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Standard Test Method for Melting Point of Waxes[1]

This standard is issued under the fixed designation F 766; 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—Keywords were added in February 2000.

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 *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:*

E 105 Practice for Probability Sampling of Materials[2]

E 122 Practice for Choice of Sample Size to Estimate a Measure of Quality for a Lot or Process[2]

3. Descriptions of Terms Specific to This Standard

3.1 *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.

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

3.3 *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[3] or suitable alternative[4].

6.2 *Microscope Slides*, 1 by 1½ in.

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

7. Materials

7.1 *Test Reagent Set*, containing substances with known melting points for checking the accuracy of the hot-stage thermometer[5].

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 E 122 and E 105.)

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[1] This test method is under the jurisdiction of ASTM Committee F-5 on Business Copy Products and is the direct responsibility of Subcommittee F05.02 on Carbon Paper and Inked Ribbons.

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[2] *Annual Book of ASTM Standards*, Vol 14.02.

[3] A Kofler Micro Hot Stage, available from Arthur Thomas Co., Philadelphia, PA, has been found suitable for use. All equipment necessary for this test including the microscope and the variable transformer are included.

[4] The Fisher-Johns instrument, operated in accord with the manufacturer's instructions and used with a microscope of 40 to 50× power, has also been found suitable for use.

[5] Also available from Arthur Thomas Co., Philadelphia, PA.

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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.

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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