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 D 87 and Test Method D 938. Results obtained may differ, depending on the method used. For pharmaceutical petrolatum, Test Method D 127 usually is used.

1.2 The values stated in SI units are to be regarded as the standard. The values 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:
D 87 Test Method for Melting Point of Petroleum Wax (Cooling Curve)\(^1\)
D 938 Test Method for Congealing Point of Petroleum Waxes, Including Petrolatum\(^2\)

3. Definition

3.1 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 D 127, 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 be supplied with corks grooved at the sides to permit air circulation and bored in the exact 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

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\(^1\) This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of subcommittee D02.10 on Properties of Petroleum Wax.


\(^3\) 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.

\(^1\) Annual Book of ASTM Standards, Vol 05.01.

\(^2\) Annual Book of ASTM Standards, Vol 05.01.

\(^3\) Annual Book of ASTM Standards, Vol 14.03.
depth of at least 100 mm and still leave a depth of 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 E 1 or in specifications for IP Standard Thermometers:

<table>
<thead>
<tr>
<th>Thermometer Range</th>
<th>Thermometer Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>32 to 127°C</td>
<td>61C</td>
</tr>
<tr>
<td>90 to 260°F</td>
<td>61F</td>
</tr>
</tbody>
</table>

6.4 Bath Thermometer, any suitable type, accurate to 0.5°C (1°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 93°C (200°F), or about 11°C (20°F) above the expected drop melting point, whichever is higher. Place sufficient sample in a flat bottom container to give a sample depth of 12 ± 1 mm. Adjust the temperature of the sample to 6 to 11°C (10 to 20°F) (Note 2) above its drop melting point using any general laboratory thermometer for measurement. Chill one of the test thermometer bulbs to 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 (12 mm submerged) and withdraw it immediately. Hold the thermometer vertically away from the heat until the surface dulls, and then place it for 5 min in water having a temperature of 16°C (60°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 D 938 usually will be 6 to 11°C (10 to 20°F) above the actual drop melting point.

7.2 Securely fix the thermometers in the test tubes by means of corks so that the tip of each thermometer is 15 mm above the bottom of its test tube. Insert the test tubes in the water bath which is at 16°C (60°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 (3°F) 1.7°C/min to 38°C (100°F) , then at a rate of 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 has been developed by the Institute of Petroleum (London) and is being investigated:

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

<table>
<thead>
<tr>
<th>Repeatability</th>
<th>Reproducibility</th>
</tr>
</thead>
<tbody>
<tr>
<td>1°C (2°F)</td>
<td>1.2°C (2.2°F)</td>
</tr>
</tbody>
</table>

(b) 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.

10. Keywords

10.1 drop melting point; petrolatum; petroleum wax; wax