



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

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 approval. 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 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 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  $-51$  to  $-11^{\circ}\text{C}$ . (see 13.4).

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

<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. Originally published as D 5950 – 96. Last previous edition D 5950 – 96.

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

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

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

3.2.2.1 *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 or  $3^{\circ}\text{C}$  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,

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

\*A Summary of Changes section appears at the end of this standard.

TABLE 1 Jacket and Specimen Temperature Cooling Profile

Specimen Temperature, °C	Jacket Temperature, °C
+27 >= ST > +9	0 ± 0.5
+9 >= ST > -6	-18 ± 0.5
-6 >= ST > -24	-33 ± 0.5
-24 >= ST > -42	-51 ± 0.5
-42 >= ST > -58	-69 ± 0.5

Specimen Temperature (°C)	Jacket Temperature (°C)
+27 >= ST > +9	0 ± 0.5
+9 >= ST > -6	-18 ± 0.5
-6 >= ST > -24	-33 ± 0.5
-24 >= ST > -42	-51 ± 0.5
-42 >= ST > -58	-69 ± 0.5

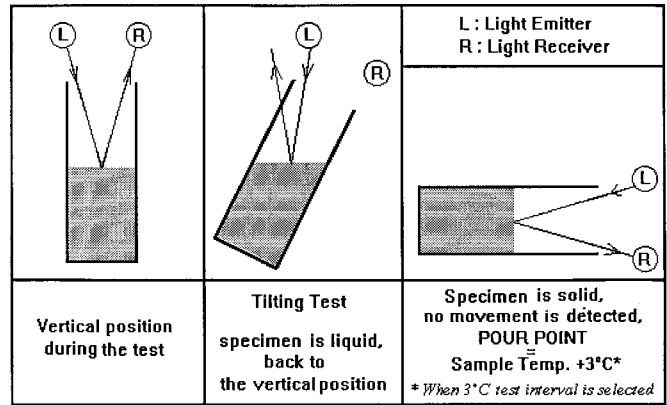
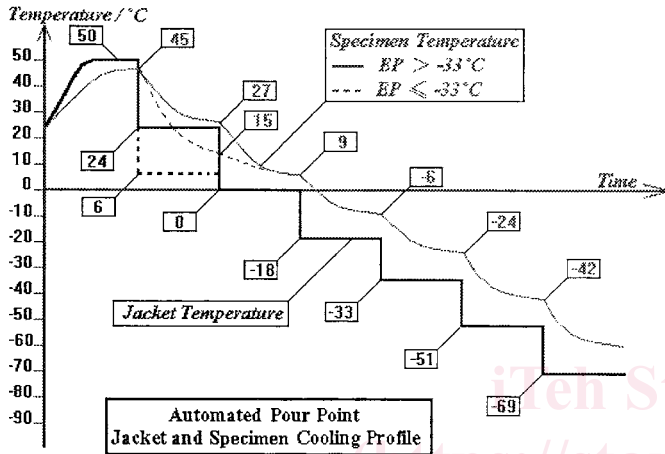


FIG. 1 Optical Detection System

5.6 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 *Optical Automatic Pour Point Apparatus*<sup>6</sup>—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: Δ T = ± (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.

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).

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 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 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. For statements on bias relative to Test Method D 97, see 13.3.

<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 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.