Designation: ~~D6749 – 02 (Reapproved 2012)~~ D6749 – 02 (Reapproved 2018)

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

This standard is issued under the fixed designation D6749; 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 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~~–57 °C to ~~+51°C;~~+51 °C; however, the range of temperatures included in the (1998) interlaboratory test program only covered the temperature range from ~~−51~~–51 °C to ~~−11°C.~~–11 °C.

1.3 Test results from this test method can be determined at either ~~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 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 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~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.*

1.7 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

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*, lowest temperature at which movement of the test specimen is observed under prescribed conditions of test.

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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.

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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~~W1G 7AR, U.K., ~~http://www.energyinst.org.uk.~~http://www.energyinst.org.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *air pressure, n*—regulated slightly positive air pressure gently applied onto the specimen surface in the airtight 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*, 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 °C or ~~3°C~~,3 °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 airtight test jar equipped with a 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 in accordance with Test Method D6749.

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 °C or ~~3°C~~3 °C intervals.

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. However, when specification requires Test Method D97/IP 15, do not substitute this test method.

NOTE 2—Since some users may wish to report their results in a format similar to Test Method D97/IP 15 (in ~~3°C~~3 °C intervals), the precision data were derived for the ~~3°C~~3 °C intervals. For statements on bias relative to Test Method D97/IP 15, see 13.3.1.

5.5 This test method has better repeatability and reproducibility relative to Test Method D97/IP 15 as measured in the 1998 interlaboratory test program (see Section 13).

6. Apparatus

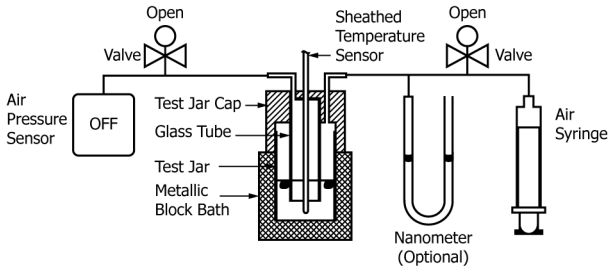
6.1 *Automatic Apparatus*[4,5]—The automatic pour point apparatus described in this test method is a microprocessor controlled apparatus that is capable of heating and cooling a specimen, applying air pressure onto the specimen's surface, detecting the specimen's surface movement, and then computing 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~~ 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.

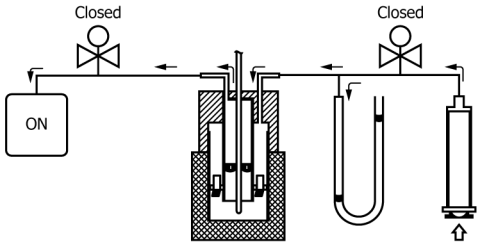
6.3 *Test Jar Cap Assembly*—A plastic cap is installed on top of the test jar with the provision of sealing air. A glass tube with a metallic tip shall be inserted from underneath the plastic cap into the round hole in the center of the test jar cap. The top end of the round hole is connected to an air 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

[4] The sole source of supply of the apparatus known to the committee at this time is Tanaka model MPC series Pour Point Analyzers available from Tanaka Scientific Limited, Adachiku, Tokyo, Japan. Various models included in this model series are differentiated by their cooling capacities or number of test heads, or both. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

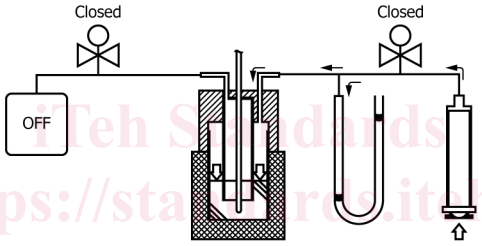
[5] This pour point analyzer is covered by a patent. If you are aware of an alternative(s) to the patented item, please attach to your ballot return a description of the alternatives. All the suggestions will be considered by the committee.



a. Before Testing for No-Flow Point - specimen in fluid state



b. No-Flow Point Tested - specimen in fluid state



c. No-Flow Point Tested - specimen in no-flow state

FIG. 1 Automatic Apparatus

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—** ~~Warning—~~ Flammable.) (**Warning—** ~~Warning—~~ May be harmful by itself or when evaporated.)

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 jar 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.

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.