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Standard Test Method for No Flow Point of Petroleum Products[1]

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1. Scope

1.1 This test method covers the determination of the no flow point temperature of petroleum products using an automatic instrument.

1.2 The measuring range of the apparatus is from -95 to 45°C, however the precision statements were derived only from samples with no flow point temperatures from -77 to +2°C.

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

1.4 *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

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *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.

3.1.1.1 *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.

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 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.

6. Apparatus (see Annex A1)

6.1 *Automatic No Flow Point Apparatus*[3]—The apparatus consists of a microprocessor–controlled test specimen chamber

[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 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] 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.

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 to 60°C at a resolution of 0.1°C.

7. Reagents and Materials

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

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

8. Sampling

8.1 Obtain a sample in accordance with Practice D4057 or D4177.

8.2 At least 1 mL of sample is required for each test.

8.3 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 cup unless its temperature is 70°C or lower.

NOTE 2—In the event the sample has been heated above this temperature, allow the sample to cool until its temperature is below 70°C before transferring it.

9. Preparation of Apparatus

9.1 Prepare the apparatus for operation in accordance with the manufacturer's instructions.

10. Calibration and Standardization

10.1 Ensure that all of the manufacturer's instructions for calibration of the mechanical and electronic systems and operation of the apparatus are followed.

10.2 To verify the performance of the apparatus, a sample for which extensive data has been obtained by no flow point test method may be used. Such verification materials can also be prepared from intra-company cross checks.

11. Procedure

11.1 Draw 0.5 ± 0.1 mL of sample into a micropipette and transfer the specimen into a new clean, dry specimen vial. When necessary, heat the sample in a water bath or oven until it is just sufficiently fluid to transfer. Samples with an expected no flow point above 25°C or which appear solid at room temperature can be heated above 45°C but shall not be heated above 70°C (see Note 2).

NOTE 3—Some samples like residual fuels, black oils, and cylinder stock have been known to be sensitive to thermal history. In the case where such sample is tested, refer to Test Method D97 for sample treatment prior testing.

11.2 Place a new clean, dry specimen vial stopper on the specimen vial and insert the assembly into the apparatus. Start the operation of the apparatus according to the manufacturer's instructions. When the expected no flow point of the specimen is known, program it in the apparatus as EP (expected point) and start test sequence. From this point up to and including the termination of the test, recording and reporting of the result, the apparatus automatically controls the procedure.

11.3 The apparatus adjusts the specimen chamber to a preselected starting temperature. By default, the preselected starting temperature is 25°C. Alternatively, the operator can preprogram a defined starting temperature between 25 and 60°C, if desired. When the expected no flow point is known and programmed in the apparatus, the starting temperature shall be at least 30°C warmer. In the event that the preselected starting temperature is programmed lower than 30°C above the expected no flow point, the apparatus shall heat the specimen chamber to a starting temperature at least 30°C above the expected no flow point, but not more than 60°C.

11.4 When the expected no flow point of the specimen is not known, once the starting temperature is reached (see 11.3) the specimen chamber is cooled at a rate of 1.5 ± 0.15°C/min.

11.5 At the same time the cooling begins, the pressure measurement system is engaged to continuously monitor specimen behavior. When a decrease in pressure, as determined by the apparatus, is measured in the specimen vial, which signifies that the test specimen has ceased to flow due to a crystal structure formation in the specimen or its viscosity increase, or both, the temperature of the specimen chamber is recorded as the no flow point and held on a digital display. The test chamber is than reheated and the test sequence is terminated.

11.6 When a no flow point is detected prematurely, as determined by the apparatus, the specimen is automatically reheated to a higher starting temperature, at least 30°C warmer than the temperature of premature detection, and then cooled as described in 11.4 until the no flow point is detected as described in 11.5.

NOTE 4—Some typical examples of premature no flow point are: when the NFP is detected during the fast cooling phase or when the NFP is detected less than 30°C from the starting temperature.

11.7 When the expected no flow point of the specimen is known, and programmed into the apparatus, once the starting temperature (see 11.3) is reached, the specimen chamber is cooled at a rate of 10 ± 1°C/min until 30°C warmer than the programmed expected no flow point, then the cooling rate is adjusted to 1.5 ± 0.15°C/min and the no flow point temperature is detected as described in 11.5.