Designation: D 5985 – 96ε1

An American National Standard

AMERICAN SOCIETY FOR TESTING AND MATERIALS
100 Barr Harbor Dr., West Conshohocken, PA 19428
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Standard Test Method for Pour Point of Petroleum Products (Rotational Method)[1]

This standard is issued under the fixed designation D 5985; 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.

ε[1] NOTE—Figures A1.1 and A1.2 were editorially corrected in May 1999.

1. Scope

1.1 This test method covers the determination of pour point of petroleum products by an automatic instrument that continuously rotates the test specimen against a suspended detection device during cooling of the test specimen.

1.2 This test method includes the range of temperatures from − 57 to + 51°C.

NOTE 1—The range of temperatures which were included in the 1992 interlaboratory program only covered the temperature range of − 39 to + 6°C (see 13.4).

1.3 This test method determines the no-flow point of petroleum products by detection of the crystal structure or viscosity increase, or both, in the sample that is sufficient to impede flow of the specimen.

1.4 This test method is not intended for use with crude oils.

1.5 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.6 *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 Method for Pour Point of Petroleum Products[2]

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products[3]

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products[3]

2.2 *IP Standards:*

IP15 Test Method for Pour Point of Petroleum Products[4]

3. Terminology

3.1 *Definitions:*

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

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *no-flow point, n—in petroleum products,* the temperature of the test specimen at which a wax crystal structure or viscosity increase, or both, impedes movement of the surface of the test specimen under the conditions of the test.

3.2.2 *Discussion*—The no-flow point occurs when, upon cooling, the formation of wax crystal structures or viscosity increase, or both, have progressed to the point where the applied observation device no longer detects movement under the conditions of the test. The preceding observation temperature, at which flow of the test specimen is last observed, is the pour point.

3.2.3 *D 97/IP15* equivalent pour point, *n—in petroleum products,* the temperature calculated by rounding the no-flow point to the next higher integer which is a multiple of 3°C.

3.2.4 *Discussion*—The no-flow point can be measured with a resolution of 0.1°C in this test method. In Test Method D 97 observations for no-flow are in 3°C intervals and when results with a similar format to Test Method D 97 are required, this calculation shall be performed. Some apparatus can perform this calculation automatically.

3.2.5 *rotational, n—in this standard,* the technique of turning the test specimen jar in an upright position upon a turntable with a stationary positioned, temperature sensor containing pendulum, inserted into the test specimen.

3.2.6 *Discussion*—Upon cooling of the test specimen, the resultant crystal formation or viscosity increase in the specimen exerts force upon the pendulum, offsetting the stationary position and causing detection of the no-flow point.

[1] This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricantsand is the direct responsibility of Subcommittee D02.07on Flow Properties.

Current edition approved July 10, 1996. Published September 1996.

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

[3] *Annual Book of ASTM Standards,* Vol 05.02.

[4] Available from Institute of Petroleum, 61 New Cavendish St., London, England W1M 8AR.

4. Summary of Test Method

4.1 After inserting the test specimen into the automatic pour point apparatus, and initiation of the program, the test specimen is heated and then cooled by maintaining a constant temperature differential between the cooling block and the sample. The test specimen is continuously tested for flow characteristics by rotating the test specimen cup at approximately 0.1 rpm against a stationary, counter-balanced, sphere-shaped pendulum. The temperature of the test specimen at which a crystal structure or a viscosity increase, or both, within the test specimen causes the displacement of the pendulum is recorded with a resolution of 0.1°C. The test specimen is then heated to the original starting temperature.

5. Significance and Use

5.1 The pour point of a petroleum product is an index of the lowest temperature of its utility for certain applications. Flow characteristics, such as pour point, can be critical for the correct operation of lubricating systems, fuel systems, and pipeline operations.

5.2 Petroleum blending operations require precise measurement of the pour point.

5.3 This test method can determine the temperature of the test specimen with a resolution of 0.1°C at which either crystals have formed or viscosity increases sufficiently to impede movement of the petroleum product.

5.4 This test method yields a D 97/IP15 equivalent pour point when the 3°C interval results are reported.

NOTE 2—Since some users may wish to report their results in a format similar to Test Method D 97 (in 3°C intervals) the precisions were derived for the temperatures rounded to the 3°C intervals. The term equivalent is intended to mean in the same format. For statements on bias relative to Test Method D 97, see 13.3.

5.5 This test method has better repeatability and comparable reproducibility relative to Test Method D 97 as measured in the 1992 interlaboratory program.[5]

6. Apparatus

6.1 *Automatic Apparatus*[6]—The automatic pour point apparatus described in the Annex A1 consists of a microprocessor controlled measuring unit that is capable of heating, cooling, rotating, and recording the temperature of the test specimen. The apparatus shall be equipped with a digital display, cooling/heating block assembly, turntable, test specimen cup and measuring head containing a counter-balanced pendulum and temperature measuring device.

6.2 *Test Specimen Cup*—The test specimen cup is a flat bottom aluminum cup with the dimensions in A1.2. To indicate the required fill level, the inside of the test cup is scribed at a height of 36 ± 0.2 mm above the inside bottom. The outside bottom of the test cup has two indentions to facilitate the rotation of the test cup.

6.3 *Circulating Bath*—Refrigeration unit equipped with a circulating pump capable of maintaining a temperature at least 20°C colder than the lowest expected pour point to be measured.

7. Reagents and Material

7.1 *Methyl Alcohol*, anhydrous, for use as cooling medium in circulating bath.

7.2 *Cleaning Solvents*, suitable for cleaning and drying the specimen cup and pendulum, such as petroleum naptha and acetone.

NOTE 3—**Warning:** Flammable. Liquid causes eye burns. Vapor harmful. May be fatal or cause blindness if swallowed or inhaled.

8. Sampling

8.1 Obtain a sample in accordance with Practices D 4057 or by D 4177.

8.2 Samples of very viscous materials can be warmed until they are reasonably fluid before they are transferred; however, no sample shall be heated more than is absolutely necessary. The sample shall not be heated and transferred into the test specimen cup unless its temperature is 70°C or lower.

NOTE 4—In the event the sample has been heated above this temperature, allow the sample to cool until its temperature is at least 70°C before transferring.

9. Preparation of Apparatus

9.1 Prepare the instrument for operation in accordance with the manufacturer's instructions.

9.2 Clean and dry the test specimen cup and the cooling well using suitable solvents as prescribed by the apparatus manufacturer.

9.3 Prepare the refrigerated circulating bath for operation in accordance with the manufacturer's instructions and allow it to attain a temperature at least 20°C lower than the expected pour point of the sample.

10. Calibration and Standardization

10.1 Ensure that all of the manufacturer's instructions for calibrating, checking, and operating the apparatus are followed.

10.2 Adjust the position of the measuring pendulum, when necessary, according to the manufacturer's instructions.

10.3 A sample with a well documented pour point can be used to verify performance of the apparatus. Alternatively, a sample which has been extensively tested in a pour point interlaboratory study can be used.

11. Procedure

11.1 Transfer the specimen into the test specimen cup to the scribed mark. When necessary, heat the sample in a water bath or oven until it is just sufficiently fluid to transfer into the test specimen cup. Samples with an expected pour point above 36°C or which appear solid at room temperature can be heated above 45°C but shall not be heated above 70°C (see Note 4).

11.2 Samples of residual fuels, black oils, and cylinder stock, 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.

[5] The results of the 1992 Interlaboratory Cooperative Test Program are available from ASTM Headquarters in the form of a research report. Request RR:D02-1312.

[6] The following instrument has been found suitable for use in this test method: Herzog Model MC 850 available from Walter Herzog, Lauda, Germany.