Designation: D 3944 – 88 (Reapproved 2000)

An American National Standard

Standard Test Method for Solidification Point of Petroleum Wax[1]

This standard is issued under the fixed designation D 3944; 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 describes a procedure for rapidly determining the solidification point of petroleum wax.

NOTE 1—This test method is also applicable to similar materials such as synthetic waxes but the precision may vary.

1.2 The values in acceptable metric units are to be regarded as the standard. The values in parentheses are for information only.

1.3 *This standard does not purport to address all of the safety problems, 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 87 Test Method for Melting Point of Petroleum Wax (Cooling Curve)[2]

D 127 Test Method for Drop Melting Point of Petroleum Wax Including Petrolatum[2]

D 938 Test Method for Congealing Point of Petroleum Waxes Including Petrolatum[2]

E 1 Specification for ASTM Thermometers[3]

3. Terminology

3.1 Definition:

3.2 *solidification point of petroleum wax*—that temperature in the cooling curve of the wax where the slope of the curve first changes significantly as the wax sample changes from a liquid to a solid state.

4. Summary of Test Method

4.1 A 50-mg sample of wax is placed in a test tube at ambient temperature and heated above the solidification point of the wax sample. A thermocouple probe, attached to a recorder, is inserted into the wax sample, which is allowed to cool at room temperature. The thermocouple response of the cooling wax traces a curve on the chart paper of the recorder. The first significant change in the slope of the curve is the solidification point.

5. Significance and Use

5.1 The related methods of determining the melt point of petroleum wax are relatively time-consuming. This method endeavors to reduce the duration of testing significantly and at the same time maintain a reasonable precision. This method can be useful for quality control of petroleum waxes as well as research and product development work on these waxes.

5.2 For methods used for testing melt points of petroleum waxes, see Tests Method D 87, D 127, including Petrolatum and Test Method D 938.

6. Apparatus

6.1 *Thermocouple*, with an iron-constantan junction.[4]

6.2 *Recorder* capable of recording voltage and equipped with a time-base module. The recorder should have the following minimum specifications:

6.2.1 *Span*, 0 to 10 mV or other suitable ranges.

6.2.2 *Accuracy*, 0.25 % of full scale.

6.2.3 *Step Response Time*, 1-s full scale, 3-s full scale is also appropriate.

6.2.4 *Zero Junction/Reference Junction/Temperature Compensated Junction*—Must be included.

6.3 *TFE-Fluorocarbon Holder Adapter*—See Fig. 1 and Fig. 2.[5]

6.4 *TFE-Fluorocarbon Disk Centering Guide*—See Fig. 1 and Fig. 2.[5]

6.5 *Test Tubes*, 6 by 50-mm.

6.6 *Vial*, 25 by 52-mm.

6.7 *Apparatus for Calibrating Temperature Recorder*:

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricantsand is the direct responsibility of Subcommittee D02.10 on Properties of Petroleum Wax (Joint ASTM – TAPPI).

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[2] *Annual Book of ASTM Standards*, Vol 05.01.

[3] *Annual Book of ASTM Standards*, Vol 14.03.

[4] Suitable thermocouples are available from: Claud S. Gordon Co., 5710 Kenosha St., Richmond, IL 60071, (815) 678-2211.

For "J" (iron-constantan) junction the following is suitable: Xactpak Type MM Assembly, Catalog No. 402-1101.

Junction: grounded (*G*)

Transition fitting: TH 2780-020

Thermocouple wire: J30-1-305

L (length of metal sheath)

E (lead length): specify length desired.

[5] Not commercially available and hence must be made in a machine shop or elsewhere.

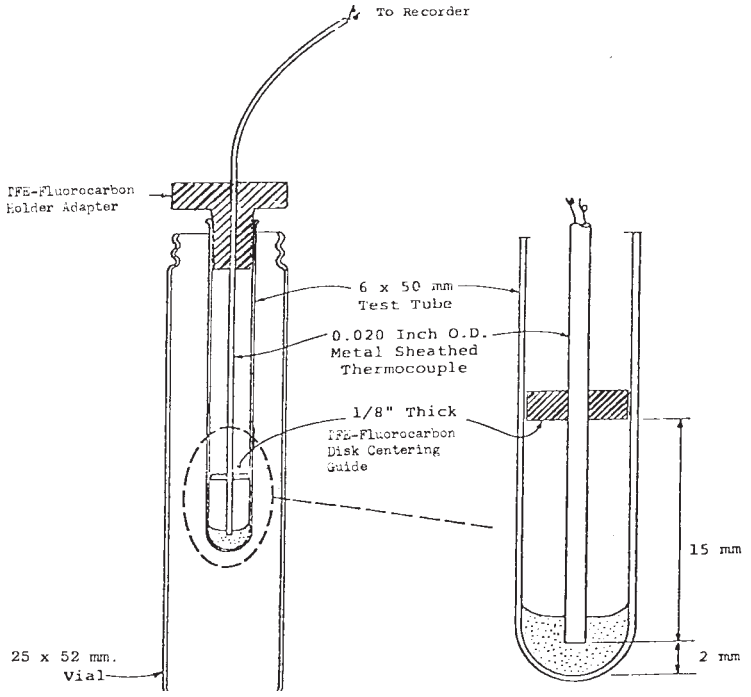

FIG. 1 Solidification Point Apparatus

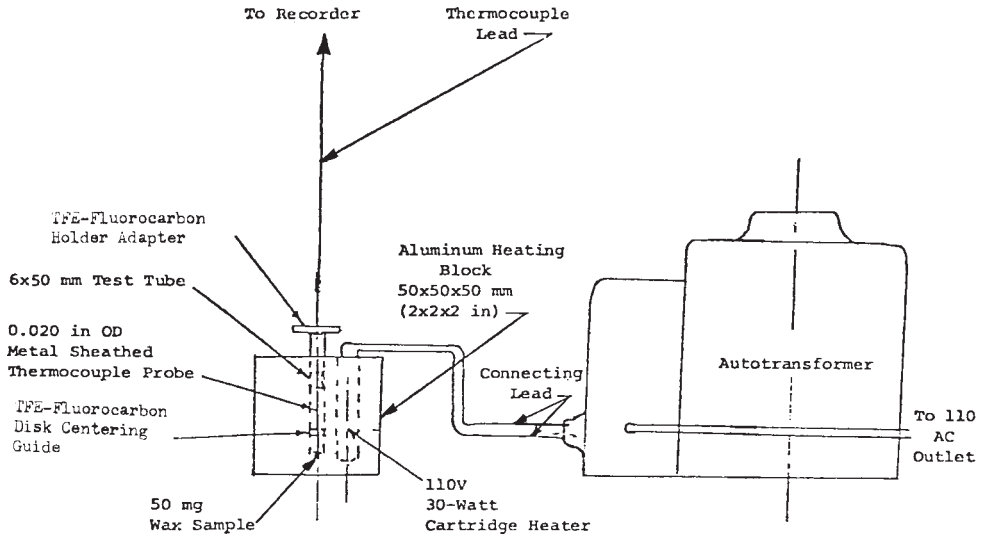

FIG. 2 Apparatus for Heating Wax Sample

6.7.1 *Stainless Steel Beaker*, 1000-mL.

6.7.2 *Heating Mantle*, to fit 6.7.1.

6.7.3 *Autotransformer* to control heat to 6.7.2.

6.7.4 *Variable-Speed-Stirrer*.

6.7.5 Thermometer, ASTM No. 61C or 61F, 79-mm immersion.

6.8 *Methods for Heating Specimen*:

6.8.1 *Hot Air Blower* at 1000 W, 1200 W, or other suitable power. This could be a laboratory or a household hair dryer type.

6.8.2 *Aluminum Heating Block*, about 50 by 50 by 50 mm. In the center of one face of the block, a hole is made 7 mm in diameter and 37 mm deep to accommodate a 6 by 50-mm test tube and another hole adjacent to it to accommodate a 110-V, 30-W cartridge heater, about 9 by 38 mm, commercially available (see Fig. 2).

6.8.2.1 *Autotransformer*, to control heat in 6.8.2.

7. Procedure

7.1 Calibrate the recorder at least every 60 days when in frequent use (see Annex A1).

7.2 Obtain a wax sample representative of the material to be tested.

7.3 Using a balance accurate to at least 1 mg, weight 50 + 5 mg of sample by putting a few tiny pieces of solid wax into a tared 6 by 50-mm test tube.

7.4 Start the temperature recorder. A horizontal pen speed of about 150 mm (5 in.)/10 min for a *X-Y* recorder or a chart speed of about 150 mm (5 in.)/10 min for a strip chart recorder is usually appropriate.

7.5 Heat the sample by any convenient method, such as by use of:

7.5.1 A hot air blower.

7.5.2 An aluminum heating block.

7.6 When using any method of heating a sample, note that the wax absorbs heat slowly. Hence caution must be used not to overheat the surface near the heating medium. Overheated wax can degrade. A minute amount of degradation affects results.

7.7 When the sample melts, insert the thermocouple probe into the melted wax.

7.8 Let the wax sample temperature rise 10 to 15°C above the expected solidification point.

7.9 Transfer the 6 by 50-mm test tube, together with its heated specimen, from the heating medium to a 25 by 52-mm vial which acts as a cooling chamber (See Fig. 1).

7.10 Let the specimen cool until the curve traced by the cooling wax on the recorder chart paper levels off.

7.11 Determine the point in the curve which shows the first significant change in the slope of the curve (see Fig. 3). This is the solidification point.

8. Report

8.1 Report the solidification point to the nearest 0.1°C.

9. Precision and Bias [6]

9.1 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 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:

Distillate waxes	0.6° C (1.0 °F)
Residual waxes	0.7 °C (1.3 °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:

Distillate waxes	1.2 °C (2.2 °F)
Residual waxes	2.4 °C (4.3 °F)

9.2 The precision data were obtained in an interlaboratory study involving five laboratories in which solidification points of five distillate waxes (51 to 69 °C/123 to 155 °F) and five residual waxes (53 to 85 °C/128 to 185 °F) were determined.

9.3 *Bias*—The procedure in this test method has no bias because the value of solidification point can be defined only in terms of a test method.

10. Keywords

10.1 petroleum wax; solidification print; wax

[6] Supporting data are filed from ASTM Headquarters and may be obtained by requesting RR:D02-1133.

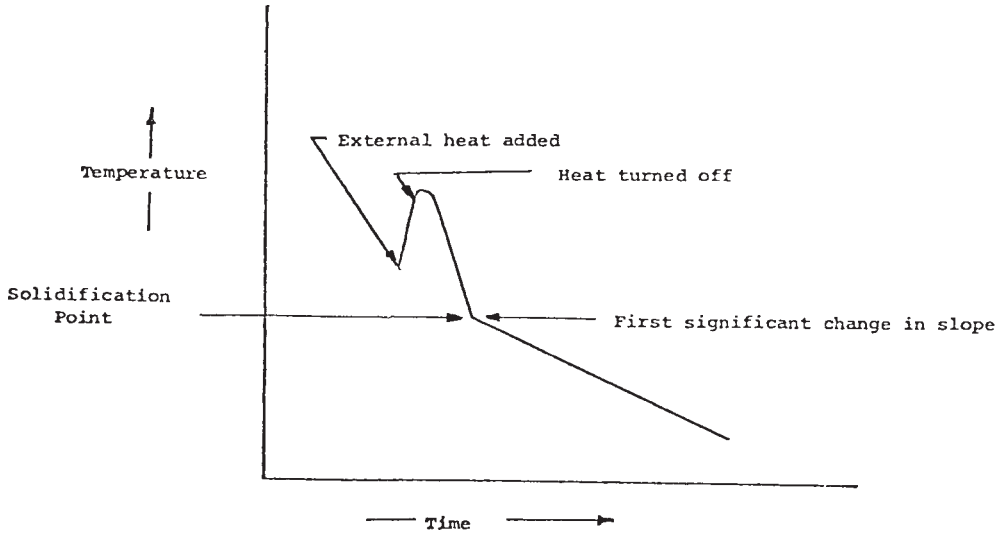

FIG. 3 A Typical Curve

ANNEX

(Mandatory Information)

A1. CALIBRATION OF RECORDER

A1.1 Set up a calibration assembly consisting of a 1000 mL-beaker, with the thermocouple probe suspended in the center and with a thermometer suspended in a closely adjacent position at the proper immersion level. The thermometer should have a range as shown below and conform to the requirements as prescribed in Specification E 1, or in Specifications for IP Standard Thermometers:

Thermometer Range	Thermometer Number
32 to 127°C	61C
90 to 260°F	61F

A1.2 Place hot water which is near boiling at about 95°C in the beaker and stir thoroughly with a variable speed stirrer. Start the recorder. As the water cools, record the exact thermometer reading and the recorder pen reading (in millivolts), taking a series of readings at intervals of even 5°C on the thermometer until the bath cools to 50°C.

A1.3 Plot a calibration curve, showing the pen reading on the abscissa (*X*-axis) and the corrected value (certified thermometer readings) on the ordinate (*Y*-axis). Use this plot for obtaining corrected solidification points.