



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

This standard is issued under the fixed designation D 5950; 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 Test Method D97/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 D97/IP15. When specification requires Test Method D97/IP15, do not substitute this test method.~~

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 tilts the test jar during cooling 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.~~

1.2 This test method is designed to cover the range of temperatures from -57 to +51°C; however, the range of temperatures included in the 1992 interlaboratory test program only covered the temperature range from -39 to +6°C, and the range of temperatures included in the 1998 interlaboratory test program was -51 to -11°C. (See Section 13.)

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

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

~~NOTE 1—The range of temperatures which was included in the (1992) interlaboratory test program only covered the temperature range from -39 to +6°C
(1—The applicability of this test method on residual fuel samples has not been verified. For further information on applicability, refer to 13.4).~~

~~1.3 Test results from this test method can be determined at 1, 2, and 3°C intervals.~~

~~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.~~

1.5 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

D 97 Test Method for Pour Point of Petroleum Products

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

2.2 ~~IP Standard: Energy Institute Standard:~~³

IP 15 Test Method for Pour Point of Petroleum Products

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:OD on Wax-Related Viscometric Properties of Fuels and Oils.

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² 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 Nov. 1, 2007. Published January 2008. Originally approved in 1996. Last previous edition approved in 2002 as D 5950-02.

³ 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*, Vol 05.01, volume information, refer to the standard's Document Summary page on the ASTM website.

⁴ *Annual Book of ASTM Standards*, Vol 05.02.

⁵ Available from Energy Institute, 61 New Cavendish St., London, W1G 7AR, U.K., <http://www.energyinst.org.uk>.

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 this test method.

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 the 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 *D97/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, and 3°C intervals. Should the user wish to provide results with a similar format to Test Method D97/IP15, then testing at 3°C intervals shall be used.

3.2.3 *tilting*—technique of movement where the test jar in a vertical position is moved towards a horizontal position to induce specimen movement.

3.2.3.1

3.2.3.1 *Discussion*—When the test jar is tilted and held in a horizontal position for 5 s without detection of specimen movement, this is the no-flow point and the test is complete.

4. Summary of Test Method

4.1 After preliminary heating, the test specimen is inserted into the automatic pour point apparatus. After starting the program, the specimen is cooled according to the cooling profile listed in Table 1 and examined at either 1, 2, or 3°C intervals. The lowest temperature at which movement of specimen is detected, by the automatic equipment, is displayed as the pour point.

NOTE 2—If the automatic pour apparatus's preheat option is utilized, place the test specimen into the apparatus. After starting the program, the apparatus will automatically carry out the preliminary heating.

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 This test method can determine the pour point of the test specimen with a resolution of 1.0°C.

5.4 Test results from this test method can be determined at 1, 2, and either 1 or 3°C intervals.

5.5 This test method yields a pour point in a format similar to Test Method D 97/IP15 equivalent pour point when the 3°C interval results are reported.

NOTE 3—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 for the temperatures rounded to the 3°C 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.

5.6 This test method has comparable better repeatability and better reproducibility relative to Test Method D 97/IP15 as measured in the 1992/1998 interlaboratory test program. (See Section 13.)

6. Apparatus

6.1 *Optical Automatic Pour Point Apparatus*⁴—The automatic pour point apparatus described in this test method consists of a microprocessor controller that is capable of controlling one or more independent test cells. The apparatus shall include provisions for independently controlling the temperature of each cell according to the specified cooling profile, monitoring continuously the specimen temperature, and detecting any movement of the specimen during tilting (see Fig. 1). The instrument shall be operated according to the manufacturer's instructions.

6.2 *Temperature Probe*, IEC 751 Class A: $\Delta T = \pm (0.15 + 0.002 |T|)$, capable of measurement from +70 down to -80°C. The temperature probe shall be in the center of the test jar and the top of the platinum tip immersed 3 mm below the surface of the oil.

6.3 *Test Jar*, clear cylindrical glass, flat bottom, 34 ± 0.1 -mm outside diameter, 31 ± 0.3 -mm inside diameter, 120 ± 0.5 -mm height, thickness of the bottom 2.4-mm maximum. It shall be marked with a line to indicate the sample height 54 ± 0.5 -mm above the inside bottom.

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

⁴ The following apparatus have been found suitable for use in this test method: ISL models CPP 97-6 and CPP 97-2; available from ISL SA, BP 40, 14790 Verson, France.

TABLE-2 1 Precision Data

| 95 % Conf. Specimen Temperature, °C | Base Oil Lubricants | Diesel Fuel | Additive Oil Lubricant |
|-------------------------------------|---------------------|-------------|------------------------|
| Repeatability, °C | | | |
| Jacket Temperature, °C | | | |
| 1 ^o Method | | | |
| + 27 >= ST > + 9 | 1 ± 0.33 | | 5 |
| | 0 ± 0.33 | | 5 |
| 1.75 | | | |
| + 9 > = ST > - 6 | -18 ± 0.5 | | |
| 2 ^o Method | | | |
| -6 > = ST > - 24 | ± 5.02.00 | 2.00 | |
| | -33 ± 0.00 | 2.00 | |
| Reproducibility, °C | | | |
| Reproducibility, °C5 | | | |
| 1 ^o Method | | | |
| -24 > = ST > - 42 | ± 5.0 | 1.95 | 2.85 |
| | -50 | 1 ± 0.95 | 2.85 |
| 2 ^o Method | | | |
| | ± 9.5 | | |
| -42 > = ST > - 58 | -69 ± 0.5 | | |

2.25

3.05

Specimen Temperature (°C)

- +27 >= ST > +9
- + 9 >= ST > -6
- 6 >= ST > -24
- 24 >= ST > -42
- 42 >= ST > -58

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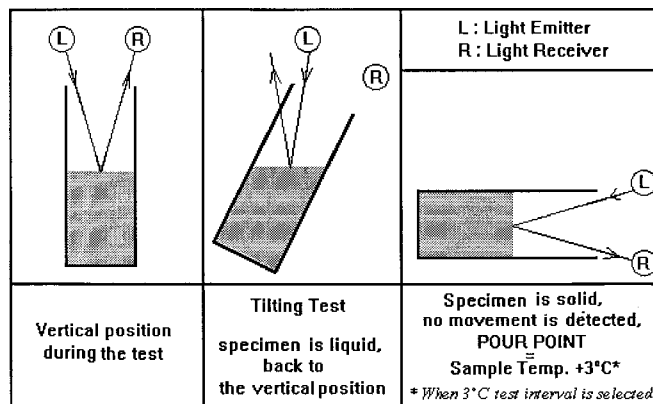
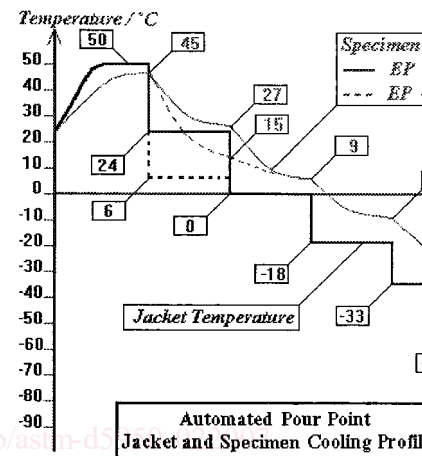


FIG. 1 Optical Detection System

6.4 *Jacket*, brass, cylindrical, flat bottom, 113 ± 0.2-mm in depth, 45 +0, -0.1-mm inside diameter. It shall be cooled according to the cooling profile specified.

6.5 *Cooling Circulating Bath*, equipped with a circulating pump and capable of maintaining a temperature at least 10°C below the last required jacket temperature level (see Table 1) and Fig. 2).

6.6 *Cork Disk*, 6 ± 0.2 mm thick to fit loosely inside the jacket. Felt may be used but special attention must be paid to avoid