



Designation: D8420 – 21

Standard Test Method for Wax Appearance Temperature and Wax Disappearance Temperature of Petroleum Products and Liquid Fuels¹

This standard is issued under the fixed designation D8420; 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 wax appearance temperature and the wax disappearance temperature of petroleum products and liquid fuels by an automatic instrument using optical light scattering detection.

1.2 This test method is applicable to such materials as: crude oil, distillate fuels, residual fuels such as No. 6 Fuel Oil, marine fuels such as VLSFO, and including mixtures of these fuels and liquid biofuels.

1.3 This test method covers the range of temperatures from $-30\text{ }^{\circ}\text{C}$ to $+75\text{ }^{\circ}\text{C}$ with temperature resolution of $0.1\text{ }^{\circ}\text{C}$.

1.4 This test method contains interim precision with repeatability only; a full ILS will be completed within five years of its approval.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 ASTM Standards:²

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.

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

D5773 Test Method for Cloud Point of Petroleum Products and Liquid Fuels (Constant Cooling Rate Method)

D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products, Liquid Fuels, and Lubricants

3. Terminology

3.1 Definitions:

3.1.1 *cloud point, n*—in petroleum products and biodiesel fuels, the temperature of a liquid specimen when the smallest observable cluster of wax crystals first occurs upon cooling under prescribed conditions.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *wax appearance temperature, WAT, n*—when the appearance of wax crystals in the specimen is determined under the conditions of this test method.

3.2.1.1 *Discussion*—The wax appearance temperature in this test method is determined by an automatic instrument using an optical device for detection of the crystal formation.

3.2.2 *wax disappearance temperature, WDT, n*—when the disappearance of wax crystals is determined under the conditions of this test method.

3.2.2.1 *Discussion*—The wax disappearance temperature in this test method is determined by an automatic instrument using an optical device for detection of the crystal formation.

4. Summary of Test Method

4.1 A specimen in a chamber cooled by a Peltier device at a constant rate of $1.5\text{ }^{\circ}\text{C}/\text{min} \pm 0.5\text{ }^{\circ}\text{C}/\text{min}$ while continuously being illuminated by a near infrared (NIR) light source. The specimen is continuously monitored by an array of optical detectors for the appearance of wax crystals. The detectors are sufficient in number to ensure that any solid phase wax crystals that may form are detected. The temperature at which the appearance of wax crystals is detected in the specimen, as determined by the apparatus, is recorded to $0.1\text{ }^{\circ}\text{C}$ resolution as the wax appearance temperature. Once the wax appearance

temperature is detected, when the wax disappearance temperature is desired, the specimen is warmed at a constant rate of $1.5\text{ }^{\circ}\text{C}/\text{min} \pm 0.5\text{ }^{\circ}\text{C}/\text{min}$ while continuously being illuminated by a NIR light source until the crystals in the specimen disappear. The temperature at which the disappearance of the wax crystals is detected in the specimen, as determined by the apparatus, is recorded as the wax disappearance temperature.

5. Significance and Use

5.1 The wax appearance temperature of petroleum products and liquid fuels is an indicator of the lowest temperature of their utility for certain applications. Wax crystals of sufficient quantity can plug filters or impede flow in some fuel systems.

5.2 The wax disappearance temperature of petroleum products and liquid fuels is an indicator of the warmest temperature to remove thermal history. Wax crystals of sufficient quantity can plug filters or impede flow in some fuel systems.

5.3 NIR light scattering technology is useful for recognition of wax crystal formation in dark and opaque fuels, and the cloud point in transparent fuels.

5.4 The wax appearance temperature is an earlier indicator of wax crystal formation than pour point and has better resolution than pour point.

5.5 This test method can determine the temperature of the test specimen at which wax crystals have formed sufficiently to be observed with a resolution of $0.1\text{ }^{\circ}\text{C}$.

6. Apparatus

6.1 *Automatic Apparatus*^{3,4}—The automatic apparatus described in this test method consists of a specimen chamber controlled by a microprocessor that is capable of adjusting a Peltier device for the heating and the cooling of the test specimen, optically observing the appearance and disappearance 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 chamber, optical detector array, NIR 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 $1.5\text{ }^{\circ}\text{C}/\text{min} \pm 0.5\text{ }^{\circ}\text{C}/\text{min}$.

6.4 The temperature measuring device in the specimen chamber shall be capable of measuring the temperature of the test specimen from $-30\text{ }^{\circ}\text{C}$ to $+75\text{ }^{\circ}\text{C}$ at a resolution of $0.1\text{ }^{\circ}\text{C}$.

6.5 The apparatus shall be equipped with a dry air system for transferring the test specimen into the specimen chamber, and drying the specimen chamber.

³ The automatic wax appearance and wax disappearance apparatus is covered by a patent. Interested parties are invited to submit information regarding the identification of an alternative(s) to this patented item to the ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

⁴ The sole source of supply of the apparatus known to the committee at this time is Phase Analyzer Co, 11168 Hammersmith Gate, Richmond, B.C. Canada V7A 5H8. 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.

7. Reagents and Materials

7.1 *Sample syringe (Luer type)*, capable of dispensing 3 mL to 5 mL of sample.

7.2 *Syringe filter (optional)*, $2.7\text{ }\mu\text{m}$ disposable syringe filter (Luer type).

8. Sampling

8.1 Obtain a sample in accordance with Practices [D4057](#) or [D4177](#).

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 should not be heated above $75\text{ }^{\circ}\text{C}$. When the sample is heated above $75\text{ }^{\circ}\text{C}$, allow the sample to cool below $75\text{ }^{\circ}\text{C}$ before filtering or inserting into the apparatus.

8.4 When moisture or particulates, or both, are present in the sample, remove the moisture by a method, such as centrifugation for opaque fuels, or filtration through dry lint-free filter paper for transparent fuels until the oil is clear, but make such filtration at a temperature at least $14\text{ }^{\circ}\text{C}$ above the expected cloud point.

9. Preparation of Apparatus

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

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 Verify the performance of the apparatus at least once per year by determining the wax appearance temperature of a certified reference material (CRM) such as those for cloud point (Test Method [D5773](#)), or other suitable material with a mutually agreed cloud point. For transparent liquids, wax appearance temperature is synonymous with cloud point.

11. Procedure

11.1 When necessary, heat the sample in a bath or oven to a temperature above the expected wax disappearance temperature and maintain this temperature for approximately thirty to sixty minutes to erase the thermal history. Samples with an expected wax appearance temperature (cloud point) above $35\text{ }^{\circ}\text{C}$ or samples which appear solid at room temperature can be heated above $65\text{ }^{\circ}\text{C}$, but should not be heated above $75\text{ }^{\circ}\text{C}$.

11.2 Draw approximately 3 mL to 5 mL of sample into the Luer type sample syringe, when necessary, preheat the syringe to the same temperature as the sample.

11.3 When necessary, attach a $2.7\text{ }\mu\text{m}$ disposable Luer type syringe filter to the sample syringe to remove particulates. Samples with a large concentration of particulates or high viscosity at the preheat temperature should not be forced into the sample syringe or into the analyzer injection port. Remove the metal Luer cap on the analyzer injection port, insert the syringe into the injection port, and deliver from 2.5 mL to 3 mL