Designation: D5949 – 16

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

1.3 Test results from this test method can be determined at 1 °C or 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

D6708 Practice for Statistical Assessment and Improvement of Expected Agreement Between Two Test Methods that Purport to Measure the Same Property of a Material

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:*

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

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

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[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**

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 °C/min ± 0.1 °C/min. At temperature intervals of 1 °C or 3 °C, depending on the selection made by the user, a moving force in the form of a pressurized pulse of compressed 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 compressed 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 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 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 °C or 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]

[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.

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 compressed 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 mm ± 2 mm long and 1.1 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 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 mm ± 2 mm.

6.4 The Peltier device shall be capable of heating or cooling the test specimen at a rate of 1.5 °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 °C to +70 °C at a resolution of 0.1 °C.

6.6 The apparatus, if required,[5] 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. Newer models[5] have internal sources of liquid cooling media and do not require such fittings.

6.7 The apparatus shall be equipped with fittings to permit the delivery of nitrogen gas to the pressure pulsing unit. Newer models[5] have internal sources of compressed gas and do not require such fittings.

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)

[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.

Model series 30, 50, 70, and 70V require external sources of coolant and compressed gas (dry nitrogen). Model series 70X has built-in internal sources of coolant and compressed gas.

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.

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, supply circulation of liquid cooling medium at +25 °C or lower, if required,[5] 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 (**Warning—**Compressed gas.)(**Warning—**Inert gas can be an asphyxiant when inhaled.) Newer models[5] have internal sources of compressed gas and do not require external dry nitrogen gas.

7.3 *Precision Volume-Dispensing Device,* capable of dispensing 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 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 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, if required,[5] 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, if required,[5] 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 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 or which appear solid at room temperature may be heated above 45 °C, but shall not be heated above 70 °C (see Note 4).

11.3 Clean the specimen out of the cup. The cup shall be cleaned to the point where no visible droplets of specimen remain in the cup. Non-abrasive absorbent materials, such as cotton swabs, are suitable for use in cleaning the specimen cup. Cleaning solvents able to clean the specimen and compatible with the components of the apparatus may also be used. Naphtha, hexane, and heptane are suitable as cleaning solvents.

11.4 Repeat steps 11.2 and 11.3.

11.5 Carefully measure 0.150 mL ± 0.005 mL of the specimen into the specimen cup.

11.6 Close and lock the test chamber lid.

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

11.8 Select the desired pour point testing interval: 1 °C or 3 °C.

11.9 Start the test program following the manufacturer's instructions. The specimen is first heated as specified in 11.7. It is then cooled by the Peltier device at a rate of 1.5 °C/min ± 0.1 °C/min. The apparatus will apply a pulse of compressed gas onto the specimen surface every 1 °C or 3 °C drop in temperature depending on the testing interval specified in 11.8. 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 compressed 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 compressed gas is recorded as the pour point. The pour point value shall be displayed as an integer temperature in multiples of 1 °C or 3 °C depending on the selected testing interval.

11.10 Open the test chamber lid to access the specimen cup and clean the specimen out of the specimen cup (see 11.3).

12. Report

12.1 Report the temperature recorded in 11.9 together with the testing interval as pour point Test Method D5949.

13. Precision and Bias

13.1 *Precision*—The precision of this test method as determined by the statistical examination of the interlaboratory test results[4] 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[5]	1.6 °C	2.2 °C
Models 30, 50, 70[5]	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[5]	3.2 °C	3.8 °C
Models 30, 50, 70[5]	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 °C testing intervals were compared to the results from Test Method D97. Relative bias[4] among certain samples was observed; however, the observed bias does not appear to be of a systematic nature. Biases relative to Test Method D97/IP 15 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 °C testing intervals were examined for bias relative to the pour point results at 3 °C intervals. A bias of 1.1 °C on average was observed.

13.3.2.1 It shall be noted that when a specimen is tested at 1 °C intervals, statistically the results will be 1 °C lower than the results produced by 3 °C testing intervals. This is due to test increment and reporting differences. Differences greater than 1 °C over a number of samples would be from another cause. In the interlaboratory test program, the tests at 1 °C intervals yielded a pour point lower than those obtained from the tests at 3 °C intervals by 1.1 °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 °C to −11 °C. Nine laboratories participated with the Models 30, 50, 70 apparatus and six laboratories participated with the Model 70V, all testing at 1 °C and 3 °C intervals. Seven laboratories participated with the manual Test Method D97 apparatus. Information on the types of samples and their average pour points are in the research report available at ASTM International Headquarters.[6,7]

13.5 *Relative Bias Between Models 70V and 70X*—The statistical analysis by Practice D6708 of between-method bias indicates the following statistical bias between the average results of the models of instruments.[8]

13.5.1 For 3 °C interval, no correction can statistically improve agreement between 70V and 70X.

13.5.2 For 1 °C interval, the degree of agreement from Models 70V and Models 70X can be further improved by applying a constant bias-correction outlined in Eq 1.

$$X = Y - 2.5 \quad (1)$$

where:

X = 70V Models result in °C, and
Y = 70X Models result in °C.

13.6 Participants in 2013 analyzed two sets of duplicate diesel fuel oils, two sets of duplicate base oils, and two sets of duplicate formulated lubricating oils. Four laboratories participated with the Models 70V apparatus and eight laboratories participated with the Model 70X, all testing at 1 °C and 3 °C intervals. Information on the types of samples and their average pour points are in the research report available at ASTM International Headquarters.[8]

14. Keywords

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

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[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1312. Contact ASTM Customer Service at service@astm.org.

[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1499. Contact ASTM Customer Service at service@astm.org.

[8] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1832. Contact ASTM Customer Service at service@astm.org.

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 airtight. 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 °C and a minimum accuracy to 0.2 °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 nm ± 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.

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 manufacturer's manual supplied with each instrument.

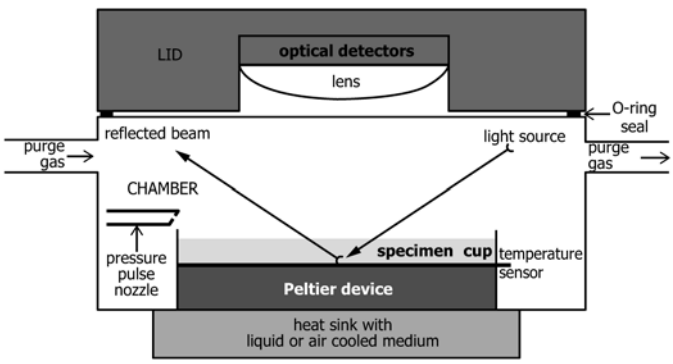

FIG. A1.1 Schematic of Test Chamber

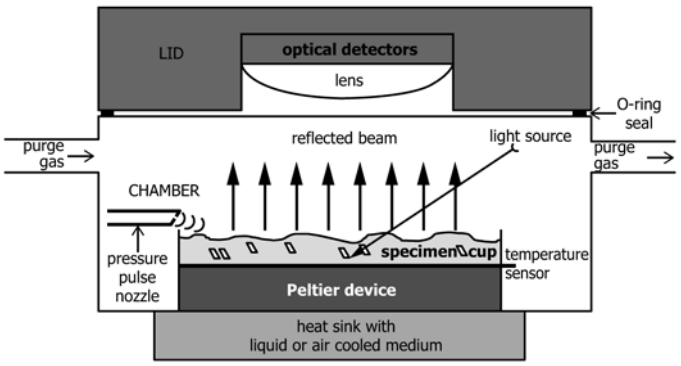

FIG. A1.2 Detection of Specimen Movement

FIG. A1.3 Apparatus Exterior

SUMMARY OF CHANGES

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D5949 – 14) that may impact the use of this standard. (Approved April 1, 2016.)

(1) Revised subsections 4.1, 6.1, 6.6, 6.7, 7.1, 7.2, 9.2, 9.3, 11.9, and footnote 5.

(2) Added new subsections 13.5 and 13.6; added new footnote 8.