 2—The precision statements are the derived values rounded up to the next testing interval value. The actual derived precision values appear in Table X1.1.

10.1.2 *Middle Distillate and Residual Fuel*[7]:

10.1.2.1 *Repeatability*—The difference between successive test results obtained by the same operator using the same apparatus under constant operation conditions on identical test material would, in the long run, in the normal and correct operation of this test method, exceed 3°C only in one case in twenty. Differences greater than this should be considered suspect.

10.1.2.2 *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 the normal and correct operation of this test method, exceed 9°C only in one case in twenty. Differences greater than this should be considered suspect.

10.1.2.3 The precision statements[7] were prepared with data on sixteen middle distillate and residual fuels tested by twelve cooperators. The fuels had pour points ranging from −33 to +51°C.

NOTE 3—The precision statements are the derived values rounded up to the next testing interval value. The actual derived precision values can be seen in Table X1.1.

NOTE 4—The precisions in 10.1.2 are not known to have been derived using Practice D6300.

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

11. Keywords

11.1 petroleum products; pour point

[6] Supporting data (the results of the 1998 interlaboratory cooperative test program) have been filed at ASTM International Headquarters and may be obtained by requesting Research Report D02-1499.

[7] Based on the results of the 1983 interlaboratory cooperative test program.

APPENDIX

(Nonmandatory Information)

X1. ACTUAL DERIVED PRECISION VALUES

X1.1 See Table X1.1.

TABLE X1.1 Actual Derived Precision Values

95 % Confidence	1998 Research Program Lubricating Oil, °C	1983 Research Program Middle Distillate and Residual Fuels, °C
Repeatability	5.3	2.5
Reproducibility	8.0	6.6

SUMMARY OF CHANGES

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D97–08) that may impact the use of this standard. (Approved April 15, 2009.)

(*1*) Revised 1.1.

(*2*) Deleted Appendix X1.

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D97–07) that may impact the use of this standard. (Approved Sept. 1, 2008.)

(*1*) Revised 6.7.

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D97–06) that may impact the use of this standard. (Approved Dec. 1, 2007.)

(*1*) Revised 6.7.

(*2*) Revised 8.3.2.