Designation: ~~D5949 – 10~~ D5949 – 14

Standard Test Method for Pour Point of Petroleum Products (Automatic Pressure Pulsing Method)[1]

This standard is issued under the fixed designation D5949; 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.

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 applies a controlled burst of nitrogen gas onto the specimen surface while the specimen is being cooled and detects movement of the surface of the test specimen with an optical device.

1.2 This test method is designed to cover the range of temperatures ~~from −57 to +51°C.~~from −57 °C to +51 °C. However, the range of temperatures included in the 1992 interlaboratory test program only covered the temperature range from ~~−39 to +6°C~~ −39 °C to +6 °C and the range of temperatures included in the 1998 interlaboratory test program was from ~~−51~~−51 °C to ~~−11°C.~~−11 °C. (see 13.4).

1.3 Test results from this test method can be determined at ~~1~~1 °C or ~~3°C~~3 °C testing intervals.

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

NOTE 1—The applicability of this test method or 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]
D97 Test Method for Pour Point of Petroleum Products
D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
2.2 *Energy Institute Standard:*
IP 15 Test Method for Pour Point of Petroleum Products[3]

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 the prescribed conditions of the test.

3.2 *Definitions of Terms Specific to This Standard:*

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

Current edition approved ~~May 15, 2010~~May 1, 2014. Published ~~August 2010~~June 2014. Originally approved in 1996. Last previous edition approved in ~~2008~~2010 as ~~D5949~~D5949 – 10.~~–08.~~ DOI: ~~10.1520/D5949-10.~~10.1520/D5949-14.

[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.

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

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

3.2.1.1 *Discussion—*

The no-flow point occurs when, upon cooling, the formation of wax crystal structures or viscosity increase, or both, has 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.

3.2.2 *pulse, n*—a controlled burst of nitrogen gas of a fixed pressure and flow rate sufficient to cause movement on the surface of the test specimen without fracturing the wax structure which may be formed in the specimen.

3.2.3 *Peltier device, n*—a solid-state thermoelectric device constructed with dissimilar semiconductor materials, configured in such a way that it will transport heat to or away from a test specimen dependent on the direction of electric current applied to the device.

4. Summary of Test Method

4.1 After inserting the test specimen into the automatic pour point apparatus, and initiation of the test program, the test specimen is heated and then cooled by a Peltier device at a rate of ~~1.5~~1.5 °C/min ± ~~0.1°C/min.~~0.1 °C/min. At temperature intervals of ~~1~~1 °C or ~~3°C,~~3 °C, depending on the selection made by the user, a moving force in the form of a pressurized pulse of nitrogen gas is imparted onto the surface of the specimen. Multiple optical detectors are used in conjunction with a light source to monitor movement of the surface of the specimen. The lowest temperature at which movement of the specimen surface is observed upon application of a pulse of nitrogen gas is recorded as the pour point, Test Method D5949.

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, like pour point, can be critical for the correct operation of lubricating oil systems, fuel systems, and pipeline operations.

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

5.3 In most cases, this test method does not require the use of mechanical refrigeration apparatus (see 7.1).

5.4 This test method yields a pour point in a format similar to Test Method D97/IP 15 when the ~~3°C~~3 °C interval results are reported.

NOTE 2—Since some users may wish to report their results in a format similar to Test Method D97 (in ~~3°C~~3 °C intervals) the precisions were derived from the temperatures rounded to the 3° intervals. For statements on bias relative to Test Method D97, see 13.3.

5.5 Test results from this test method can be determined at either ~~1~~1 °C or ~~3°C~~3 °C intervals.

5.6 This test method has better repeatability and reproducibility relative to Test Method D97/IP 15 as measured in the 1992 and 1998 interlaboratory test programs.[4]

6. Apparatus

6.1 *Automatic Apparatus*[5]—The automatic pour point apparatus described in this test method consists of a microprocessor controlled test chamber that is capable of heating and cooling the test specimen, providing a controlled pulse of nitrogen gas onto the specimen surface, optically detecting the specimen surface movement, and recording the temperature of the specimen as described in detail in Annex A1. It is specifically designed to detect the lowest temperature at which movement of the surface of the specimen is observed upon application of the pulse.

6.2 The apparatus shall be equipped with a specimen cup, an array of optical detectors, light source, pressure pulsing unit, digital display, Peltier device, and a specimen temperature measuring device.

6.3 The pressure pulsing unit consists of a stainless steel tubing, ~~250~~250 mm ± ~~2 mm~~ 2 mm long and ~~1.1~~1.1 mm ± ~~0.1 mm~~ 0.1 mm inside diameter. This tubing is connected to a constant pressure source at one end, which serves as an inlet. The other end

[4] The results of this interlaboratory test program are available from ASTM International Headquarters in the form of a research report. Request RR:D02-1312 for the 1992 program and RR:D02-1499 for the 1998 program.

[5] The following instrument has been found suitable for use in this test method: Phase Technology Pour Point Analyzer model series 30, 50, 70, 70V and 70X; available from Phase Technology, 11168 Hammersmith Gate, Richmond, B.C. Canada V7A 5H8. In the 1998 research report, the 70V was referred to as the *current* model; whereas models 30, 50, and 70 were referred to as *pre-1998* models. The various model series mentioned above are differentiated by their cooling capacities and user interfaces; however, all of them are capable of covering the entire temperature range specified in the scope.

This pour point analyzer is covered by a patent. Interested parties are invited to submit information regarding the identification of an alternative to this patented item to the ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee which you may attend.

of the tubing, which serves as the outlet, is bent and positioned such that it is pointing to the center of the specimen at an acute angle. The distance between the outlet and the center of the specimen is ~~8~~8 mm ± ~~2 mm.~~2 mm.

6.4 The Peltier device shall be capable of heating or cooling the test specimen at a rate of ~~1.5~~1.5 °C/min ± ~~0.1°C/min.~~0.1 °C/min.

6.5 The temperature measuring device in the specimen cup shall be capable of measuring the temperature of the test specimen ~~from −80 to +70°C~~from −80 °C to +70 °C at a resolution of ~~0.1°C.~~0.1 °C.

6.6 The apparatus shall be equipped with fittings to permit the circulation of water or other liquid cooling media to remove heat generated by the Peltier device and other electronic components of the apparatus.

6.7 The apparatus shall be equipped with fittings to permit the delivery of nitrogen gas to the pressure pulsing unit.

6.8 *Ultrasonic Bath, Unheated—(optional)*—with an operating frequency between 25 kHz to 60 kHz and a typical power output of ≤100 W, of suitable dimensions to hold container(s) placed inside of bath, for use in effectively dissipating and removing air or gas bubbles that can be entrained in viscous sample types prior to analysis. It is permissible to use ultrasonic baths with operating frequencies and power outputs outside this range, however it is the responsibility of the laboratory to conduct a data comparison study to confirm that results determined with and without the use of such ultrasonic baths does not materially impact results.

7. Reagents and Materials

7.1 *Coolant*—Tap water or other liquid heat exchange medium sufficient to remove heat generated by the Peltier device and other electronic components from the apparatus. To achieve specimen cooling ~~to −60°C,~~to −60 °C, supply circulation of liquid cooling medium ~~at +25°C~~at +25 °C or lower to the apparatus. Obtain cooling performance data from the apparatus manufacturer if lower specimen temperatures are desired or if the tap water temperature is higher than ~~25°C.~~25 °C.

7.2 *Dry Nitrogen Gas*—Nitrogen gas with a dew point below the lowest temperature attained by the specimen (**Warning—** Compressed gas.)(**Warning—**Inert gas can be an asphyxiant when inhaled.)

7.3 *Precision Volume-Dispensing Device,* capable of dispensing ~~0.150~~0.150 mL ± 0.005 mL of sample.

7.4 *Cotton Swab,* plastic shaft cotton swabs to clean the sample cup.

8. Sampling

8.1 Obtain a sample in accordance with Practice D4057 or by Practice D4177.

8.2 Samples of very viscous materials may 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~~70 °C or lower.

NOTE 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~~70 °C before transferring.

8.3 For some sample types, such as viscous lube oils that are prone to having entrained air or gas bubbles present in the sample, the use of an ultrasonic bath (see 6.8) without the heater turned on (if so equipped), has been found effective in dissipating bubbles typically within 1 min.

9. Preparation of Apparatus

9.1 Install the analyzer for operation in accordance with the manufacturer's instructions.

9.2 Turn on the liquid cooling medium and ensure its temperature is appropriate for the specimen being tested in accordance with manufacturer's instructions (see 7.1).

9.3 Turn on the nitrogen gas and ensure that it is regulated to the appropriate pressure in accordance with the manufacturer's instructions.

9.4 Turn on the main power switch of the analyzer.

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 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 Inspect the specimen cup to ensure that it is clean and dry. If needed, clean the cup in accordance with 11.3.

11.2 Deliver ~~0.150~~0.150 mL ± 0.005 mL of specimen into the specimen cup. Pipettes, syringes, or precision positive-displacement devices are suitable for use in delivering the specimen. Samples with an expected pour point above ~~36°C~~36 °C or which appear solid at room temperature may be heated above ~~45°C,~~45 °C, but shall not be heated above ~~70°C~~70 °C (see Note 4).