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.

Designation: D127 – 19

Designation: 133/79 (87)

Standard Test Method for Drop Melting Point of Petroleum Wax, Including Petrolatum[1]

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

E1 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:*

[1] This 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 on Properties of Petroleum Waxes and Alternative Wax-like Materials.

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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.

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

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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 °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 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 ± 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 ± 1 °C (60 °F ± 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 ± 1 °C (60 °F ± 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,

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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 TOLEDO[3] 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.

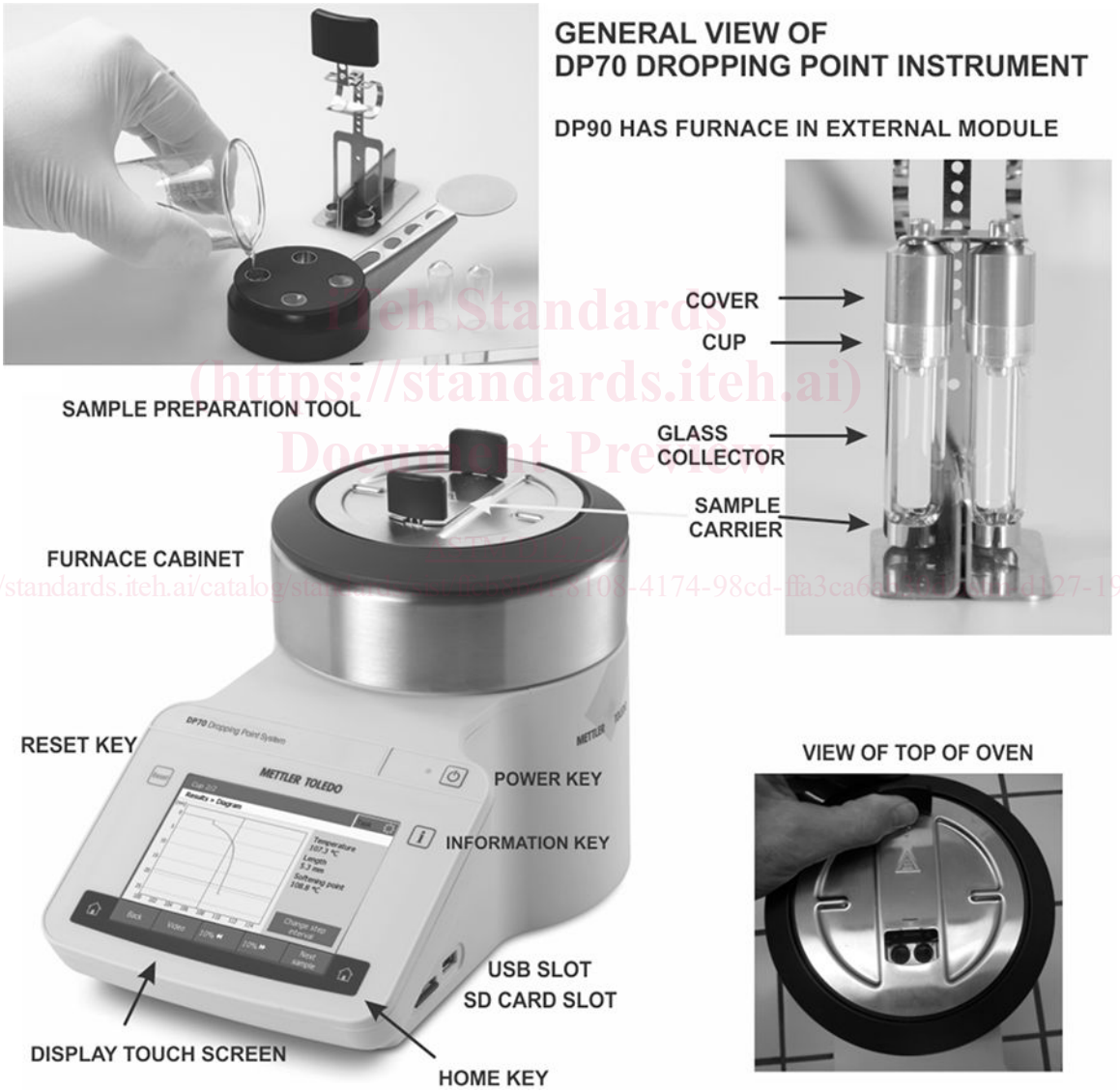

FIG. 1 Overview of DP70 Dropping Point Instrument

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or attached, sample cartridges, and accessories. The control unit automatically regulates the heating rate of the furnace. The dropping point is indicated on the readout, and the heating program stopped, when the sample flow triggers a dropping point detection. A general view of the contents of the METTLER TOLEDO dropping point instrument is shown in Fig. 1.

13. Procedure

13.1 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. The sample should be completely melted. The wax should be hot enough that the wax, when poured, remains transparent enough to see the opening in the bottom of the cup when the cup is poured full. This ensures that no air pockets are trapped in a solidifying wax that is not hot enough. (See Note 4.) The cup should be poured full to the top plus 1 mm to 2 mm above the rim. As the wax cools in the cup, it cools from the outside in and there is shrinkage in the center. After cooling, any wax remaining above the top of the cup should be trimmed off with a flat sharp edge. Any wax on the outside of the cup should be cleaned off. The cup should temper at room temperature (20 °C to 25 °C) for 2 h to allow the wax time to harden. If the wax is too hot when poured, there will be more shrinkage as it cools, and this will cause a cavity down in the center. Pouring more wax in after it hardens is not recommended because an air pocket can be trapped in the cavity. A small cavity is almost unavoidable for some waxes but this should not cause a problem so long as the cup is otherwise full.

NOTE 4—Center cavities are caused by the outside of the cup cooling and hardening before the center. This can be minimized by heating the sample preparation tool to near the expected drop temperature before pouring into the dropping point cups. The cooling becomes more uniform and the cavity less pronounced.

13.2 Petrolatums should be dropped into ice water after they have cooled and gelled so that they retain the shape. The cup should sit in the ice water for at least one hour.

13.3 The exterior of the cup is cleaned of any wax and water, the cap is put on and the glass receptacle fitted to the bottom and held. It is then inserted in the sample holder. Usually samples are analyzed in duplicate, as the sample holder holds two cups. The start temperature should be at least 15° below the expected drop temperature, with a 120 s wait time before the temperature ramp of 1 °C/minute is started. When both cups have dropped, the temperature should return to the insert temperature and the results presented on the display. The video of the drop can be examined if there is any doubt about the drop result, and the report can be viewed to see a photo of the drop at the moment it fell.

13.4 *Illustrations:*

13.4.1 Fig. 2—The dropping point cups have been recently poured. The lower and right cups have been cooling for several minutes. The top cup was just poured. The bottom can be seen through the transparent molten wax, ensuring that there are no entrapped air pockets. The left cup has not been poured. In the right cup, the center is still molten. These cups were poured with as much wax as possible over the level of the cup in order to minimize the cavity that forms in the center, after the excess wax is cut off.

13.4.2 Fig. 3—The four cups from Fig. 2 have been poured and cooled. The two cups sitting on top did not get enough wax. Cavities extend way down in the center of those. The one on the left had a dropping point of 0.5° lower than the one on the right. More wax should be poured to begin with, as is evident in the samples in the holder. These will all need trimming, but they will have a small cavity in the center that does not materially influence the dropping point.

13.4.3 Fig. 4—A cup with wax after the drop. Note the hanging solidified drop and the pool of wax in the bottom of the receiver glass.

13.4.4 Fig. 5—Two cups in the holder shortly after removal from the DP70, showing the drops and wax in the bottom of the receiver glass.

13.4.5 Fig. 6—The photo from the report with at the moment of the drop on the right side at 62.2 °C. The sample on the left had already dropped at 61.8 °C. The second drop is about to fall.

14. Report

14.1 Report both determinations and the average of the two determinations as the drop melting point of the sample under test.

14.1.1 The PDF report generated on a USB stick or transferred to an Ethernet connected PC may be examined for photos of the drop to verify that the drop occurred correctly. See Fig. 6.

15. Precision and Bias

15.1 The precision of this test method is based on an interlaboratory study of ASTM D127 – 08 (2014), Standard Test Method for Drop Melting Point of Petroleum Wax, Including Petrolatum, conducted in 2014. Eleven laboratories tested a total of nineteen different wax samples. Every "test result" represents an individual determination. All labs were asked to report duplicate test results for every material tested. Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report No. RR:D02-1921.[4]

15.1.1 *Repeatability Limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "*r*" value for that material; "*r*" is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

15.1.1.1 Repeatability limits are listed in Table 3.

15.1.2 *Reproducibility Limit (R)*—Two test results shall be judged not equivalent if they differ by more than the "*R*" value for that material; "*R*" is the interval representing the critical

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1921. Contact ASTM Customer Service at service@astm.org.

FIG. 2 Four Recently Poured Dropping Point Cups

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difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

15.1.2.1 Reproducibility limits are listed in Tables 1-3.

15.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

15.1.4 Any judgment in accordance with statements 15.1.1 and 15.1.2 would have an approximate 95 % probability of being correct.

15.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

15.3 The precision statement was determined through statistical examination of all reported results, from eleven laboratories, on nineteen materials. These nineteen materials were identified generically in the tables above. ÷DSC analysis showed in general that waxes with a narrow melting range had a consistent dropping point with small variation. Waxes with a broad melting range, such as the plant wax blends, showed much greater variability in the dropping point by both methods A and B.

15.4 To judge the equivalency of two test results, it is recommended to choose the material closest in characteristics to the test material.

16. Keywords

16.1 drop melting point; petrolatum; petroleum wax; wax

FIG. 3 Four Dropping Point Cups after Pouring and Cooling

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