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Technical Association of Pulp and Paper Industry
Standard Method T 630m-61
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 or equivalent temperature measuring device 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 change 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 to be 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 exhibit a plateau.

6. Apparatus

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

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

[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.**

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 temperature measuring device through the center of a one-holed stopper, such as a cork. In the case of a thermometer, position the 79-mm (3⅛-in.) immersion line at the lower surface of the stopper. Insert the stopper into the test tube so that the bottom of the thermometer bulb or temperature measuring probe 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 Take a temperature reading every 15 s. Record each reading to at least the nearest 0.05°C (0.1°F). Monitor 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). You may 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 for 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 temperature 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 at least 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 RR:D02-1617.

Automatic apparatus	0.23°C

12.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories 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	0.41°C
Automatic apparatus	0.50°C

12.2 This test method is considered suitable for waxes of a melting point between 38°C (100°F) and 82°C (180°F). These precision values have been obtained by statistical examination of interlaboratory test results from 2005. These values are based on a study among 14 laboratories, using seven paraffin waxes with a melting point range of 52 to 70°C (126 to 158°F).

12.3 *Bias*—The bias of this test method cannot be determined since no generally acceptable standard reference material is available for this analysis.

12.4 *Relative Bias*—The sample set was analyzed using both manual and automatic apparatus. The difference between the two sets of measurements was not very significant. Automatic mode results were biased very slightly low (averaging 0.064 difference).

12.4.1 Based on the comparison of analysis of three sets of wax samples in the melting point range of 115 to 159°F (a total of 70 data points by each method) by both manual and automatic apparatus in one laboratory, a correlation coefficient of r^2 of 0.9999 was obtained indicating that there is no bias between the two modes of measurement.

13. Keywords

13.1 cooling curve; melting point; petroleum wax; wax

ANNEX

(Mandatory Information)

A1. APPARATUS

A1.1 *Test Tube*—A standard glass test tube, 25 mm (1 in.) in outside diameter, and 100 mm (4 in.) in length. It may be marked with a reference line for sample filling at 51 mm (2 in.) above the bottom, and a reference line for positioning of the bottom of the temperature measuring device at 10 mm (⅜ in.) above the bottom.

A1.2 *Air Bath*—A cylinder 51 mm (2 in.) in inside diameter and 114 mm (4½ in.) in depth, equipped to hold the test tube firmly in a vertical position in the center of the air bath. As examples, a tight-fitting cork having a central opening or a metal plate top with a spring clamp that holds the test tube firmly in place have been found suitable to use.

A1.3 *Water Bath*—A suitable cylindrical vessel, 130 mm (5⅛ in.) in inside diameter and 152 mm (6 in.) in depth. Provide a fitted cover equipped to support the air bath vertically so that the sides and bottom of the air bath are surrounded by a layer of water 38 mm (1½ in.) thick. Provide the cover with an opening through which the bath temperature measuring device may be suspended 19 mm (¾ in.) from the outside wall of the water bath.

NOTE A1.1—The air bath, water bath, and water bath cover may be made in one assembly as shown in Fig. A1.1.

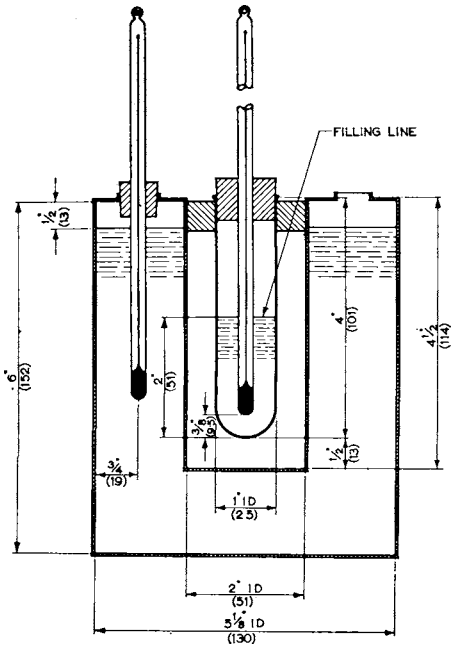
A1.4 *Melting Point Thermometer*—A wax melting point thermometer having the range shown below and conforming to the requirements as prescribed in Specification E1 or in the specifications for IP thermometers:

	Thermometer Number	
Temperature Range	ASTM	IP
38 to 82°C	14C	17C
100 to 180°F	14F	17F

NOTE A1.2—Other means of monitoring the temperature (for example, a resistance temperature detector, in conjunction with a data logger or computer) may be used in place of the specified melting point thermometer, provided that the results are found to be statistically equivalent and that the laboratory making the substitution has collected the necessary data to demonstrate this equivalency.

A1.5 *Bath Temperature Measuring Device*—Any suitable partial immersion thermometer, or other equivalent device, accurate to at least 1.0°C (2°F) throughout the required range.

A1.6 *Timer*—Interval timer or stop watch.



NOTE 1—Dimensions in inches (millimetres).

FIG. A1.1 Apparatus for Determination of Melting Point (Cooling Curve) of Petroleum Wax

APPENDIX

(Nonmandatory Information)

X1. QUALITY CONTROL MONITORING

X1.1 Confirm the performance of the instrument or the test procedure by analyzing a quality control (QC) sample(s).

X1.2 Prior to monitoring the measurement process, the user of the method needs to determine the average value and control limits of the QC sample (see Practice D6299 and MNL7[4]).

X1.3 Record the QC results and analyze by control charts or other statistically equivalent techniques to ascertain the statistical control status of the total testing process (see Practice D6299 and MNL7[4]). (In the absence of explicit requirements given in the test method, this clause provides guidance on QC testing frequency.) Investigate any out of control data for root cause(s). The results of this investigation may, but not necessarily, result in instrument recalibration.

X1.4 The frequency of QC testing is dependent on the criticality of the quality being measured, the demonstrated stability of the testing process, and customer requirements. Generally, a QC sample should be analyzed each testing day with routine samples. The QC frequency should be increased if a large number of samples are routinely analyzed. However, when it is demonstrated that the testing is under statistical control, the QC testing frequency may be reduced. The QC sample testing precision should be periodically checked against the ASTM method precision to ensure data quality (see Practice D6299 and MNL7[4]).

X1.5 It is recommended that, if possible, the type of QC sample that is regularly tested be representative of the material routinely analyzed. An ample supply of QC sample material should be available for the intended period of use, and must be homogenous and stable under the anticipated storage conditions.

X1.6 See Practice D6299 and MNL7[4] for further guidance on QC and Control Charting techniques.

[4] MNL7, *Manual on Presentation of Data Control Chart Analysis*, 6th ed., ASTM International, W. Conshohocken, PA.

SUMMARY OF CHANGES

Subcommittee D02.10 has identified the location of selected changes to this standard since the last issue (D87–07a) that may impact the use of this standard.

(*1*) Revised 4.1, 4.2, 5.1, 8.3, 8.4, 10.1, 10.3, A1.1, A1.3, and A1.5.