



# Standard Test Method for Pour Point of Petroleum Products (Automatic Pressure Pulsing Method)<sup>1</sup>

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 ( $\epsilon$ ) 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  $+51^{\circ}\text{C}$ . However, the range of temperatures included in the 1992 interlaboratory test program only covered the temperature range from  $-39$  to  $+6^{\circ}\text{C}$  and the range of temperatures included in the 1998 interlaboratory test program was from  $-51$  to  $-11^{\circ}\text{C}$ . (see 13.4).

1.3 Test results from this test method can be determined at 1 or  $3^{\circ}\text{C}$  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 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 Products<sup>2</sup>

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products<sup>3</sup>

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products<sup>3</sup>

#### 2.2 IP Standard:

IP15 Test Method for Pour Point of Petroleum Products<sup>4</sup>

### 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:

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

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.07.0D on Wax Related Viscometric Properties of Fuels and Oils.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 05.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 05.02.

<sup>4</sup> Available from Institute of Petroleum, 61 New Cavendish St., London, England W1M 8AR.

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 or  $3^\circ\text{C}$ , 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 pour point in a format similar to Test Method D 97/IP15 when the  $3^\circ\text{C}$  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  $3^\circ\text{C}$  intervals) the precisions were derived from the temperatures rounded to the  $3^\circ$  intervals. For statements on bias relative to Test Method D 97, see 13.3.

5.5 Test results from this test method can be determined at either 1 or  $3^\circ\text{C}$  intervals.

5.6 This test method has better repeatability and reproducibility relative to Test Method D 97/IP15 as measured in the 1992 and 1998 interlaboratory test programs.<sup>5</sup>

## 6. Apparatus

6.1 *Automatic Apparatus*<sup>6</sup>—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.

<sup>5</sup> 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.

<sup>6</sup> The following instrument has been found suitable for use in this test method: Phase Technology Pour Point Analyzer model series 30, 50, 70, and 70V; available from Phase Technology, Richmond, B.C. Canada, V7A 5C9. In the 1998 research report, the 70V was referred to as the *current* model; whereas the other models were referred to as *pre-1998* models. 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 International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee which you may attend.

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,  $250 \pm 2$  mm long and  $1.1 \pm 0.1$  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  $8 \pm 2$  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  $0.1^\circ\text{C}$ .

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  $-60^\circ\text{C}$ , 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  $25^\circ\text{C}$ .

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 *Adjustable Volume Pipette*,<sup>7</sup> capable of dispensing  $0.150 \pm 0.005$  mL of sample.

7.4 *Cotton Swab*,<sup>8</sup> 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. The sample shall not be heated and transferred into the test specimen cup unless its temperature is  $70^\circ\text{C}$  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  $70^\circ\text{C}$  before transferring.

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

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

## 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 Open the test chamber lid and clean the specimen cup inside the test chamber with a cotton swab.

11.2 Use the pipette to deliver  $0.150 \pm 0.005$  mL of specimen into the specimen cup. Samples with an expected pour point above  $36^{\circ}\text{C}$  or which appear solid at room temperature can be heated above  $45^{\circ}\text{C}$ , but shall not be heated above  $70^{\circ}\text{C}$  (see Note 4). Clean the specimen out of the cup by using a cotton swab. The cup shall be cleaned to the point where no visible droplets of specimen remain in the cup.

11.3 Repeat step 11.2.

11.4 Carefully measure  $0.150 \pm 0.005$  mL of the specimen into the specimen cup.

11.5 Close and lock the test chamber lid.

11.6 Follow the manufacturer's instructions for preheating the specimen.

11.7 Select the desired pour point testing interval: 1 or  $3^{\circ}\text{C}$ .

11.8 Start the test program following the manufacturer's instructions. The specimen is first heated as specified in 11.6. It is then cooled by the Peltier device at a rate of  $1.5 \pm 0.1^{\circ}\text{C}/\text{min}$ . The apparatus will apply a pulse of pressurized nitrogen gas onto the specimen surface every 1 or  $3^{\circ}\text{C}$  drop in temperature depending on the testing interval specified in 11.7. The specimen is illuminated by the light source, and the movement of the specimen surface upon application of a pulse is monitored by an array of optical detectors. The test will continue until application of a pulse of nitrogen gas causes no observable movement on the specimen surface. This is the no-flow point of the specimen. The lowest temperature at which movement of the specimen surface is observed upon application of the pulse of nitrogen gas is recorded as the pour point. The pour point value shall be displayed as an integer temperature in multiples of 1 or  $3^{\circ}\text{C}$  depending on the selected testing interval.

11.9 Unlock and open the test chamber lid and clean the specimen out of the specimen cup with a cotton swab.

## 12. Report

12.1 Report the temperature recorded in 11.8 together with the testing interval as pour point Test Method D 5949.

## 13. Precision and Bias

13.1 *Precision*—The precision of this test method as determined by the statistical examination of the interlaboratory test results<sup>5</sup> is as follows:

13.1.1 *Repeatability*—The difference between successive test results, obtained by the same operator using the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of this test method, exceed the following values only in one case in twenty.

	1°C Test Interval	3°C Test Interval
Model 70V <sup>6</sup>	1.6°C	2.2°C
Models 30, 50, 70 <sup>6</sup>	2.9°C	4.1°C

13.1.2 *Reproducibility*—The difference between two single and independent test results, obtained by different operators working in different laboratories on identical test material, would in the long run, in normal and correct operation of this test method, exceed the following values only in one case in twenty.

	1°C Test Interval	3°C Test Interval
Model 70V <sup>6</sup>	3.2°C	3.8°C
Models 30, 50, 70 <sup>6</sup>	6.2°C	6.3°C

13.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedure in this test method, bias has not been determined.

13.3 *Relative Bias*:

13.3.1 Pour points at  $3^{\circ}\text{C}$  testing intervals were compared to the results from Test Method D 97. Relative bias<sup>5</sup> among certain samples was observed; however, the observed bias does not appear to be of a systematic nature. Biases relative to Test Method D 97/IP15 may conceivably occur for sample types not included in the 1998 interlaboratory test program.

NOTE 4—Large differences in results were observed between methods for one sample in the 1998 interlaboratory test study. The sample was a high-sulfur winter diesel. When cooled during the performance of a test method, this sample formed thin, but very large, crystals, that could be described as large plates. These crystals formed wherever sample-glass contact was made as well as covered the top surface of the sample. The entire sample, except for this all encasing thin skin of crystals, remained liquid with apparent low viscosity. When this occurred and the sample was handled gently, the sample did not pour, but with rougher handling, the crust broke and the sample poured readily. Users of this test method are advised to be alert for differences in results between test methods when this behavior is observed in the sample being tested.

13.3.2 Pour point results at  $1^{\circ}\text{C}$  testing intervals were examined for bias relative to the pour point results at  $3^{\circ}\text{C}$  intervals. A bias of  $1.1^{\circ}\text{C}$  on average was observed.

13.3.2.1 It shall be noted that when a specimen is tested at  $1^{\circ}\text{C}$  intervals, statistically the results will be  $1^{\circ}\text{C}$  lower than the results produced by  $3^{\circ}\text{C}$  testing intervals. This is due to test increment and reporting differences. Differences greater than  $1^{\circ}\text{C}$  over a number of samples would be from another cause. In the interlaboratory test program, the tests at  $1^{\circ}\text{C}$  intervals yielded a pour point lower than those obtained from the tests at  $3^{\circ}\text{C}$  intervals by  $1.1^{\circ}\text{C}$  in average.

13.4 The precision statements and the relative bias information were derived from a 1998 interlaboratory test program. Participants analyzed two sets of duplicate diesel fuel oils, five sets of duplicate base oils, three sets of duplicate multigrade lubricating oils, and one set each of duplicate hydraulic oils and automatic transmission fluid in the temperature range from  $-51$  to  $-11^{\circ}\text{C}$ . Nine laboratories participated with the Models 30, 50, 70 apparatus and six laboratories participated with the Model 70V, all testing at  $1^{\circ}\text{C}$  and  $3^{\circ}\text{C}$  intervals. Seven laboratories participated with the manual Test Method D 97 apparatus. Information on the types of samples and their

average pour points are in the research report available at ASTM International Headquarters.<sup>9,10</sup>

**14. Keywords**

14.1 Peltier; petroleum products; pour point; pressure pulsing; thermoelectric

<sup>9</sup> Supporting data are available from ASTM International Headquarters. Request research report RR:D02-1312.

<sup>10</sup> Supporting data are available from ASTM International Headquarters. Request research report RR:D02-1499.

**ANNEX**

**(Mandatory Information)**

**A1. DETAILED DESCRIPTION OF APPARATUS**

A1.1 *Test Chamber*, comprised of optical detectors, lens, light source, specimen cup, temperature sensor, Peltier device, and heat sink arranged in a configuration as shown in Fig. A1.1. The lid of the test chamber can be opened to allow cleaning of specimen cup and introduction of a new specimen. Once closed and locked, the chamber becomes air-tight. An O-ring is used to seal the mating surfaces between the lid and the rest of the chamber. The air trapped in the closed chamber is purged by dry gas. The dry gas inlet and outlet are shown in Fig. A1.1. The test chamber wall is made of black-colored metal and plastic components to minimize light reflection.

A1.1.1 *Specimen Cup*, comprised of a black plastic wall and a highly polished metal bottom. The polished surface of the bottom serves as a reflective surface for light. The transfer of heat to and away from the specimen, through the metal bottom, is controlled by the Peltier device.

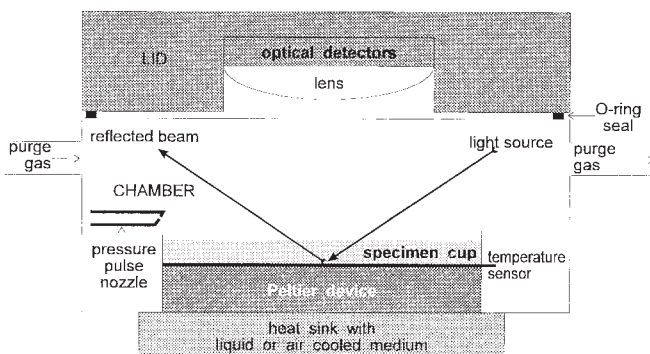
A1.1.2 *Temperature Sensor*, reading to  $0.1^{\circ}\text{C}$  and a minimum accuracy to  $0.2^{\circ}\text{C}$ , permanently embedded into the bottom of the specimen cup and positioned less than 0.1 mm below the top surface of the cup bottom. This temperature sensor, which is made of a single strand platinum, provides accurate measurement of the specimen temperature.

A1.1.3 *Peltier Device*, capable of controlling the specimen temperature over a wide range. The range varies depending on the model series. During specimen cooling, heat is transferred

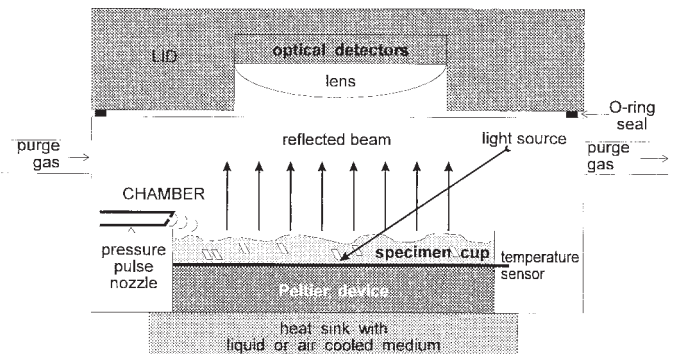
from the top of the device to the bottom. Since the top is in thermal contact with the bottom of the specimen cup, the specimen will be chilled. The bottom of the Peltier device is in thermal contact with the heat sink, where heat is dissipated to the cooling media. During specimen warming, the reverse process will take place.

A1.1.4 *Light Source*, to provide a beam of light with a wavelength of  $660 \pm 10$  nm. The light source is positioned such that it provides an incident beam (see Fig. A1.1) impinging onto the specimen at an acute angle. The light is reflected from the polished bottom of the specimen cup as well as the specimen surface. When the specimen is a homogeneous liquid, the reflected beam impinges onto the chamber lid, which is black in color. The reflected light is then absorbed by the black surface. When surface movement appears in the specimen, the reflected beam is deflected by the surface movement. A significant amount of deflected light impinges onto the lens (see Fig. A1.2).

A1.1.5 *Optical Detectors*, positioned above the lens to monitor the clarity of the specimen. The distance between the optical detectors and the lens is adjusted such that the image of the specimen is projected onto the light sensitive surface of the optical detectors. Sufficient optical detectors are used to cover the image area.



**FIG. A1.1 Schematic of Test Chamber**



**FIG. A1.2 Detection of Specimen Movement**



FIG. A1.3 Apparatus Exterior

A1.1.6 *Apparatus Exterior Interface*, the exact layout of controls and display may vary; however, a typical apparatus is shown in Fig. A1.3.

NOTE A1.1—A full description, installation, setup instructions, and maintenance instruction are contained within the manufacturers' manual supplied with each instrument.

### SUMMARY OF CHANGES

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D5949-96) that may impact the use of this standard.

- (1) The equivalency relation to D 97 has been deleted from the Introduction; any sections containing this relation are also removed.
- (2) The additional range of temperatures from the latest 1998 interlaboratory program are added to 1.2; Note 1 is incorporated into the section.

- (3) References to 2°C testing are deleted from all sections and notes of this test method.
- (4) References to residual fuel testing are deleted from 11.3 as applicability has not been verified.
- (5) Information on the 1998 interlaboratory test study have been incorporated in the related sections of Precision and Bias.

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