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Standard Test Method for Congealing Point of Petroleum Waxes, Including Petrolatum[1]

This standard is issued under the fixed designation D 938; 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 Department of Defense.

ε[1] NOTE—Added thermometer 54C to 6.1 editorially in November 2003.

1. Scope

1.1 This test method covers determination of the congealing point of petroleum waxes, including petrolatum.

1.2 The values stated in inch-pound 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.*

NOTE 1—This test method is an alternative to Test Method D 127. Results obtained are usually lower than the results obtained by Test Method D 127 – IP 133, the amount of the deviation varying with the nature of the petroleum wax.

2. Referenced Documents

2.1 *ASTM Standards:*

D 127 Test Method for Drop Melting Point of Petroleum Wax Including Petrolatum[2]

E 1 Specification for ASTM Thermometers[3]

3. Terminology

3.1 *Definition:*

3.1.1 *congealing point, n— of petroleum wax*, that temperature at which molten petroleum wax, when allowed to cool under prescribed conditions, ceases to flow.

4. Summary of Test Method

4.1 A sample of wax is melted and a droplet is made to adhere to the bulb of a thermometer. Using a prewarmed flask as an air jacket, the droplet on the bulb is allowed to cool at a fixed rate until it congeals. The congealing point is observed as the temperature at which the droplet ceases to flow as the thermometer is turned.

5. Significance and Use

5.1 Congealing point is a wax property that is of interest to many petroleum wax consumers. The procedure described here measures the temperature at which a sample being cooled develops a "set" or resistance to flow. At that temperature, the wax may be at or close to the solid state, or it may be semisolid and quite unctuous, depending on the composition of the wax or petrolatum being tested. In the case of petrolatums, congealing property is associated with the formation of a gel structure as the sample cools.

6. Apparatus

6.1 *Thermometer*, having the following range and conforming to the requirements prescribed in Specification E 1 or in the specifications for IP Standard Thermometers:

	Thermometer Number	
Temperature Range	ASTM	IP
20 to 100°C	54C	18C
68 to 213°F	54F	18F

6.2 *Erlenmeyer Flask*, 125-mL, glass, to serve as a thermometer jacket.

6.3 *Cork or Rubber Stopper*, for attaching the thermometer to the Erlenmeyer flask.

[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 D02.0M on Petroleum Wax.

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[2] *Annual Book of ASTM Standards*, Vol 05.01.

[3] *Annual Book of ASTM Standards*, Vol 14.03.

7. Procedure

7.1 Adjust the thermometer through the stopper so that the bottom of the bulb will be 10 to 15 mm above the bottom of the Erlenmeyer flask when the stopper is fitted snugly in the flask. After making this adjustment, remove the thermometer and stopper from the flask, being careful not to change the position of the stopper relative to the thermometer stem.

7.2 Place approximately 50 g of sample, which is representative of the material under inspection, in a porcelain evaporating dish or other suitable container.

7.3 Place the empty Erlenmeyer flask (without the thermometer assembly) and the container holding the specimen in a temperature-controlled oven set at 99 ± 3°C (210 ± 5°F) until the specimen and the flask reach oven temperature.

NOTE 2—For nonreferee, routine testing of samples known to have low congealing points, the oven may be set at a lower temperature, but it must be at least 11°C (20°F) above the expected congealing point of the sample.

7.4 Remove the specimen from the oven and completely immerse the thermometer bulb in it without covering any part of the thermometer stem with specimen. Gently stir the specimen with the thermometer until the mercury column has stopped rising.

7.5 While holding the thermometer bulb in the molten wax specimen, remove the heated flask from the oven, using a towel or gloves to protect the hands. Now carefully remove the thermometer from the specimen, taking care to retain a relatively large drop of specimen adhering to the bulb. Holding the thermometer in a horizontal position, firmly fit the thermometer and stopper into the flask. Keep the assembly in a horizontal position.

7.6 While observing the drop on the thermometer bulb at an eye level position, rotate the thermometer and flask about a horizontal axis. Use a steady and even rate for each continuous full revolution, and complete each revolution in not less than 2 s, nor more than 3 s. Do not pause at the completion of each revolution any longer than required to reindex the fingers for the next full and continuous rotation (Note 3). When the drop is observed to rotate with the bulb, immediately read the thermometer to the nearest 0.25°C (0.5°F) and record this determination. Make a repeat determination on the wax specimen. If the variation of these two determinations does not exceed 1°C (2°F), record the average of these determinations as the congealing point of the specimen under test. If the variation of two determinations is greater than 1°C (2°F), make one additional determination and record the average of the three determinations as the congealing point.

NOTE 3—Operators should periodically check themselves for compliance with this turning rate. The brief pause time is not to be included in the 2 to 3-s rotation time.

8. Report

8.1 Report the average of the multiple determinations as the congealing point, shown to the nearest 0.25°C (0.5°F).

9. Precision and Bias

9.1 The precision of this test method is not known to have been obtained in accordance with RR:D02-1007 "Manual on Determining Precision Data for ASTM Methods on Petroleum Products."

9.2 The precision of this test method as determined by statistical examination of interlaboratory results is as follows:

9.2.1 *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:

Distillate waxes	0.5°C (1.0°F)
Residual waxes, including petrolatums	1.0°C (2.0°F)

9.2.2 *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:

Distillate waxes	1.5°C (2.5°F)
Residual waxes, including petrolatums	2.5°C (4.5°F)

9.3 *Bias*—The procedure in this test method for measuring the congealing point of petroleum waxes has no bias because the value of the congealing point can be defined only in terms of a test method.

10. Keywords

10.1 congealing point; petrolatum; petroleum waxes