

Standard Test Method for Drop Melting Point of Petroleum Wax, Including Petrolatum¹

This standard is issued under the fixed designation D127; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope*

1.1 This test method covers the determination of the drop melting point of petroleum wax. It is used primarily for petrolatums and other microcrystalline wax.

Note 1—Additional methods used for petroleum waxes are Test Method D87 and Test Method D938. Results obtained may differ, depending on the method used. For pharmaceutical petrolatum, Test Method D127 usually is used.

1.1.1 *Test Method A*—The dropping point of wax is determined with a mercury in glass thermometer, as stated below in6.3. (Warning—Mercury has been designated by many regulatory agencies as a hazardous substance that can cause serious medical issues. Mercury, or its vapor, has been demonstrated to be hazardous to health and corrosive to materials. Use Caution when handling mercury and mercury-containing products. See the applicable product Safety Data Sheet (SDS) for additional information. The potential exists that selling mercury or mercury-containing products, or both, is prohibited by local or national law. Users must determine legality of sales in their location.)

1.1.2 Test Method B—The dropping point of wax determined in a dropping point cup in an instrument which detects the drop and measures the temperature electronically, with a platinum thermometer instead of with mercury. Mercury has been recognized as a poison and a health hazard. Removing mercury from laboratories is a way of making the measuring process more inherently safe. The instrumental dropping point method has shown to produce results that are close to those determined by the original Test Method D127, Method A.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.

1.4 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
- D87 Test Method for Melting Point of Petroleum Wax (Cooling Curve)
- D938 Test Method for Congealing Point of Petroleum Waxes, Including Petrolatum
- D3104 Test Method for Softening Point of Pitches (Mettler Softening Point Method)
- D3954 Test Method for Dropping Point of Waxes
- El Specification for ASTM Liquid-in-Glass Thermometers E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
- E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

TEST METHOD A

3. Terminology

3.1 Definitions:

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

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This test method was adopted as a joint ASTM-IP standard in 1964. In the IP, this test method is under the jurisdiction of Standardization Committee.

In 1963, the title, scope, and definition were changed to define the determination of "drop melting point." Sections on procedure, report, and precision were revised, and a new section on significance was added.

In 1964, minor editorial changes and additions to this method were made for its publication as a joint ASTM-IP standard.

Only Method A of this test method is equivalent to IP 133/79 (87).

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.

3.1.1 *drop melting point of petroleum wax*—the temperature at which material becomes sufficiently fluid to drop from the thermometer used in making the determination under definite prescribed conditions.

4. Summary of Test Method

4.1 Specimens are deposited on two thermometer bulbs by dipping chilled thermometers into the sample. The thermometers bearing the specimens are placed in test tubes and heated by means of a water bath until the specimens melt and the first drop falls from each thermometer bulb. The average of the temperatures at which these drops fall is the drop melting point of the sample.

5. Significance and Use

5.1 Melting point is a wax property that is of interest to most wax consumers. It can be an indication of the performance properties of the wax. Drop melting point, Test Method D127, is often used to measure the melting characteristics of petrolatums and other high viscosity petroleum waxes.

6. Apparatus

6.1 *Test Tubes*—Standard test tubes, 25 mm (1 in.) in outside diameter and 150 mm (6 in.) long. The test tubes shall utilize stoppers, such as corks, grooved at the sides to permit air circulation and bored in the center to receive the thermometer.

6.2 *Bath*—A transparent container of not less than 1500 mL capacity, that will permit the immersion of the test tubes to a depth of at least 90 mm and still leave a depth of approximately 15 mm of water below the bottoms of the test tubes.

6.3 *Thermometer*, having a range as shown below and conforming to the requirements as prescribed in Specification E1 or in specifications for IP Standard Thermometers:

	Thermometer Number	
Thermometer Range	ASTM	IP
32 °C to 127 °C	61C	63C
90 °F to 260 °F	61F	

6.4 *Bath Thermometer,* any suitable type, accurate to 0.5 $^{\circ}$ C (1 $^{\circ}$ F) throughout the required range.

7. Procedure

7.1 Secure a sample of sufficient size that is representative of the material under inspection. Use a fresh portion of the sample for each set of two determinations. Melt the sample slowly until the temperature reaches at least 11 °C (20 °F) above the expected drop melting point. Place sufficient sample in a flat bottom container to give a sample depth of 12 mm \pm 1 mm. Adjust the temperature of the sample to at least 6 °C (10 °F) (Note 2) above its drop melting point using any general laboratory thermometer for measurement. Chill one of the test thermometer bulbs to approximately 4 °C (40 °F). Wipe dry, and, quickly but carefully, immerse the chilled bulb vertically into the heated sample until it touches the bottom of the container (about 12 mm submerged) and withdraw it immediately. Hold the thermometer vertically away from the heat until the surface dulls, and then place it for at least 5 min in water having a temperature of 16 °C \pm 1 °C (60 °F \pm 2 °F). Prepare another specimen from the same sample using this procedure.

Note 2—A dipping temperature of 11 °C (20 °F) above the congealing point in accordance with Test Method D938 usually will be 6 °C to 11 °C (10 °F to 20 °F) above the actual drop melting point.

7.2 Securely fix the thermometers in the test tubes by means of suitable stoppers, such as corks, so that the tip of each thermometer is approximately 15 mm above the bottom of its test tube. Insert the test tubes in the water bath which is at 16 °C \pm 1 °C (60 °F \pm 2 °F) and adjust the height of the test tubes so that the immersion marks on the thermometers are level with the top surface of the water. Raise the temperature of the bath at a rate of approximately 2 °C (3 °F)/min to 38 °C (100 °F), then at a rate of approximately 1 °C (2 °F)/min until the first drop of material leaves each thermometer. Record in each case the temperature at which the first drop falls from the thermometer.

8. Report

8.1 Report the average of the two determinations as the drop melting point of the sample under test.

9. Precision and Bias

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

9.1.1 *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:

0.8 °C (1.4 °F)

9.1.2 *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:

1.3 °C (2.4 °F)

NOTE 3—The following information on the precision of this test method was developed by the Institute of Petroleum (now Energy Institute) in London and is being investigated:

(1) Results of duplicate tests should not differ by more than the following amounts:

Repeatability	Reproducibility
1 °C (2 °F)	1.2 °C (2.2 °F)

(2) These precision values were obtained in 1954 by statistical examination of interlaboratory test results.

9.2 *Bias*—The procedure in this test method has no bias because the value of drop melting point can be defined only in terms of a test method.

TEST METHOD B

10. Summary of Test Method

10.1 In this test method, the dropping point is defined as the temperature at which the wax suspended in a cylindrical cup,

with a 2.8 mm diameter hole in the bottom, flows downward and releases a drop as the sample is heated at a constant rate in air. The cups may be nickel plated brass or aluminum. A glass receptacle contains the molten wax underneath the drop.

11. Significance and Use

11.1 This closely defined method may be used as an alternate to Test Method D127 part A to measure the melting characteristics of natural waxes, paraffin waxes, microcrystalling waxes and petrolatums and other high viscosity petroleum waxes.

12. Apparatus

12.1 A METTLER TOLEDO³ dropping point furnace shall be used to determine pitch softening points by this test method. These commercially available instruments consist of a control unit with a digital temperature indicator, with furnace built in

³ The sole source of supply of the apparatus known to the committee at this time is METTLER TOLEDO LLC, 1900 Polaris Parkway, Columbus, OH 43240, 1-800-METTLER, www.mt.com. 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.



