



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

This standard is issued under the fixed designation D 5949; 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 Test Method D 97/IP15 using an automatic apparatus. The results from this test method based on observations at 3°C temperature intervals have been found to be equivalent to Test Method D 97/IP15. When specification requires Test Method D 97/IP15, do not substitute this test method.

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 includes the range of temperatures from -57 to +51°C.

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

1.3 Test results from this test method can also be determined at 1 and 2°C testing intervals; however, precision data on these testing intervals are not available.

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

1.5 The values stated in SI units are regarded as 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:

D 97 Test Method for Pour Point of Petroleum Products²

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products³

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products³

2.2 IP Standard:

IP15 Test Method for Pour Point of Petroleum Products⁴

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 Definition 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.

3.2.1.1 *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 *D 97/IP15 equivalent pour point, 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.

3.2.2.1 *Discussion*—Test results from this test method can be determined at 1, 2, or 3°C intervals. Should the user wish to provide results with a similar format to Test Method D 97/IP15, then testing at 3°C intervals shall be used.

3.2.3 *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.

3.2.4 *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

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 D02.07.0D on Wax Related Viscometric Properties of Fuels and Oils.

Current edition approved Apr. 10, 1996. Published June 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.

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 $1.5 \pm 0.1^\circ\text{C}/\text{min}$. At temperature intervals of 1, 2 or 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 D 5949.

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 Test Method 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 from the temperatures rounded to the 3° 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 Test results from this test method can also be determined at 1 or 2°C intervals to improve resolution; however, precision data on these temperature intervals are not available.

5.6 This test method has better repeatability and reproducibility relative to Test Method D 97/IP15 as measured in the 1992 interlaboratory test program.⁵

6. Apparatus

6.1 *Automatic Apparatus*⁶—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 ± 2 mm long and 1.1 ± 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 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 ± 2 mm.

6.4 The Peltier device shall be capable of heating or cooling the test specimen at a rate of $1.5 \pm 0.1^\circ\text{C}/\text{min}$.

6.5 The temperature measuring device in the specimen cup shall be capable of measuring the temperature of the test specimen from -80 to $+70^\circ\text{C}$ at a resolution of 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.

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 , supply circulation of liquid cooling medium at $+25^\circ\text{C}$ 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 .

7.2 *Dry Nitrogen Gas*—Nitrogen gas with a dew point below the lowest temperature attained by the specimen.

NOTE 3—**Warning:** Compressed gas.

NOTE 4—**Warning:** Inert gas can be an asphyxiant when inhaled.

7.3 *Adjustable Volume Pipette*,⁷ capable of dispensing 0.150 ± 0.005 mL of sample.

7.4 *Cotton Swabs*,⁸ plastic shaft cotton swabs to clean the sample cup.

8. Sampling

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

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.

⁵ The results of this interlaboratory 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: Phase Technology Pour Point Analyzer model series 30, 50 and 70 available from Phase Technology, Richmond, B.C. Canada, V7A 5C9. The various model series mentioned above are differentiated by their cooling capacities; 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 Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee which you may attend.

⁷ A suitable pipette is the Eppendorf pipette available from Brinkmann Instruments Inc., One Cantiague Rd., P.O. Box 1019, Westbury, NY 11590-0207.

⁸ Suitable cotton swabs are Q-tips available from Cheesebrough Ponds, Trumbull Corp. Park, 55 Merritt Blvd., Trumbull, CT 06611.