



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 $1.5\text{ °C/min} \pm 0.5\text{ °C/min}$ 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 °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 $1.5\text{ °C/min} \pm 0.5\text{ °C/min}$.

6.4 The temperature measuring device in the specimen chamber shall be capable of measuring the temperature of the test specimen from -30 °C to $+75\text{ °C}$ at a resolution of 0.1 °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 °C . When the sample is heated above 75 °C , allow the sample to cool below 75 °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 °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 °C or samples which appear solid at room temperature can be heated above 65 °C , but should not be heated above 75 °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

of bubble-free sample into the analyzer. Remove the syringe and filter, if used, and replace the metal Luer type cap securely to prevent leakage.

11.4 When so equipped, the sample may be delivered to the analyzer with an automatic sample system.

11.5 Program the analyzer according to the manufacturer's instructions. Typical programming could include selection of the expected wax appearance temperature, and whether the wax disappearance temperature is desired in addition to the wax appearance temperature. The specimen temperature is displayed on the user interface of the apparatus. A typical interface is shown in Fig. A1.4.

11.6 Start the operation of the apparatus in accordance with the manufacturer's instructions. The specimen is heated between 65 °C and 75 °C, and is then cooled by the Peltier device while the optical detectors continuously monitor the specimen for the appearance of wax crystals. The temperature when the analyzer determines the wax crystals appearance is then recorded.

11.7 When the wax disappearance temperature is selected, the specimen is re-warmed by the Peltier device while the optical detectors continuously monitor the specimen for the disappearance of wax crystals. The temperature when the analyzer determines the wax crystals disappearance is then recorded.

11.8 When the measurement is complete, the wax appearance temperature, and the wax disappearance temperature when selected, per Test Method D8420 will be displayed on the user interface of the apparatus.

11.9 The apparatus can automatically clean and dry the sample injection chamber and the specimen chamber after the measurement completion.

12. Report

12.1 The test report shall contain at least the following information:

12.1.1 A reference to this standard,

12.1.2 All details necessary for complete identification of the product tested,

12.1.3 Report the temperature recorded in 11.6 as the wax appearance temperature (WAT).

NOTE 1—When the sample is a transparent fuel the wax appearance temperature can be synonymous with the cloud point found by Test Method D5773.

12.1.4 Report the temperature recorded in 11.7 as the wax disappearance temperature (WDT) Test Method D8420 (when selected).

12.1.5 Any deviations, by agreement or otherwise, from the procedures specified, and

12.1.6 The time and date of the test.

13. Precision and Bias

13.1 *Precision*—This test method has interim repeatability precision only. An interlaboratory study of this test method will be conducted and a complete precision statement is expected to be available on or before 2027.

13.2 The repeatability precision of this test method was determined by the statistical evaluation of an interim repeatability study which met the requirements of Practice D6300. Two laboratories measured a total of twenty samples, nine and eleven respectively, five times under repeatability conditions. The samples were ultra-low sulfur distillate and residual marine very low sulfur fuel oil (VLSFO) including the viscosity range of 5.7 mm² to 384.5 mm², the pour point range of 9 °C to 33 °C, the WAT from 34 °C to 60 °C, and the WDT from 41 °C to 75 °C.

13.2.1 *Repeatability*—The difference between two independent results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within short intervals of time would exceed the following value about 5 % of the time (one case in 20 in the long run) in the normal and correct operation of the test method:

WAT Repeatability = 1.96 °C

WDT Repeatability = 2.39 °C

13.2.2 *Reproducibility*—The difference between two single and independent results obtained by different operators applying the same test method in different laboratories using different apparatus on identical test material would exceed the following value about 5 % of the time (one case in 20 in the long run) in the normal and correct operation of the test method:

TBD

13.3 *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.

14. Keywords

14.1 cloud point; constant cooling rate; crude oil; marine fuel; Peltier; petroleum products; residual fuel; thermoelectric; wax appearance temperature; wax crystals; wax disappearance temperature

A1. DETAILED DESCRIPTION OF APPARATUS

A1.1 *Test Chamber*, comprised of optical detectors, lens, light source, specimen chamber, temperature sensor, Peltier device, and heat sink arranged in a configuration as shown in Fig. A1.1. The air trapped in the closed chamber is purged by dry gas. The dry gas inlet and outlet are shown in Fig. A1.1. The test chamber wall is made of black colored metal and plastic components to minimize light reflection.

A1.1.1 *Specimen Chamber*, comprised of black plastic wall and a highly polished metal bottom. The polished surface of the bottom serves as a reflective surface for light. The transfer of heat to and away from the specimen through the metal bottom is controlled by the Peltier device.

A1.1.2 *Temperature Sensor*, reading to 0.1 °C, permanently embedded into the bottom of the specimen cup and positioned less than 0.1 mm below the top surface of the cup bottom. This temperature sensor, which is made of a single strand platinum, provides accurate measurement of the specimen temperature.

A1.1.3 *Peltier Device*, capable of controlling the specimen temperature over a wide range. The range varies depending on the model series. During specimen cooling, heat is transferred from the top of the device to the bottom. Since the top is in thermal contact with the bottom of the specimen cup, the specimen will be chilled. The bottom of the Peltier device is in thermal contact with the heat sink where heat is dissipated. During specimen warming, the reverse process will take place.

A1.1.4 *Light Source*, to provide a beam of light with a wavelength of $940 \text{ nm} \pm 50 \text{ nm}$. The light source is positioned such that it provides an incident beam (Fig. A1.2) impinging onto the specimen at an acute angle. The light is reflected from the polished bottom of the specimen cup. When the specimen is a homogeneous liquid, the reflected beam impinges outside of the lens area onto the chamber lid, which is black in color. The reflected light is then absorbed by the black surface. When wax crystals appear in the specimen, the reflected beam is scattered by the solid-liquid phase boundaries. A significant amount of scattered light impinges onto the lens (Fig. A1.3).

A1.1.5 *Optical Detectors*, positioned above the lens to monitor the clarity of the specimen. The distance between the optical detectors and the lens is adjusted such that the image of the specimen is projected onto the light sensitive surface of the optical detectors. Sufficient optical detectors are used to cover the image area.

A1.2 *Apparatus Exterior Interface*, composed of several displays and buttons as shown in Fig. A1.4 (the exact layout of the displays and buttons may vary depending on the model series).

A1.3 *Sample injection port*, example shown in Fig. A1.5.

A1.4 *Typical apparatus arrangement*, shown in Fig. A1.6.

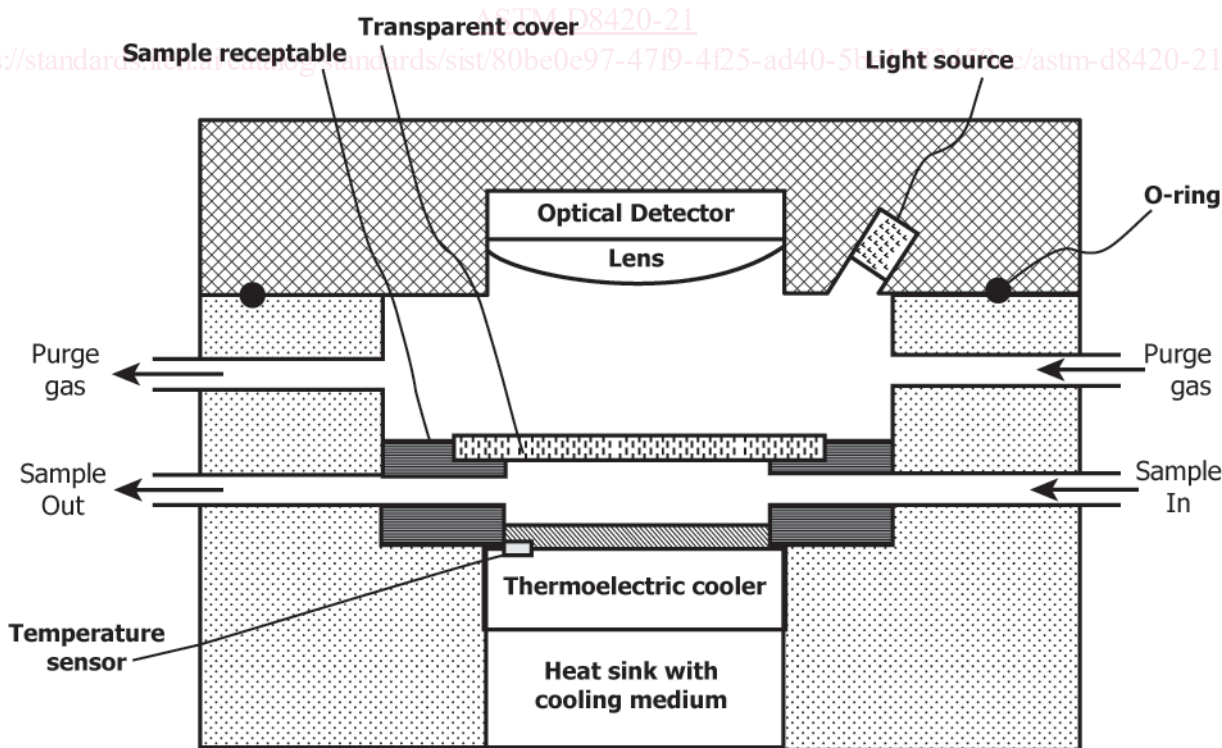


FIG. A1.1 Schematic of Test Chamber