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Standard Test Method for No Flow Point and Pour Point of Petroleum Products and Liquid Fuels[1]

This standard is issued under the fixed designation D7346; 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.

1. Scope*

1.1 This test method covers the determination of the no flow point ~~temperature~~ and pour point of petroleum ~~products~~ products, liquid fuels, biodiesel, and biodiesel blends using an automatic instrument.

1.2 The measuring range of the apparatus is from –95 °C to 45 °C, however the precision statements were derived only from samples with no flow point temperatures from –77 °C to +2 °C and samples with pour point in the temperature range of –58 °C to +12 °C.

1.3 Pour point results from this test method can be reported at 1 °C or 3 °C intervals.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *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]

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
D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants
D6708 Practice for Statistical Assessment and Improvement of Expected Agreement Between Two Test Methods that Purport to Measure the Same Property of a Material

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 the test.

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 formation or viscosity increase, or both, is sufficient to impede movement of the surface of the test specimen under the conditions of the test.

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum 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.

3.2.1.1 *Discussion—*

The no-flow point occurs when, upon cooling, the formation of wax crystal structures or viscosity increase, or both, have progressed to the point where the applied observation device no longer detects movement under the conditions of the test.

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

4. Summary of Test Method

4.1 After inserting the test specimen into the automatic no flow point apparatus and initiating the program, the test specimen is heated, if necessary, to a starting temperature and then cooled by prescribed rates. The test specimen is continuously tested for flow characteristics by continuously monitoring the air pressure variation inside the test specimen vial. When the specimen is still fluid, its movement will partially compensate for the reduction in air pressure in the test chamber above the test specimen surface. At some temperature the pressure measuring system detects a pressure decrease due to incapability of the test specimen to flow caused by a crystal structure formation in the specimen or its viscosity increase, or both. This temperature is recorded as no flow point with a resolution of 0.1 °C. The pour point is recorded by rounding the no flow point temperature to either the next warmer 1 °C interval or 3 °C interval. The test specimen is then reheated to allow for removal from the test chamber.

5. Significance and Use

5.1 The no flow point of a petroleum product is an index of the lowest temperature of its utility for some applications. Flow characteristics, such as no flow point, can be critical for the proper operation of lubricating systems, fuel systems, and pipeline operations.

5.2 Petroleum blending operations require precise measurement of the no flow point.

5.3 This test method can determine the temperature of the test specimen with a resolution of 0.1 °C at which either crystals have formed or viscosity has increased sufficiently, or both, to impede flow of the petroleum product.

5.4 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.

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

5.6 Pour point results from this test method can be reported at either 1 °C or 3 °C intervals.

5.7 This test method yields a pour point in a format similar to Test Method D97/IP15 when the 3 °C interval results are reported.

5.8 This test method has better repeatability and reproducibility relative to Test Method D97/IP15 as measured in the 2011 interlaboratory test program (see 13.1.2).

6. Apparatus (see Annex A1)

6.1 *Automatic No Flow Point Apparatus*[3]—The apparatus consists of a microprocessor–controlled test specimen chamber that is capable of heating and cooling the test specimen at required rate, measuring the pressure inside the test specimen vial, and recording the temperature of the test specimen chamber. A detailed description of the apparatus is provided in Annex A1.

6.2 The apparatus shall be equipped with a thermostatically controlled specimen chamber, digital display, cooling and heating systems, pressure measuring system, and a specimen chamber temperature measuring device.

6.3 The temperature measuring device in the specimen chamber shall be capable of measuring the temperature from –105 °C to 60 °C at a resolution of 0.1 °C.

6.4 *Ultrasonic Bath, Unheated—(optional),* with an operating frequency between 25 kHz to 60 kHz and a typical power output of ≤100 W, of suitable dimensions to hold container(s) placed inside of bath, for use in effectively dissipating and removing air or gas bubbles that can be entrained in viscous sample types prior to analysis. It is permissible to use ultrasonic baths with operating frequencies and power outputs outside this range, however it is the responsibility of the laboratory to conduct a data comparison study to confirm that results determined with and without the use of such ultrasonic baths does not materially impact results.

7. Reagents and Materials

7.1 *Specimen Vial*—Disposable, clear glass cylinder with closed flat bottom, 1 mL capacity.

Dimensions:
Outer diameter: 8.0 mm to 8.3 mm
Wall thickness: 0.75 mm to 0.85 mm
Outer length: 39.25 mm to 40.25 mm

NOTE 1—Standard NWV type vial was found suitable for the application.

7.2 *Specimen Vial Stopper*—Disposable, proprietary designed for use in this apparatus.

7.3 *Micropipette*—Capable of delivering 0.5 mL ± 0.1 mL of sample. Positive displacement type micropipette with capillary piston is preferred for use. Air-displacement type micropipettes are not recommended for viscous samples.

[3] The sole source of supply of the apparatus known to the committee at this time is ISL model MPP 5Gs Analyzer, available from ISL, B.P. 70285 14653 Verson, France. 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.