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Standard Test Method for Melting Point of Waxes[1]

This standard is issued under the fixed designation F766/F766M; 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.

ε[1] NOTE—Units information was editorially revised in December 2010.

1. Scope

1.1 This test method covers the determination of the initial and final melting points and recovery point of waxes using a hot stage and microscope.

1.2 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the 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 and health practices and determine the applicability of regulatory limitations prior to use.* See Section 8 for a specific precautionary statement.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E105 Practice for Probability Sampling of Materials

E122 Practice for Calculating Sample Size to Estimate, With Specified Precision, the Average for a Characteristic of a Lot or Process

3. Descriptions of Terms Specific to This Standard

3.1 *final melting point*—the temperature in degrees celsius observed on the hot-stage thermometer at the time the wax is completely melted into droplets.

3.2 *initial melting point*—the temperature in degrees celsius observed on the hot-stage thermometer at the time the first movement is noted in the wax.

3.3 *recovery point*—the temperature in degrees celsius observed on the hot-stage thermometer at the time the first crystal is observed forming in the melted wax during cooling.

4. Summary of Test Method

4.1 A few shavings or granules from representative wax sample are placed on a microscope slide and covered with a cover glass. The slide assembly is set up on the hot stage with the microscope lighted and focused for best viewing. The hot stage is heated rapidly at first and then adjusted so that its temperature will increase approximately 2°C/min during the last 10°C preceding the expected melting point. The wax shavings or granules are observed through the microscope, and the temperature at which first movement of wax is noted is recorded as initial melting point. The temperature at the time the wax is observed to be completely melted is recorded as the final melting point. The hot stage is slowly cooled and the temperature at which the first crystal appears in the melted wax is recorded as the recovery point. The recorded temperatures are adjusted, where applicable, by appropriate temperature correction factors.

5. Significance and Use

5.1 This test method determines the initial melting point and melting range of waxes. The data obtained are important in the application of waxes in one-time carbon inks.

6. Apparatus

6.1 *Controlled Micro Hot Stage and Microscope,* 40 to 50× power or suitable alternative.

6.2 *Microscope Slides,* 1 by 1½ in.

6.3 *No. 2 Cover Slides* (glasses), 18 mm square.

[1] This test method is under the jurisdiction of ASTM Committee F05 on Business Imaging Products and is the direct responsibility of Subcommittee F05.02 on Inked Transfer Imaging Products.

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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.

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7. Materials

7.1 *Test Reagent Set,* containing substances with known melting points for checking the accuracy of the hot-stage thermometer[3].

8. Precautions

8.1 Not all waxes exhibit a uniform flow. Some waxes exhibit a sharp initial flow while others may exhibit a slower, gradual plastic flow.

9. Sampling

9.1 Ensure that the sample represents the material tested and that the portion of the test sample is representative of the whole sample. (Practices E122 and E105.)

9.2 Prepare sufficient wax to give 10 ± 2 mg of sample for each melting point determination. Brittle wax (such as carnauba) is prepared by grinding the sample in a mortar and pestle or other suitable means and passing the wax through a 200-mesh sieve. Non-brittle wax (such as paraffin) is prepared by shaving off a few small particles with a razor or other sharp instruments.

10. Calibration

10.1 Check the hot-stage apparatus and thermometer regularly with test reagents having known melting points. To ensure maximum accuracy, record at least three readings over the range of the thermometer utilized and average each group of readings to obtain temperature corrections. Record any temperature corrections for the thermometer tested.

10.2 The heat control dial of the hot-stage apparatus is generally graduated. The temperature gradient produced by the various settings of the control must be predetermined to maintain a specific heating rate when determining melting point. Periodically check these temperature gradients and record any revisions regarding the heat control dial heating rate.

[3] The sole source of supply of the apparatus known to the committee at this time is Thomas Scientific, PO Box 99, Swedesboro, NJ 08085. 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.

11. Procedure

11.1 Using a spatula, transfer 10 ± 2 mg of the prepared test wax onto a microscope slide.

11.2 Cover the wax sample with a cover glass and carefully place the sample assembly on the hot stage. Follow the manufacturer's instructions for the instruments being used.

11.3 Adjust the microscope lamp or the mirror, or both, for best viewing. Focus the microscope using 40 to 50× power to bring the selected wax sample into sharp detail.

11.4 Turn on the hot-stage heat control and set the control so that the stage will heat to within 10°C of the expected melting point of the sample at a rate of approximately 3°C/min.

11.5 When the temperature of the stage reaches to approximately 10°C of the expected melting point, reset the heat control so that the temperature of the stage increases approximately 2°C/min.

11.6 Observe the wax through the microscope and record the temperature of initial (first movement of wax) and final (wax completely melted) melting points.

11.7 Rotate the ocular for maximum definition and continue observing the melted wax. Reduce the heat control setting and record the temperature at which the first crystal appears as the recovery point.

12. Calculation and Results

12.1 Correct the observed temperature in accordance with the thermometer calibration and report to the nearest 1°C.

13. Precision

13.1 *Repeatability*—Duplicate determinations (one operator and apparatus) on uniform samples should not differ by more than 2°C.

13.2 *Reproducibility*—Melting point differences between separate testing facilities using different manufacturers' test instruments could exist because of variability between these instruments.

14. Keywords

14.1 melting; melting point; waxes

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