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Technical Association of Pulp and Paper Industry
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Method 1402-Federal Test Method Standard No. 791b
British Standard 4695

Standard Test Method for Melting Point of Petroleum Wax (Cooling Curve)[1]

This standard is issued under the fixed designation D87; 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 melting point (cooling curve) of petroleum wax. It is unsuitable for waxes of the petrolatum group, microcrystalline waxes, or blends of such waxes with paraffin wax or scale wax.

NOTE 1—For additional methods used for testing petroleum waxes, see Test Method D127 and Test Method D938. Results may differ, depending on the method used. For pharmaceutical petrolatum, Test Method D127 usually is used.

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

1.3 *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]

D127 Test Method for Drop Melting Point of Petroleum Wax, Including Petrolatum

D938 Test Method for Congealing Point of Petroleum Waxes, Including Petrolatum

D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance

E1 Specification for ASTM Liquid-in-Glass Thermometers

3. Terminology

3.1 *Definitions:*

3.1.1 *melting point (cooling curve) of petroleum wax*—temperature at which melted petroleum wax first shows a minimum rate of temperature change when allowed to cool under prescribed conditions.

3.1.1.1 *Discussion*—The so-called "American Melting Point" is arbitrarily 1.65°C (3°F) above the Melting Point (Cooling Curve) of Petroleum Wax.

4. Summary of Test Method

4.1 In Procedure A (Manual Method), a specimen of molten wax in a test tube fitted with a thermometer is placed in an air bath, which in turn is surrounded by a water bath held at 16 to 28°C (60 to 80°F). As the molten wax cools, periodic readings of its temperature are taken. When solidification of the wax occurs, the rate of temperature decreases, yielding a plateau in the cooling curve. The temperature at that point is recorded as the melting point (cooling curve) of the sample.

4.2 In Procedure B, an automatic analyzer is used. As the molten wax cools, the sample temperature decrease is measured every 15 s in 0.01°C (0.1°F) readings. The melting point is considered reached when five consecutive measurements are constant within a given temperature interval, usually 0.1°C (0.2°F).

5. Significance and Use

5.1 Melting point (cooling curve) is a test that is widely used by wax suppliers and consumers. it is particularly applied to petroleum waxes that are rather highly paraffinic or crystalline in nature. A plateau occurs with specimens containing appreciable amounts of hydrocarbons that crystallize at the same temperature, giving up heat of fusion, thus temporarily retarding the cooling rate. In general, petroleum waxes with large amounts of non-normal hydrocarbons or with amorphous solid forms will not show the plateau.

6. Apparatus

6.1 The necessary apparatus for Procedure A is described in Annex A1.

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.10.0A on Physical/Chemical Properties.

In the IP, this test method is under the jurisdiction of the Standardization Committee. This test method was adopted as a joint ASTM-IP standard in 1966.

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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.

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

6.2 The automatic instrument consists of a bath (for example, an aluminum block with two measuring locations, two apertures to place the test tubes, and two apertures for the temperature probes). The apparatus may have an accessory digital display and a printer.

6.2.1 The temperature of the bath is maintained between 22 and 26°C (72 and 79°F). A heating device is used to increase the temperature, and a cooling device with cold water circulation is used to decrease the temperature.

6.2.2 The temperature may be monitored with a calibrated thermometer or an electronic temperature measuring device of equivalent precision and accuracy.

6.3 Other types of equivalent automatic apparatus are acceptable.

7. Test Specimen

7.1 Obtain a sample of wax representative of the shipment to be tested. From each test unit obtain a portion of wax weighing at least 25 g for each melting point determination.

8. Procedure A (Manual Method)

8.1 Support the air bath in its proper position in the water bath. Fill the water bath to within 13 mm (½ in.) of the top with water at a temperature of 16 to 28°C (60 to 80°F). The bath temperature is kept within these limits throughout the test.

8.2 Heat the wax sample to at least 8°C (15°F) above its expected melting point (see Note 2). To heat the wax sample use a suitable container in an oven or water bath which is held at a temperature not exceeding 93°C (200°F). Avoid the use of direct heat such as flame or hot plate. Do not keep the sample in the molten state longer than 1 h.

NOTE 2—If no estimate of the melting point is available, heat the wax sample to 10°C (15°F) above the temperature at which the wax is completely molten, or to from 90 to 93°C (195 to 200°F) before proceeding to the next step.

8.3 Fill the test tube to a height of 51 mm (2 in.) with the melted sample. Insert the melting point thermometer through the center of a one-holed stopper, such as a cork so that the 79-mm (3⅛-in.) immersion line is at the lower surface of the stopper. Insert the stopper into the test tube so that the bottom of the thermometer bulb is 10 mm (⅜ in.) from the bottom of the test tube. Support the test tube assembly in the air bath, as shown in Fig. A1.1, while the temperature of the molten wax is still at least 8°C (15°F) above its expected melting point (Note 2).

8.4 Read the melting point thermometer every 15 s. Record each reading to the nearest estimated 0.05°C (0.1°F). Observe the progress of these sequential readings to determine the appearance of the plateau. Identify the plateau as the first five consecutive readings all of which agree within 0.1°C (0.2°F). Discontinue the test after obtaining these five plateau readings.

NOTE 3—If no plateau appears as defined above, the reading procedure is continued until either (*1*) the temperature reached 38°C (100°F) or (*2*) the temperature reaches a point 8°C (15°F) below a temperature where the wax has solidified (as may be observed through a transparent bath). In either of these cases the test is discontinued and the method is judged Not Applicable to the sample (see Note 1 for other methods).

9. Procedure B (Automatic Method)

9.1 Place a clean test tube held in a PTFE holder ring in the aperture provided in the apparatus.

9.2 Insert the temperature probe into a centrally bored, one-holed stopper, and insert it in the test tube. Check the probe height to reach manufacturer's suggested height. Place the stopper with the probe back in the resting holder provided.

9.3 Bring the sample to a temperature at least 8°C (15°F) above the expected melting point. Heat the sample in a 93°C (200°F) maximum temperature water bath.

9.4 Add the molten sample to the test tube to the filling mark. Place the stopper with the probe on the test tube assembly.

9.5 Insert the assembly into the aluminum block aperture, and initiate the analysis in accordance with the manufacturer's instructions.

9.6 When the melting point is detected, the analysis will automatically stop. Per available options on the instrument, the resulting melting point will be displayed on the digital monitor, or printed on a printer, or both.

10. Calculation and Report

10.1 When using a manual apparatus, average the first five consecutive thermometer readings of the identified plateau, which agree within 0.1°C (0.2°F). Correct this average for error in the thermometer scale where necessary.

10.2 The automatic apparatus will average the first five consecutive temperature probe readings within ±0.1°C (±0.2°F).

10.3 Report the result to the nearest 0.05°C (0.1°F) as the Petroleum Wax Melting Point (Cooling Curve), Test Method D87. Also report whether the test was performed manually or using automatic apparatus, as applicable.

11. Quality Control (QC)

11.1 Confirm the performance of the instrument or the test procedure by analyzing a quality control (QC) sample.

11.1.1 When QC/Quality Assurance (QA) protocols are already established in the testing facility, these may be used when they confirm the reliability of the test result.

11.1.2 When there is no QC/QA protocol established in the testing facility, Appendix X1 can be used as the QC/QA system.

12. Precision and Bias [3]

12.1 *Precision*—The precision of this test method as determined by statistical examination of interlaboratory results is as follows:

12.1.1 *Repeatability*—The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

Manual apparatus 0.11°C

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report D02-1617.