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Standard Test Method for Cloud Point of Petroleum Products (Constant Cooling Rate Method)¹

This standard is issued under the fixed designation D 5773; 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 describes an alternative procedure for the determination of cloud point of petroleum products Test Method D 2500/IP219 using an automatic apparatus. The temperature results from this test method have been found to be equivalent to Test Method D 2500/IP219. When specification requires Test Method D 2500/IP219, do not substitute this test method or any other method without obtaining comparative data and agreement from the specifier.

1. Scope

1.1 This test method describes the determination of the cloud point of petroleum products that are transparent in layers 40 mm in thickness by an automatic instrument using a constant cooling rate.

1.2 This test method covers the range of temperatures from -40°C to $+49^{\circ}\text{C}$ with temperature resolution of 0.1°C .

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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:

D 2500 Test Method for Cloud Point of Petroleum Products²

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products³

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products³

2.2 IP Standard:

IP219 Test Method for Cloud Point of Petroleum Products⁴

3. Terminology

3.1 Definitions:

3.1.1 *cloud point, n*—in petroleum products, the temperature of a liquid specimen when a wax crystal structure that is similar in appearance to a cloud is formed upon cooling under prescribed conditions.

3.1.1.1 *Discussion*—The cloud point appears when the temperature of the specimen is low enough to cause wax crystals to precipitate. In a homogeneous liquid, the cloud is always noted first at the location in the specimen where the specimen temperature is the lowest. This is typically at the lower portion of the test jar when using the apparatus described in Test Method D 2500.

3.2 Description of Terms Specific to This Standard:

3.2.1 *automatic cloud point, n*—the temperature of a specimen, when the appearance of the cloud is determined under the conditions of this test method.

3.2.1.1 *Discussion*—The cloud point in this test method is determined by an automatic instrument using an optical device for detection of the crystal formation. The apparatus and the conditions are different from those established for Test Method D 2500, although according to interlaboratory examination, the results have been determined to be equivalent to Test Method D 2500.

3.2.2 *D 2500/IP219 equivalent cloud point, n*—the temperature of a specimen, in integers, calculated by rounding the results of this test method to the next lower integer.

3.2.2.1 *Discussion*—This test method produces results with

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 on Flow Properties.

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

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

⁴ IP standards are available from the Institute of Petroleum, 61 New Cavendish Street, London, England W1M 8AR.

0.1°C resolution. Should the user wish to provide results with a similar format to Test Method D 2500, then this calculation can be performed. Some apparatus can perform this calculation automatically.

3.2.3 *Peltier device, n*—a solid state thermoelectric device constructed with dissimilar semiconductor materials and configured in such a way that it will transfer heat to or away from a test specimen dependent on the direction of electric current applied to the device.

4. Summary of Test Method

4.1 A specimen is cooled by a Peltier device at a constant rate of $1.5 \pm 0.1^\circ\text{C}/\text{min}$ while continuously being illuminated by a light source. The specimen is continuously monitored by an array of optical detectors for the first appearance of a cloud of wax crystals. The detectors are sufficient in number to ensure that any solid phase hydrocarbon crystals that may form are detected. The temperature at which the appearance of a cloud of wax crystals is first detected in the specimen is recorded to 0.1°C resolution. When the recorded temperature is rounded to the next lower integer temperature, it is designated as the D 2500/IP 219 equivalent cloud point per Test Method D 5773.

5. Significance and Use

5.1 The cloud point of a petroleum product is an index of the lowest temperature of its utility for certain applications. Wax crystals of sufficient quantity can plug filters used in some fuel systems.

5.2 Petroleum blending operations require a precise measurement of the cloud point.

5.3 This test method can determine the temperature of the test specimen at which wax crystals have formed sufficiently to be observed as a cloud with a resolution of 0.1°C.

5.4 This test method provides results that are equivalent to Test Method D 2500.

NOTE 1—This is based on the Test Method D 2500 equivalent cloud point in which the 0.1°C result is rounded to the next lower integer.

5.5 This test method determines the cloud point in a shorter period of time than Test Method D 2500.

NOTE 2—In cases of samples with cloud points near ambient temperatures, time savings may not be realized.

5.6 This test method eliminates most of the operator time required of Test Method D 2500.

5.7 This test method does not require the use of a mechanical refrigeration apparatus.

NOTE 3—In certain cases of high ambient temperature, a source of cooling water may be required to measure low temperature cloud points (see 7.1).

6. Apparatus

6.1 *Automatic Apparatus*⁵—The automatic cloud point apparatus described in this test method consists of a test chamber controlled by a microprocessor that is capable of controlling the heating and cooling of the test specimen, optically observing the first appearance of a cloud of wax crystals and recording the temperature of the specimen described in detail in Annex A1.

6.2 The apparatus shall be equipped with a specimen cup, optical detector array, light source, digital display, Peltier device, and a specimen temperature measuring device.

6.3 The Peltier device shall be capable of heating or cooling the test specimen at a constant rate of $1.5 \pm 0.1^\circ\text{C}/\text{min}$.

6.4 The temperature measuring device in the specimen cup shall be capable of measuring the temperature of the test specimen from -40 to $+70^\circ\text{C}$ at a resolution of 0.1°C.

6.5 The apparatus shall be equipped with fittings to permit the circulation of a liquid cooling medium, if required, to remove heat generated by the Peltier device and other electronic components of the apparatus.

NOTE 4—Some apparatus are designed to use ambient air as a cooling medium. In such cases, a built-in fan is available to provide circulation of air and there is no need for fittings as described for a liquid cooling medium. The function of the cooling medium is to remove heat from the electronic components. The choice of the cooling medium has no impact whatsoever on the test results.

6.6 The apparatus shall be equipped with fittings to permit the circulation of purge gas to purge the test chamber containing the specimen cup of any atmospheric moisture.

7. Reagents and Materials

7.1 *Cooling Medium*—Air, tap water, or other liquid heat exchange medium sufficient to remove heat generated by the Peltier device and other electronic components from the apparatus. To achieve specimen cooling to -40°C , supply circulation of liquid cooling medium at $+25^\circ\text{C}$ or lower to the apparatus. For an apparatus which relies on air as cooling medium, the ambient air temperature has to be below $+30^\circ\text{C}$ to achieve specimen cooling to -40°C .

7.2 *Purge Gas*—A gas such as air, nitrogen, helium, or argon with a dew point below the lowest operating temperature of the analyzer.

NOTE 5—**Warning:** Compressed gas under high pressure.

NOTE 6—**Warning:** Inert gas can be an asphyxiant when inhaled.

7.3 *Adjustable Volume Pipette*, capable of dispensing 0.15 ± 0.01 mL of sample.

7.4 *Cotton Swabs*—Plastic or paper shaft cotton swabs used to clean the sample cup.

NOTE 7—**Caution:** The use of swabs with wooden shafts may damage the mirrored surface of the specimen cup.

⁵ The following instrument has been found suitable for use in this test method: Phase Technology Cloud Point Analyzer model series 10, 30, and 70 available from Phase Technology, #135-11960 Hammersmith Way, Richmond, B.C. Canada V7A 5C9. The various model series mentioned above are differentiated by their cooling capacities; however, all of them are capable of covering the entire temperature range specified in the scope.

8. Sampling

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

8.2 Samples of very viscous materials may be warmed until they are reasonably fluid before they are tested. However, no sample should be heated more than absolutely necessary.

8.3 The sample shall not be heated above 70°C. When the sample is heated above 70°C, allow the sample to cool below 70°C before filtering or inserting into the apparatus.

8.4 When moisture is present in the sample, remove the moisture by a method, such as filtration through dry lint-free filter paper, until the oil is perfectly clear, but make such filtration at a temperature at least 14°C above the expected cloud point.

NOTE 8—Moisture will be noticed in the sample as a separate phase or as a haze throughout the entire sample. Generally, a slight haze will not interfere with the detection of the wax cloud.

9. Preparation of Apparatus

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

9.2 Make liquid cooling medium connections if required (see Note 4) and ensure that they do not leak.

9.3 Make purge gas connections and ensure that they do not leak.

9.4 Turn on the liquid cooling medium if required (see Note 4).

9.5 Turn on the purge gas.

9.6 Turn on the main power switch of the analyzer. After the automatic self diagnostics start-up sequence is completed, the instrument will display a **READY** message.

10. Calibration and Standardization

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

10.2 A sample with a mutually agreed upon cloud point can be used to verify performance of the apparatus.

11. Procedure

11.1 Open the test chamber lid and clean the specimen cup inside the test chamber with a cotton swab.

11.2 Use a pipet to deliver 0.15 ± 0.01 mL of specimen into the specimen cup. Clean the specimen out of the cup by using a cotton swab. The cup must be cleaned to the point where no visible droplets of specimen remain in the cup.

11.3 Repeat step 11.2.

11.4 Carefully measure 0.15 ± 0.01 mL of specimen into the specimen cup.

11.5 Close and lock the test chamber lid.

11.6 Select the **PRE-HEAT** menu on the apparatus if the expected cloud point is less than 14°C below the specimen ambient temperature. The specimen ambient temperature is displayed on the front panel of the apparatus. With this selection, the apparatus will automatically heat the specimen to a starting temperature of 50°C prior to cooling. If the **PRE-HEAT** menu is not selected, the apparatus will cool the specimen from ambient temperature without any initial heating. When the cloud point is expected to be higher than 35°C,

select a higher starting temperature according to manufacturer's instructions. The highest starting temperature that can be programmed is 70°C.

11.7 Push the **RUN** button located on the front panel of the apparatus. With the push of this button, the apparatus will allow the flow of liquid cooling medium, if required, (see Note 4) and the flow of purge gas through the apparatus.

NOTE 9—**Caution:** The apparatus will display appropriate warning signals if any of these flows are not properly established. Refer to manufacturer's operating manual for corrective procedures.

11.8 The specimen is heated if specified as described in 11.6. It is then cooled by the Peltier device while the optical detectors continuously monitor the specimen for the appearance of a cloud of wax crystals. The measurement is automatically terminated once the cloud point is detected.

11.9 When the measurement is complete, the automatic cloud point value per Test Method D 5773 will be displayed on the front panel of the apparatus.

11.10 Unlock and open the test chamber lid and clean the specimen out of the specimen cup with a cotton swab.

12. Report

12.1 Report the temperature recorded in 11.9 as the automatic cloud point Test Method D 5773.

12.2 When specified, round the temperature recorded in 11.9 to the next lower integer and report as the Test Method D 2500 equivalent cloud point per Test Method D 5773.

13. Precision and Bias ⁶

13.1 *Precision*—The precision of this test method as determined by the statistical examination of the interlaboratory⁸ test results is as follows:

13.1.1 *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 0.5°C only in one case in twenty.

13.1.2 *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 2.6°C only in one case in twenty.

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*—The results of the interlaboratory program were examined for bias relative to Test Method D 2500/IP219. A statistically insignificant bias of -0.03°C was observed.

13.4 The precision statements were derived from a 1990 interlaboratory cooperative test program.⁸ Participants analyzed 10 sample sets comprised of various distillate fuels and lubricating oils with a cloud point range of -1 to -37°C . Eight laboratories participated with the automatic apparatus and eight

⁶ The results of the 1990 interlaboratory cooperative test program are available from ASTM Headquarters. Request ASTM RR:D02-1373.