



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

This standard is issued under the fixed designation D 6749; 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 apparatus that applies a slightly positive air pressure onto the specimen surface while the specimen is being cooled.

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 (1998) interlaboratory test program only covered the temperature range from  $-51$  to  $-11^{\circ}\text{C}$ .

1.3 Test results from this test method can be determined at either 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 on residual fuel samples has not been verified. For further information on the 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 prescribed conditions of test.

#### 3.2 Definitions of Terms Specific to This Standard:

3.2.1 *air pressure, n*—a regulated slightly positive air pressure gently applied onto the specimen surface in the air-tight test jar that causes upward movement of the specimen in the communicating tube, which has one end inserted into the test specimen and the other end at atmospheric pressure.

3.2.2 *no-flow point, n—in petroleum products*, the temperature of the test specimen at which a wax crystal structure of the test specimen or viscosity increase, or both, impedes movement of the surface of the test specimen under the conditions of the test.

3.2.2.1 *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.

### 4. Summary of Test Method

4.1 After inserting the test jar containing the specimen into the automatic pour point apparatus and initiating the test program, the specimen is automatically heated to the designated temperature and then cooled at a controlled rate. At temperature intervals of 1 or  $3^{\circ}\text{C}$ , depending on the selection made by the user prior to the test, a slightly positive air pressure is gently applied onto the surface of the specimen which is contained in an air-tight test jar equipped with a

<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 on Flow Properties.

Current edition approved Jan. 10, 2002. Published March 2002.

<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 W1G 7AR.

communicating tube. Since one end of the communicating tube is inserted into the specimen while the other end is maintained at atmospheric pressure, a small amount of downward movement or deformation of the specimen surface, as a result of the application of air pressure, is observed by means of upward movement of the specimen in the communicating tube. This upward movement of the specimen is detected by a pressure sensor which is installed at the atmospheric end of the communicating tube. The lowest temperature at which deformation of the specimen is observed upon application of air pressure is recorded as the pour point, Test Method D 6749.

**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 systems, fuel systems, and pipeline operations.

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

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

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. However, when specification requires Test Method D 97/IP15, do not substitute this test method.

NOTE 2—Since some users may wish to report their results in a format similar to Test Method D 97/IP15 (in 3°C intervals), the precision data were derived for the 3°C intervals. For statements on bias relative to Test Method D 97/IP15, see 13.3.1.

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

**6. Apparatus**

6.1 *Automatic Apparatus*<sup>6,7</sup>—The automatic pour point apparatus described in this test method is a microprocessor controlled apparatus that is capable of heating and cooling a specimen, applying air pressure onto the specimen’s surface, detecting the specimen’s surface movement, and then computing and reporting the pour point (see Fig. 1). The detail is described in Annex A1.

6.2 *Test Jar*, clear cylindrical glass with a flat bottom with an approximate capacity of 12 mL. Approximately 4.5 mL of sample specimen is contained when filled to the scribed line. The test jar is fitted with a test jar cap assembly on its top to form an air chamber over the test specimen.

6.3 *Test Jar Cap Assembly*—A plastic cap is installed on top of the test jar with the provision of sealing air. A glass tube with a metallic tip shall be inserted from underneath the plastic cap

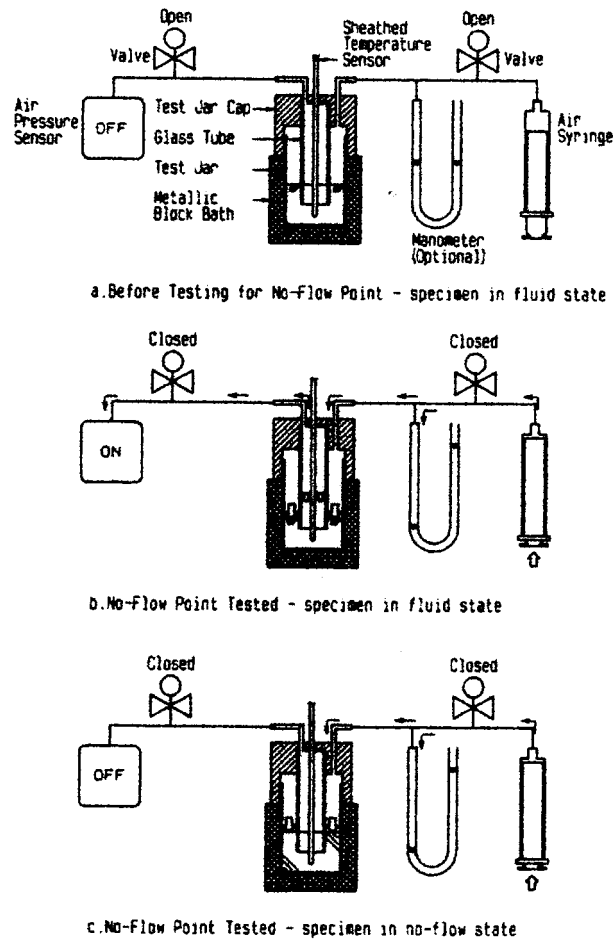


FIG. 1 Automatic Apparatus

into the round hole in the center of the test jar cap. The top end of the round hole is connected to an air pressure sensor by way of a vinyl tube. To supply air pressure to the specimen’s surface, a vinyl tube connected to an air syringe is located adjacent to the glass tube through an orifice in the plastic cap. When a specimen is to be tested, the test jar cap assembly is installed on the test jar with the lower end of the glass tube inserted into the specimen in the test jar. The glass tube and the test jar form a communicating tube. A temperature sensor in a small diameter metallic sheath shall be installed in the center of the glass tube.

6.4 *Metallic Block Bath*, a metallic block with a cylindrical hole to fit the test jar. The metallic block assembly shall have a provision for cooling/heating. A temperature sensor is embedded in the metallic block to monitor its temperature.

**7. Reagents and Materials**

7.1 *Cleaning Agents*, capable of cleaning and drying the test jar, temperature sensor, and glass tube after each test. Chemical agents such as alcohol, petroleum-based solvents, and acetone have been found suitable to use. (Warning—Flammable.) (Warning—May be harmful by itself or when evaporated.)

**8. Sampling**

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

<sup>5</sup> Available from ASTM International Headquarters. Request RR:D02-1499.  
<sup>6</sup> Tanaka model MPC series Pour Point Analyzers available from Tanaka Scientific Limited, Adachiku, Tokyo, Japan, have been found suitable for use in this test method. Various models included in this model series are differentiated by their cooling capacities or number of test heads, or both.  
<sup>7</sup> This pour point analyzer is covered by a patent. If you are aware of an alternative(s) to the patented item, please attach to your ballot return a description of the alternatives. All the suggestions will be considered by the committee.

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 jar 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.

## 9. Preparation of Apparatus

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

9.2 Clean and dry the test jar, temperature sensor, and glass tube.

9.3 Turn on the main power switch of the automatic apparatus.

## 10. Calibration and Standardization

10.1 Ensure that all of the manufacturer's instructions for calibrating, checking, and operating the automatic apparatus are followed.

10.2 Check the position of the temperature sensor and glass tube according to the manufacturer's instructions and, when necessary, make appropriate adjustments.

10.3 A sample with a well documented pour point can be used to verify the performance of the automatic apparatus. Alternatively, a sample which has been extensively tested in a pour point interlaboratory study can be used.

## 11. Procedure

11.1 Pour the sample specimen into the test jar to the scribed mark. When necessary, heat the sample in a bath or oven until it is just sufficiently fluid to pour into the test jar. Samples with an expected pour point above 36°C or samples which appear solid at room temperature can be heated above 45°C, but shall not be heated above 70°C.

11.2 Insert the charged test jar into the metallic block bath and install the test jar cap assembly snugly.

11.3 Select the desired test mode, including the pour point testing interval, according to the manufacturer's instructions. Should the user wish to provide results with a similar format to Test Method D 97/IP15, then testing at a 3°C interval shall be selected. Start the automatic sample preheating function, and then enter the expected pour point (EPP). When the 3°C testing interval is chosen, the EPP needs to be a multiple of 3°C.

11.4 Start the test program. The sample is automatically preheated by the automatic apparatus to 45°C or to EPP + 9°C, whichever is higher, but no higher than 70°C, by maintaining the bath temperature at 48°C or EPP + 12°C, whichever is higher.

11.5 After the preheating is completed, the specimen is cooled down automatically.

11.5.1 The metallic block bath is cooled down at a rate of 3 to 4°C/min, to the EPP + 40°C.

11.5.2 From the EPP + 40°C to the no-flow point, the metallic block bath is cooled at a rate of 0.8 to 1.1°C/min.

11.6 As the specimen temperature reaches a predetermined temperature, which is dependent on the EPP, the automatic apparatus starts testing for no-flow state by applying air

pressure to the specimen surface at the programmed testing interval. When the specimen is still in a fluid state, the specimen level moves up in the glass tube as air pressure is applied on the specimen surface; when the specimen reaches a no-flow state, the specimen level does not move at all in the glass tube. Upon detecting the no-flow point, the automatic apparatus computes and displays the pour point, which is the sum of the no-flow point temperature and the testing interval. Also, the automatic apparatus stops cooling and starts heating the specimen.

11.6.1 *Specimen Having Expected Pour Point Equal To or Above +36°C*—The automatic apparatus starts testing for the no-flow state at EPP + 9°C.

11.6.2 *Specimen Having Expected Pour Point Equal To or Between +31°C and +35°C*—The automatic apparatus starts testing for the no-flow state at 45°C.

11.6.3 *Specimen Having Expected Pour Point Equal To or Below +30°C*—The automatic apparatus starts testing for the no-flow state at EPP + 15°C.

11.7 Remove the test jar cap assembly and clean the test jar and test jar cap assembly.

NOTE 4—Residual fuels have been known to be sensitive to thermal history. In the case where a residual fuel sample is tested, refer to Test Method D 97 for sample treatment.

## 12. Report

12.1 Report the temperature recorded in 11.6 and the testing interval as the pour point in accordance with Test Method D 6749.

## 13. Precision and Bias

13.1 *Precision*—The precision of this test method as determined by statistical examination of interlaboratory test results is as follows:

13.1.1 *Pour Point at 3°C Testing Intervals (Test Method D 6749):*

13.1.1.1 *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, only in one case in twenty.

2.5°C

13.1.1.2 *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, only in one case in twenty.

3.1°C

13.1.2 *Pour Point at 1°C Testing Intervals (Test Method D 6749):*

13.1.2.1 *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, only in one case in twenty.

1.1°C

13.1.2.2 *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, only in one case in twenty.

2.2°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 point results at 3°C testing intervals were compared to the pour point results from Test Method D 97. Relative bias 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 5—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 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 points results at 1°C testing intervals were examined for bias relative to the pour point results at 3°C testing intervals. A bias of 1.1°C in average was observed in the 1998 interlaboratory test program.

NOTE 6—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 pour points 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 distillate diesel fuels, 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 of -51 to -11°C. Nine laboratories participated with the automatic apparatus, testing at 1°C and 3°C intervals, and seven laboratories participated with the manual Test Method D 97 apparatus. Information on the types of samples and their average pour points are available in the form of a research report.<sup>5</sup>

## 14. Keywords

14.1 automatic air pressure method; petroleum products; pour point

## ANNEX

### (Mandatory Information)

#### A1. DETAILED DESCRIPTION OF APPARATUS

A1.1 *Test Jar*, clear cylindrical glass with a flat bottom, 21 ± 0.2 mm outside diameter, 45 ± 0.5 mm height, and 1 ± 0.2 mm wall thickness. A scribed line is permanently marked at 16 ± 0.3 mm from the bottom of the jar.

NOTE A1.1—A round aluminum seal may be stuck on the bottom surface for optical cloud point detection.

A1.2 *Test Jar Cap Assembly*, consists of the following:

A1.2.1 A plastic cap with a provision to make the test jar airtight.

A1.2.2 A glass tube with a metallic tip, 8 ± 0.2 mm outside diameter, 55 ± 1 mm long, and a wall thickness of 1 ± 0.1 mm. When the test jar cap assembly is installed on the test jar, approximately 9 mm of the tip of the glass tube is inserted into the test specimen.

A1.2.3 A temperature sensor such as platinum resistance probe in a small diameter stainless steel sheath, typically 2 mm in outside diameter, to read the specimen temperature to 0.1°C. The tolerance of the temperature sensor shall be less than 1 %.

A1.3 *Air Pressure Sensor*, an electric transducer connected to the glass tube through a vinyl tube to detect upward movement of the specimen in the glass tube by sensing the increase of air pressure in the glass tube. The sensor shall be highly sensitive and accurate to at least 21 Pa (2 mm H<sub>2</sub>O).

A1.4 *Air Syringe*, a syringe driven by an electric actuator capable of applying a weak positive air pressure, such as 310 Pa (30 mm H<sub>2</sub>O), to the specimen surface at a controlled rate.

A1.5 *Metallic Block Bath*, a metallic block with a cylindrical hole to fit the test jar. The metallic block is equipped with a provision for cooling/heating the sample specimen in the test jar. The cooling system shall be capable of maintaining the cooling at a controlled rate as prescribed in 11.5.1 and 11.5.2.

A1.6 *Manometer*—A U-tube manometer can be installed between the air syringe and the glass tube for verifying, and calibrating as needed, the air pressure generated by the air syringe.



## D 6749

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