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

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°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 13.4).

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 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[2]

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products[3]

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products[3]

2.2 *IP Standard:*

IP15 Test Method for Pour Point of Petroleum Products[4]

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°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,

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[1] 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.OD on Wax Related Viscometric Properties of Fuels and Oils.

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[2] *Annual Book of ASTM Standards*, Vol 05.01.

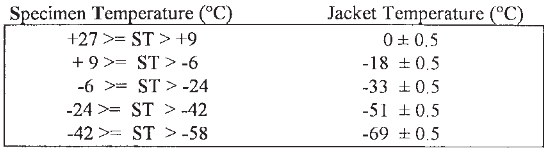
[3] *Annual Book of ASTM Standards*, Vol 05.02.

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

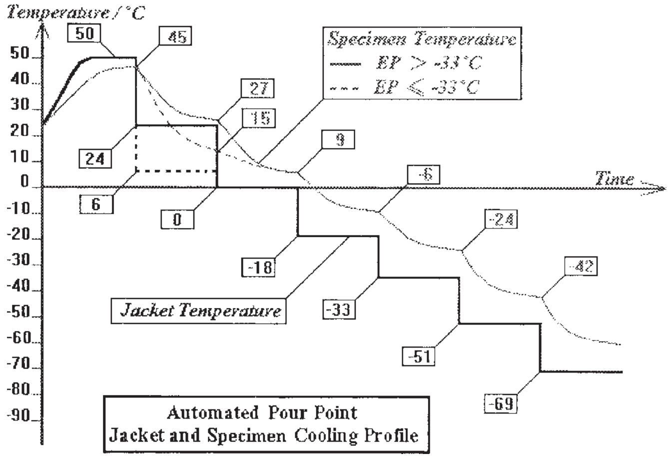
***A Summary of Changes section appears at the end of this standard.**

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



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.

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.[5]

6. Apparatus

6.1 *Optical Automatic Pour Point Apparatus*[6]—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.

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).

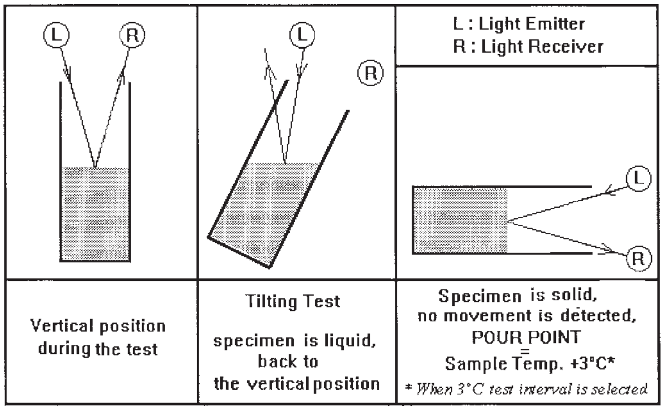


FIG. 1 Optical Detection System

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[5] 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] 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.

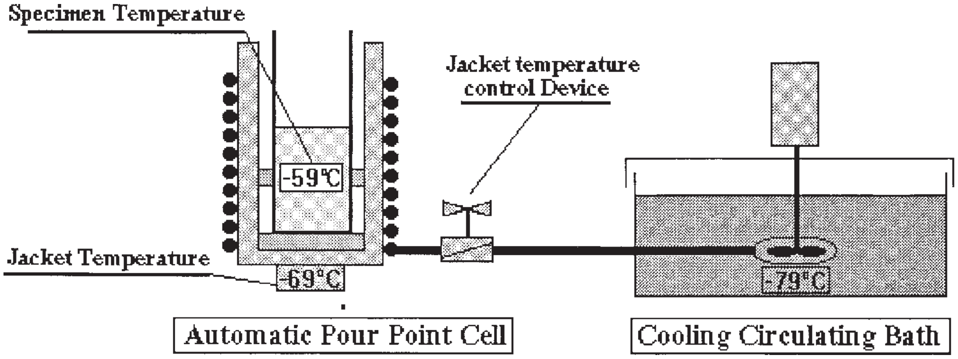

FIG. 2 Description of Pour Point Cell and Cooling Circulating Bath

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 moisture in the felt disk. The felt disk must be dried before each test.

6.7 *Cork Ring*, to fit snugly around the outside of the test jar and loosely inside the test cell. Its purpose is to prevent the test jar from touching the cooling jacket.

7. Reagents and Materials

7.1 *Methyl Alcohol*, anhydrous, for use as cooling medium in circulating bath.

7.2 *Cleaning Solvents*, suitable for cleaning and drying the test jar and test head, such as petroleum naphtha and hexane. (**Warning:** Flammable. Liquid causes eye burns. Vapor harmful. May be fatal or cause blindness if swallowed or inhaled.)

8. Sampling

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

8.2 Samples of very viscous materials can 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 jar unless its temperature is 70°C or lower.

NOTE 4—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 Prepare the instrument for operation in accordance with the manufacturer's instructions.

9.2 Clean and dry the test head and test jar using suitable solvents as prescribed by the manufacturer.

9.3 Adjust the set-point of the recirculating cooler to the appropriate temperature to cool the jackets to the required temperatures (see Table 1).

NOTE 5—For most applications the recirculating cooler will be set at its lowest operating temperature.

10. Calibration and Standardization

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

10.1.1 A test head simulator, Part No. V02306, is used to calibrate the equipment. The test head simulator uses precision resistors in place of the PT 100 temperature probe to calibrate the jacket and specimen temperature electronics. Follow the manufacturer's calibration instructions.

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 Pour the sample into the test specimen jar to the scribed mark. When necessary, heat the sample in a water bath or oven until it is just sufficiently fluid to pour the sample into the test specimen 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 should not be heated above 70°C (see Note 4).

11.2 Subject the test specimen to the following preliminary treatment or use the instrument's automatic preheat option.

NOTE 6—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.

11.2.1 When the expected pour point (EP) is known to be ≤ -33°C, heat the test specimen to 45°C in a bath or oven maintained at 48°C.

11.2.2 When the expected pour point (EP) is known to be > -33°C, heat the test specimen to EP + 9°C, or at least to 45°C but no higher than 70°C (see Note 4).

11.3 Place a cork disk at the bottom of the jacket in the required cell and fit a cork ring to the test jar. The cork ring should be 25 ± 3 mm above the bottom of the test jar.

11.4 Place the test jar in the selected test cell. Attach the detector head according to the manufacturer's instructions.

11.5 Select the desired testing interval (1 or 3°C).

11.6 Enter the expected pour point (EP). If 3°C testing intervals are chosen (11.5) you must enter an expected pour point that is a multiple of 3°C.

11.7 Start the test in accordance with the manufacturer's instructions.

11.8 At this point, the instrument shall monitor the test specimen with the optical detector, adjusting the jacket temperature to the first temperature level (according to Table 1) and measuring the specimen temperature. The instrument shall automatically change the jacket temperature in accordance with the specimen temperature (according to Table 1). The time to move the jacket temperature from one level to the next lower level shall not exceed 90 s. The instrument shall start tilting the specimen (without removing it from the jacket) in the prescribed manner when the temperature of the test specimen is at 9°C higher than the expected pour point. If the specimen flows during the tilting movement, the no-flow point is not reached and the jacket returns to the waiting vertical position for the next test. The test will continue until the jacket is in a complete horizontal position and the detector does not detect any movement of the specimen for 5 s. This temperature, the no-flow point, plus 1 or 3°C (depending on the test interval selected) is the pour point of the oil (see Fig. 1). When the pour point is determined, the instrument shall display the pour point result and start to reheat the test specimen.

11.9 If the instrument detects the no-flow point on the first tilting cycle (EP + 9°C), disregard the result and start with 11.1 using a higher expected pour point.

11.10 Record the result as the pour point without any correction.

NOTE 7—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 11.10 together with the testing interval as pour point in accordance with Test Method D 5950.

13. Precision and Bias

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

13.1.1 *Pour Point at 3°C Testing Intervals*

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.

3.9°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.

6.1°C

13.1.2 *Pour Point at 1°C Testing Intervals*

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.

2.7°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.

4.5°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 D 97. Relative bias[5] 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 8—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 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 *Discussion*—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 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 of −51 to −11°C. Eight 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.[7]

14. Keywords

14.1 D 97 equivalent; petroleum products; pour point; tilt method

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[7] Information on the types of samples and their average pour points are available from ASTM International Headquarters. Request Research Report RR:D02-1499.

SUMMARY OF CHANGES

Committee D02 has identified the location of selected changes to this standard since the last issue (D 5950-96) that may impact the use of this standard.

(*1*) Introduction—the equivalency relation to Test Method D 97 has been removed, any sections containing this relation are also removed.

(*2*) Paragraph 1.2—the additional range of temperatures from the latest 1998 interlaboratory program are added, and Note 1 is incorporated into the section.

(*3*) References to 2 C testing are removed from all sections and notes of this test method.

(*4*) Paragraph 11.3—References to residual fuel testing are removed as applicability has not been verified.

(*5*) Section 13, Precision—information on the 1998 interlaboratory program have been incorporated in the related sections of Precision and Bias.

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