



# Standard Test Method for Dropping Point of Waxes<sup>1</sup>

This standard is issued under the fixed designation D 3954; 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 ( $\epsilon$ ) 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 *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 566 Test Method for Dropping Point of Lubricating Grease<sup>2</sup>

D 2265 Test Method for Dropping Point of Lubricating Grease Over Wide Temperature Range<sup>2</sup>

## 3. Summary of Test Method

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

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 05.01.

this test method. Instruments are available commercially<sup>3</sup> 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 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°C  $\pm$  0.3°C/min linear rate from 25 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 D 566. 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.

## 6. Preparation of Sample

6.1 For waxes heat the sample to 15 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 D 566.

8.2 Preheat or cool the furnace unit to a temperature of 20 to

<sup>3</sup> Mettler FP5/53 model, FP80/83 model, and FP90/83 model are available from the Mettler Instrument Corp., Princeton-Hightstown Road, Hightstown, NJ 08520.

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°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°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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