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An American National Standard



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Standard Test Method for Pour Point of Crude Oils¹

This standard is issued under the fixed designation D 5853; 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 two procedures for the determination of the pour point temperatures of crude oils down to -36°C . Procedure A provides a measure of the maximum (upper) pour point temperature and is described in 9.1. Procedure B provides a measure of the minimum (lower) pour point temperature and is described in 9.2.

1.2 The use of this test method is limited to use for crude oils. Pour point temperatures of other petroleum products can be determined by Test Method D 97.

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

1.4 *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:*²

- D 97 Test Method for Pour Point of Petroleum Products
- D 130 Test Method for Corrosiveness to Copper from Petroleum Products by Copper Strip Test
- D 323 Test Method for Vapor Pressure of Petroleum Products (Reid Method)
- D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products

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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² 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.

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

E 1 Specification for ASTM Liquid-in-Glass Thermometers

E 77 Test Method for Inspection and Verification of Thermometers

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *maximum (upper) pour point, n*—the pour point obtained after the test specimen has been subjected to a prescribed treatment designed to enhance gelation of wax crystals and solidification of the test specimen.

3.1.2 *minimum (lower) pour point, n*—the pour point obtained after the test specimen has been subjected to a prescribed treatment designed to delay gelation of wax crystals and solidification of the test specimen.

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

4. Summary of Test Method

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

5. Significance and Use

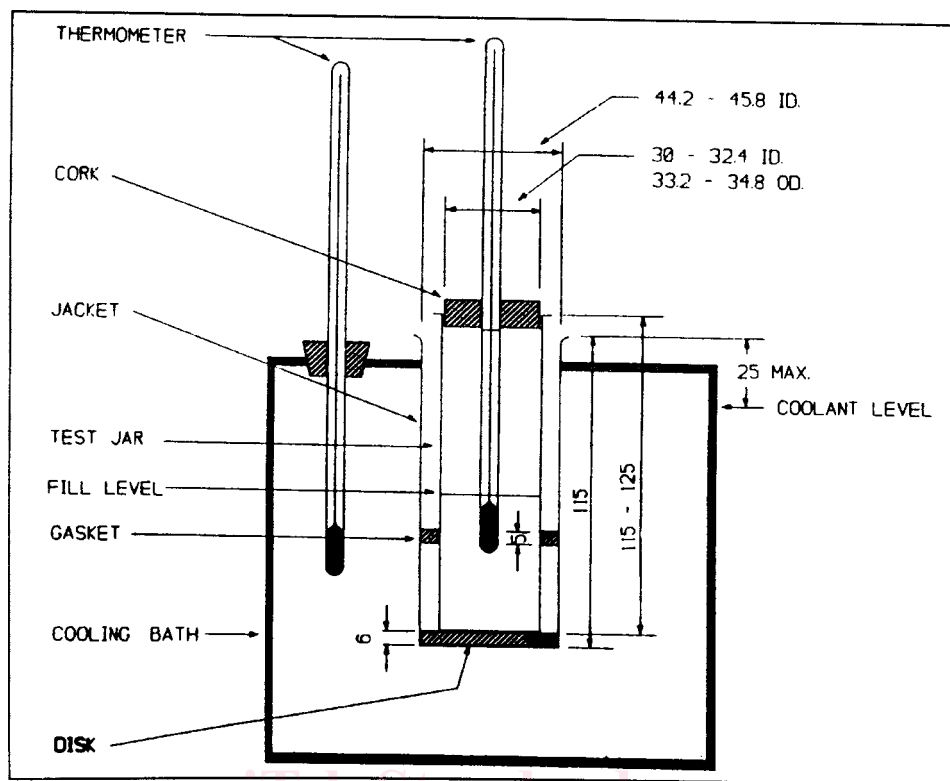
5.1 The pour point of a crude oil is an index of the lowest temperature of handleability for certain applications.

5.2 This is the only pour point method specifically designed for crude oils.

5.3 The maximum and minimum pour point temperatures provide a temperature window where a crude oil, depending on its thermal history, might appear in the liquid as well as the solid state.

5.4 The test method can be used to supplement other measurements of cold flow behavior. It is especially useful for the screening of the effect of wax interaction modifiers on the flow behavior of crude oils.

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



NOTE—All dimensions are stated in millimetres.

FIG. 1 Apparatus for Pour Point Test

6. Apparatus

6.1 Pour Point Test Apparatus Assembly (see Fig. 1):

6.1.1 *Test Jar*, cylindrical, of clear glass, flat bottomed, outside diameter 33.2 to 34.8 mm, and height 115 to 125 mm. The inside diameter of the jar can range from 30.0 to 32.4 mm, within the constraint that the wall thickness shall 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. The inside of the test jar (up to the mark) shall be visibly clean and free of scratches.

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

Thermometer	Temperature Range	Thermometer	
		ASTM	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

6.1.2.1 Since separation of liquid column thermometers occasionally occurs and may escape detection, the ice point of the thermometers shall be checked prior to the test and used only if they are accurate within $\pm 1^\circ\text{C}$ (see Test Method E 77).

6.1.3 *Cork*, to fit the test jar, center bored for the test thermometer.

6.1.4 *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 (6.1.7) so that no more than 25 mm projects out of the cooling medium. The jacket shall be capable of being cleaned.

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

6.1.6 *Gasket*, to fit snugly around the outside of the test jar and loosely inside the jacket. The gasket shall be made of rubber, leather, or other material that is sufficiently elastic 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.

6.1.7 *Cooling Bath or Baths*, of a type suitable for obtaining the required temperatures. The size and shape of the bath are options, but a support to hold the jacket firmly in a vertical position is essential. The bath temperature shall be monitored by means of the appropriate thermometer (6.1.2) or any other temperature measuring device capable of measuring and displaying the designated temperature with the required precision and accuracy. The required bath temperatures shall either be maintained by refrigeration or by suitable freezing mixtures (Note 1) and shall maintain the designated temperatures to within $\pm 1.5^\circ\text{C}$.

NOTE 1—The cooling mixtures commonly used are as follows:

	For Temperatures
Ice and water	Down to 9°C
Crushed ice and sodium chloride crystals	-12°C
Crushed ice and calcium chloride crystals	-27°C

Acetone or petroleum naphtha (see Section 7) chilled in a covered metal beaker with an ice-salt mixture to -12°C and then with enough solid carbon dioxide to give the desired temperature.

-57°C

6.2 *Water Bath*—The size and shape of the bath are optional, but a support to hold the test jar immersed in the bath to above the sample height in the test jar and in a firm vertical position is required. The required bath temperature may be maintained by any suitable means, provided the temperature can be monitored and controlled to the designated temperature ($\pm 1^{\circ}\text{C}$ (9.1.4; 9.2.4)).

6.3 *Pressure Vessel*, constructed of stainless steel according to the dimensions given in Fig. 2, and capable of withstanding a test pressure of 700 kPa. Alternative designs for the pressure vessel cap and synthetic rubber gasket may be used provided that the internal dimensions of the pressure vessel are the same as those shown in Fig. 2.

NOTE 2—This pressure vessel is identical to the pressure vessel described in Test Method D 130.

6.4 *Timing Device*, capable of measuring up to 30 s with a resolution of at least 0.1 s and an accuracy of ± 0.2 s or better.

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.)

NOTE 3—Typical petroleum naphtha used for cleaning purposes are VM and P naphthas.

7.2 *Toluene*, technical grade (**Warning**—Flammable. Vapor harmful.)

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

8. Sampling, Test Samples, and Test Specimens

NOTE 4—Sampling is defined as all steps required to obtain a portion of the contents of any pipe, tank, or other system and to place the sample into the laboratory test container.

8.1 *Laboratory Sample*—It is essential that the sample received by the laboratory is representative of the batch or lot of crude oil from which it was taken. Practices D 4057 and D 4177 provide guidance for obtaining such representative samples.

8.2 *Preparation of Test Samples*—The pour point of crude oils is very sensitive to trace amounts of high melting waxes. Exercise meticulous care to ensure such waxes, if present, are either completely melted or, if volatility constraints prevent heating to complete melting, homogeneously suspended in the sample (Appendix X1). Inspect the walls of the original container to ensure that no high melting point material is left sticking to the wall.

NOTE 5—It is not possible to define universal mandatory rules for the preparation of crude oil test samples. Guidelines for sample handling for the most common situations are given in Appendix X1.

9. Procedure

9.1 *Procedure A for Maximum (Upper) Pour Point*:

9.1.1 Pour the test sample into the test jar to the level mark. If necessary, reheat the test sample to a temperature at least 20°C above the expected pour point (8.2 and Appendix X1) but not higher than a temperature of 60°C (**Warning**—The vapor pressure of crude oils at temperatures higher than 60°C will usually exceed 100 kPa. Under these circumstances the sample container may rupture. Opening of the container may induce foaming with resultant loss of sample and possible injury to personnel.)

9.1.2 Immediately close the test jar with the cork carrying the high cloud and pour thermometer, or, if the expected pour point is above 36°C , the melting point thermometer. 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 to a depth that places the beginning of the capillary 3 mm below the surface of the test specimen.

9.1.3 Keep the test jar with the test specimen at room temperature (between 18 and 24°C) for at least 24 h.

NOTE 6—The pour point of a crude oil is dependent on the state of crystallization of the wax in the test specimen. In crude oils, achieving equilibrium between crystallized wax and dissolved wax is a rather slow process. However, experience has shown that in a majority of cases, such an equilibrium is reached within 24 h.

9.1.4 If the expected pour point is greater than 36°C , heat the sample to 9°C above the expected pour point. If the expected pour point is less than 36°C , heat the sample to a temperature of $45 \pm 1^{\circ}\text{C}$. Maintain the water bath (6.2) to heat the sample at $48 \pm 1^{\circ}\text{C}$ or at a temperature 12°C higher than the expected pour point, whichever is higher.

9.1.4.1 As soon as the test specimen has reached the required temperature, remove the cork carrying the thermometer and stir the test specimen gently with a spatula or similar device. Put the cork carrying the thermometer back in place (see 9.1.2).

9.1.5 Ensure 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 disk and jacket 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. Remove the test jar from the water bath and dry with a tissue. Place the gasket around the test jar, 25 mm from the bottom. Insert the test jar into the jacket in the first bath maintained at 21°C and commence observations for pour point. Never place a test jar directly into the cooling medium.

9.1.6 Exercise care not to disturb the mass of test specimen nor permit the thermometer to shift in the test specimen; any disturbance of the spongy network of wax crystals will lead to a lower pour point and erroneous results (Note 6).

NOTE 7—With dark colored materials, such as crude oils, it is impractical to observe, in the test jar, the onset of crystallization and network formation in the test specimen. Hence, it is presumed that network formation will begin at the very early stages of the cooling sequence.