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Standard Test Method for Softening Point of Pitch (Cube-in-Air Method)[1]

This standard is issued under the fixed designation D2319/D2319M; 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—Editorial changes were made throughout the test method in September 2014.

1. Scope*

1.1 This test method covers the determination of the softening point above ~~80°C [176°F]~~80 °C [176 °F] of pitch. Test Method D3104 gives comparable results.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D61 Test Method for Softening Point of Pitches (Cube-in-Water Method)
D3104 Test Method for Softening Point of Pitches (Mettler Softening Point Method)
D4296 Practice for Sampling Pitch
E1 Specification for ASTM Liquid-in-Glass Thermometers

3. Summary of Test Method

3.1 Two cubes of pitch, supported on wire hooks, are heated in a standardized air oven at a linear rate. The softening point is the mean of the temperatures at which the cubes sag downwards a distance of 60 mm [2.4 in.].

4. Significance and Use

4.1 Pitch does not go through a solid-liquid phase change when heated and therefore does not have a true melting point. As the temperature is raised, pitch softens and becomes less viscous. The softening point is arbitrarily defined and must be determined by a closely controlled method that must be carefully followed if test results are to be reproducible.

4.2 This test method is useful in determining the consistency of pitch as one element in establishing the uniformity of shipments and sources of supply.

5. Apparatus[3]

5.1 *Air Oven*—The oven shall be cylindrical, 150 mm [6 in.] in inside diameter by 155 mm [6.2 in.] in height. It shall have two 75 mm ~~[3-in.]~~[3 in.] diameter windows of mica, centered vertically on opposite sides, and shall be fitted with a cover having a central opening 25 mm [1 in.] in diameter to hold a thermometer, and a concentric ring suspended below to support the hooks with attached specimens. The ring shall be made of 6 mm [¼~~-in.]~~ in.] brass, shall be 54 mm [2⅛ in.] in outside diameter, and shall be

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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] The sole source of supply of the softening point apparatus items, obtainable as a unit on special order, known to the committee at this time is Humboldt Manufacturing Co., 7302 W. Agatite Ave., Chicago, IL 60656. 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. All other apparatus items described may be obtained from any regular laboratory equipment supply house.

***A Summary of Changes section appears at the end of this standard**

centrally suspended 30 mm [1.2 in.] below the cover. The cover shall have two 3 mm ~~[0.12-in.]~~[0.12 in.] holes on a diagonal 50 mm [2 in.] from the center. On the bottom of the oven, an inner pan 140 mm [5.6 in.] in diameter shall rest on three brass legs 5 mm [0.2 in.] in height fastened to the bottom of the pan to provide an air space between the pan and the bottom of the oven. The side wall, cover, and pan shall be made of 0.7 mm [0.028 in.] thick or ~~22-gauge~~22 gauge (BWG) copper sheet, and the bottom of 0.8 mm [0.032 in.] thick or ~~21-gauge~~21 gauge copper.

5.2 *Tripod for Use with Gas Burner*—The tripod ring shall be approximately 125 mm [5 in.] in inside diameter so that the oven is supported only on its outer edges. It shall be 200 mm [8 in.] in height.

5.3 *Shield for Oven:*

5.3.1 *For Use with Electric Heater*—The shield shall be cylindrical, 216 mm or [8.64 in.] in inside diameter by 181 mm or [7¼ in.] in height. It shall have two 76 mm or ~~[3-in.]~~[3 in.] diameter mica windows on opposite sides, positioned to coincide with the windows of the oven when both are placed on the electric heater. It shall be made of 0.7 mm [0.028 in.] thick or ~~22-gauge~~22 gauge sheet metal, painted inside and out with aluminum paint.

5.3.2 *For Use with Gas Burner*—The shield shall be identical with that described in 5.3.1 except that it shall be 375 mm [15 in.] in height and the windows shall coincide when the shield is standing on the bench and the oven on the tripod described in 5.2. A door ~~115~~115 mm by 115 mm [4½ in. by 4½ ~~in].~~in.] shall be provided at the bottom to give access to the burner.

5.4 *Mold*—A mold suitable for forming two 12.7 mm [½~~-in.]~~ in.] cubes of pitch, having cylindrical core pins 12 gauge ~~[2.05~~[2.05 mm to 2.07 mm] in diameter located in the base plate of the assembly to produce accurately centered suspension holes in the cubes. See Fig. 1 of Test Method D61 for details.

5.5 *Hooks*—Two L-shaped hooks made of 2.06 mm or ~~12-gauge~~12 gauge copper wire. The foot of the hook shall be 25 mm [1 in.] in length and at a right angle to the upright portion for insertion into the central hole of the pitch cube. The upright portion shall have a partial loop at the top to fit over the support ring, and shall be of the proper length, approximately 50 mm [2 in.], to position the foot on an imaginary line running horizontally through the center of the windows.

5.6 *Thermometer*—ASTM High-Softening Point Thermometer, having a range from ~~30~~30 °C to ~~200°C [80~~200 °C [80 °F to ~~400°F],~~400 °F], and conforming to the requirements for Thermometer 16C as described in Specification E1. Temperature measuring devices such as precision thermocouples, resistance temperature detectors (RTDs), and liquid-in-glass thermometers with equal or better accuracies in the appropriate temperature range may be used.

5.7 *Heat Source:*

5.7.1 *Electric Heater*—A hot plate provided with a variable transformer or other device for regulating the temperature of the heating element. It should be shielded from drafts on three sides.

5.7.2 *Gas Burner,* bunsen-type, with a chimney and a sensitive valve for regulating the size of the flame.

6. Bulk Sampling

6.1 Samples from shipments shall be taken in accordance with Practice D4296 and shall be free of foreign substances. Thoroughly mix the sample before removing a representative portion for the determination or for dehydration.

7. Dehydration

7.1 If the bulk sample contains free water, air-dry a representative portion in a forced draft oven at ~~50°C.~~50 °C.

8. Test Specimens

8.1 To melt a solid pitch sample, add the material to a container having a height equal to or exceeding its width, and a volume of not less than ~~50 mL,~~ 50 mL, until it is about half full. Place the container on a hot plate, or in an oven or bath. Do not use an open flame for melting pitch. After melting is complete, stir thoroughly but gently with the thermometer or glass rod, avoiding the incorporation of air bubbles. The maximum temperature shall not exceed the expected softening point by more than ~~50°C [90°F].~~50 °C [90 °F]. Any foam that may form must be skimmed off the surface.

8.2 Coat the inner surfaces and core pins of the mold lightly with silicone oil or silicone grease. Pour molten pitch into the cavities of the mold with the lip of the container close to the surface to minimize entrainment of air bubbles. Underpouring past the blade of a spatula is helpful in this respect. Even small bubbles markedly effect the weight of the cube and the observed softening point. Use an excess of pitch to allow for shrinkage on cooling. Cool in the mold until firm, and remove the surplus by drawing the heated blade of a putty-knife or spatula across the surface of the mold so that the cubes are pressed into the cavities. Open the mold and remove the cubes carefully. Inspect each specimen for possible cracks or bubbles and reject any which are imperfect. To reduce the risk of cracking, the mold may be heated to a temperature ~~50°C [90°F]~~50 °C [90 °F] below the expected softening point before the specimens are poured.

8.3 Place two cubes of pitch on hooks, as shown in Fig. 1 of Test Method D61, warming the hooks slightly if necessary. The foot of the hook passes through the center of two opposite faces, while the other four shall be at an angle of 45° to the horizontal. The shaft of the hook shall be ~~3~~3 mm to 5 mm ~~[0.1~~[0.1 in. to 0.2 in.] from the specimen.