~~Designation:D5985–96ε1~~ Designation: D 5985 – 02 (Reapproved 2008)

An American National Standard

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.~~

INTRODUCTION

This test method covers an alternative procedure for the determination of pour point of petroleum products using an automatic apparatus.

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.

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

~~NOTE1—The range of temperatures which were included in the 1992 interlaboratory program only covered the temperature range of−39 to+6°C (see~~

1.2 This test method is designed to cover the range of temperatures from −57 to +51°C; however, the range of temperatures 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.

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

NOTE 1—The applicability of this test method on residual fuel samples has not been verified. For further information on applicability, refer to 13.4.

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

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:* [2]

D 97 Test Method for Pour Point of Petroleum Products

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

2.2 ~~*IP Standards:*~~ *Energy Institute Standards:*[3]

IP 15 Test Method for Pour Point of Petroleum Products

3. Terminology

3.1 *Definitions:*

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

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

[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.

Current edition approved May 1, 2008. Published September 2008. Originally approved in 1996. Last previous edition approved in 2002 as D 5985–02.

[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* ~~, Vol 05.01.~~volume information, refer to the standard's Document Summary page on the ASTM website.

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

[3] Available from Energy Institute, 61 New Cavendish St., London, WIG 7AR, U.K., http://www.energyinst.org.uk.

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 ~~*D97/IP15* equivalent pour point,~~ *pour point at 3°C testing intervals*, *n—in petroleum products*, the temperature calculated by rounding the no-flow point of the test specimen 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.

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.

~~5.4This test method yields a D97/IP15 equivalent pour point when the 3°C interval results are reported.~~

5.4 This test method yields a pour point in a format similar to Test Method D 97/IP15 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.~~ as measured in the 1992 interlaboratory program. (See Section 13.)

6. Apparatus

6.1 *Automatic Apparatus*[4]—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.

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

[4] The sole source of supply of the instrument known to the committee at this time is Herzog Model MC 850, available from Walter Herzog, Lauda, Germany. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

7.2 *Cleaning Solvents*, suitable for cleaning and drying the specimen cup and pendulum, such as petroleum naptha and acetone. ~~Note3—**Warning:**Flammable.~~(**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~~ 3—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~~3). ~~11.2Samples of residual fuels, black oils, and cylinder stock, which~~

NOTE 4—Residual fuels have been ~~heated~~known to ~~a temperature higher than 45°C during~~be sensitive to thermal history. In the ~~preceding 24 h or when the thermal history of these~~ case where a residual fuel sample ~~types~~ is ~~not known, shall be kept at room temperature~~ tested, refer to Test Method D 97 for ~~24 h before testing.~~ sample treatment.

~~11.3Make~~11.2 Make sure that the cooling well is free of moisture. If it is not, remove all moisture by wiping with a dry cloth. Insert the test specimen cup into the cooling well. Bring the measuring head into position over the test specimen cup and lower it into the test specimen.

11.~~4~~3 Start the test program following the manufacturer's instructions.

~~11.5When~~11.4 When the expected pour point is known to be higher than – 33°C, preselect a starting temperature which is at least 9°C higher than the expected pour point, but at least 45°C. The highest starting temperature that can be programmed is 70°C.

~~11.6When~~11.5 When the expected pour point is known to be at or below – 33°C, the test duration can be shortened by preselecting a starting temperature which is at least 9°C higher than the expected pour point. The lowest starting temperature that can be programmed is 20°C.

~~11.7When~~11.6 When the expected pour point is not known and the sample appears to be liquid, preselect a starting temperature of 45°C. The apparatus automatically heats the test specimen to approximately 45°C when a starting temperature is not selected. When the expected pour point is not known and the sample needs to be heated before transferring into the test specimen cup, preselect a starting temperature of 70°C.

~~11.8~~11.7 Initially, the test specimen is heated to approximately 45°C, or to the starting temperature preselected by the operator between 20 and 70°C. The test specimen cup is continuously rotated at approximately 0.1 rpm and flow of the cooling fluid from the circulating bath is regulated to maintain the cooling block at a temperature approximately 8°C lower than the test specimen temperature. The test specimen temperature is continuously displayed. At the detection of the last flow point, the temperature the test specimen attained is held on the digital display until reset by the operator. The test specimen is then heated to approximately 45°C, or to the preselected temperature.

~~11.9~~

11.8 The test specimen temperature rounded up to the next 3°C integer is also displayed.

12. Report

12.1 Report the temperature recorded in ~~11.8~~11.7, with resolution of 0.1°C, as the no-flow point in accordance with Test Method D 5985 (rotational method).