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: D3954 - 15 (Reapproved 2022)

Standard Test Method for Dropping Point of Waxes¹

This standard is issued under the fixed designation D3954; 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 ASTM dropping point for waxes.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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

D566 Test Method for Dropping Point of Lubricating Grease

3. Summary of Test Method log/standards/sist/7c0a1f80-

3.1 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 a distance of 19 mm to interrupt a light beam as the sample is heated at a constant rate in air.

4. Significance and Use

4.1 Waxes do not go through a sharp solid-liquid phase change when heated and therefore do not have a true melting point. As the temperature rises, waxes gradually soften or become less viscous. For this reason, the determination of the softening point must be made by an arbitrary but closely defined method if test values are to be reproducible.

4.2 This test is useful in determining the consistency of waxes, and as one element in establishing the uniformity of shipments or source of supply.

4.3 This test method has been found suitable for all types of waxes including paraffin, microcrystalline polyethylene, and natural waxes.

5. Apparatus

5.1 Suitable apparatus that meets the requirements of 5.1.1, 5.1.2, and 5.1.3 can be used to determine dropping points by this test method. Instruments are available commercially³ consisting of a control unit with a digital temperature recorder, matched furnace, sample cartridges, and accessories. The control unit automatically maintains the furnace temperature and controls the heating rate with a precision of 0.1 °C. The dropping point is automatically recorded, and the furnace heating program is turned off when the sample interrupts the light beam and triggers the photocell detector.

5.1.1 Control Unit—This unit shall provide a continuous linear temperature control from 25 °C to 250 °C at a 2 °C/min rate. A digital readout shall record the softening point with an accuracy of 0.1 °C.

5.1.2 *Furnace Unit*—This unit shall be capable of heating a sample cup assembly as described in 5.1.3 at $2 \degree C \pm 0.3 \degree C/min$ linear rate from 25 °C to 250 °C. It shall include a sensing system capable of detecting the softening point with an accuracy of 0.1 °C.

5.1.3 *Sample Cup Assembly*—A chromium-plated brass cup conforming to the dimensions shown in Test Method D566. It shall be placed in an assembly so that the sample flows down a distance of 19 mm to interrupt a light beam to cause digital display of the softening point.

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¹ This test method is under the jurisdiction of ASTM Committee D21 on Polishes and is the direct responsibility of Subcommittee D21.02 on Raw Materials.

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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.

³ To the knowledge of the committee at this time, the only instruments which satisfy the requirements of 5.1.1, 5.1.2, and 5.1.3 are Mettler-Toledo model DP70 and model DP90 fitted with FP83HT sample cup. These are available from Mettler-Toledo, LLC, 1900 Polaris Parkway, Columbus, OH 43240, 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.

6. Preparation of Sample

6.1 For waxes heat the sample to 15 °C to 20 °C above its melting point to form a pourable liquid. Place the sample cups on glass slides and pour the melted sample into the cup to a level even with the upper rim of the cup. Allow the sample to stand at room temperature for 2 h before running.

7. Preparation of Apparatus

7.1 Ensure that the furnace unit and the sample cup assembly are clean and bright, since tarnished or dirty apparatus will change the apparent dropping point. After each determination, check to see if the apparatus requires cleaning.

NOTE 1-It is advisable to keep the equipment covered when not in use.

8. Procedure

8.1 The procedure for measuring the dropping point of waxes with the Mettler instrument has been developed to duplicate the results obtained by Test Method D566.

8.2 Preheat or cool the furnace unit to a temperature of 20 °C to 25 °C less than the expected dropping point (Note 2) of the sample and maintain at this temperature. Place the cartridge assembly containing the sample in position in the furnace, taking care that the slits for the light beam are properly positioned. When the ready light becomes steady, indicating the sample and furnace have equilibrated at the present temperature, initiate the 2 °C/min heating rate by pressing the START LEVEL. Heating will then continue automatically until the drop point occurs and the dropping point temperature is displayed on the digital readout.

NOTE 2—In the event of a dispute, the purchaser and the seller should agree on the exact starting temperature to be used.

8.3 Immediately remove the cartridge assembly upon completion of the test. Check to determine if the sample has passed the light beam slot and no pretrigger has occurred. Inspect the dropping point apparatus to be sure no dirt, particles, or residue remain.

8.4 Clean the sample cups by placing them upside down on a hard surface and punching out the residue material. Use a spatula shaped to the contours of the cup, to remove the remaining particles with a gentle twirling motion.

9. Report

9.1 Report the dropping point recorded on the digital readout to the nearest 0.1 °C. If converted to degrees Fahrenheit, report to the nearest 0.2 °F. Experience has indicated that duplicate runs are not necessary. If a known error in experimental procedure is made, the result should be discarded and a second run should be made.

10. Precision

10.1 The following criteria shall be used for judging the acceptability of results (95 % probability) for the Mettler dropping point:

10.1.1 *Repeatability*—Duplicate results by the same operator shall not be considered suspect unless they differ by more than 0.5 $^{\circ}$ C.

10.1.2 *Reproducibility*—The results reported by each of two laboratories shall not be considered suspect unless the reported values differ by more than $1.5 \,^{\circ}$ C.

10.1.3 The procedure in this test method has no bias because the values derived for the drop point of waxes are defined only in terms of this test method.

11. Keywords

11.1 drop point; waxes

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