



Standard Test Method for Softening Point of Pitches (Cube-in-Water Method)¹

This standard is issued under the fixed designation D 61; 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 covers the determination of the softening point of pitches below 176°F (80°C) by this method. Pitches of higher softening point should be tested by Test Method D 2319 or Test Method D 3104.

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 140 Practice for Sampling Bituminous Materials²
- D 2319 Test Method for Softening Point of Pitch (Cube-in-Air Method)³
- D 3104 Test Method for Softening Point of Pitches (Mettler Softening Point Method)⁴
- E 1 Specification for ASTM Thermometers⁵

3. Summary of Test Method

3.1 Two cubes of pitch, supported on wire hooks, are heated at a controlled rate in water in a glass container. The softening point is defined as the mean of the temperatures at which the cubes sag downwards a distance of 25 mm.

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 rises, pitch softens and becomes less viscous. The softening point is arbitrarily defined and must be established by a closely controlled method which must be carefully followed if test results are to be reproducible.

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

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² *Annual Book of ASTM Standards*, Vol 04.03.

³ *Annual Book of ASTM Standards*, Vol 05.01.

⁴ *Annual Book of ASTM Standards*, Vol 05.02.

⁵ *Annual Book of ASTM Standards*, Vol 14.03.

5. Apparatus

5.1 *Mold*—A mold suitable for forming two ½in. (12.7-mm) cubes of pitch, having cylindrical core pins 12 gage (2.05 mm) in diameter located in the base plate of the assembly to produce accurately centered suspension holes in the cubes. (See Fig. 1.)

5.2 *Hooks*—Two L-shaped hooks, made of 12-gage (2.05-mm) copper wire. The foot of the hook shall be 1 in. (25-mm) long and at a right angle to the upright portion for insertion into the center hole of the pitch cube.

5.3 *Container*—A glass vessel that can be heated, not less than 85 mm in diameter and 105 mm deep. (A standard 600-mL low-form beaker meets these requirements).

5.4 *Thermometer*—An ASTM Low Softening Point Thermometer having a range from 30 to 180°F (–2 to 80°C) and conforming to the requirements for Thermometer 15F (15C) as described in Specification E 1.

5.5 Heat Source:

5.5.1 *Electric Heater*—A hot plate or immersion heater provided with a variable transformer or other device suitable for regulating the temperature of the heating element.

5.5.2 *Gas Heater*—A bunsen-type burner, fitted with a chimney.

6. Sampling

6.1 Samples from shipments shall be taken in accordance with Practice D 140 and shall be free of foreign substances. Thoroughly mix the sample before removing a representative portion for the determination or for dehydration.

6.2 If the presence of water is indicated by surface foam on heating, maintain the sample at a temperature of about 260°F (125°C) in an open container until the surface is free of foam. Take care not to overheat, and remove from the heat source as soon as the foam has subsided.

7. Test Specimens

7.1 Coat the inner surfaces and core pins of the mold very lightly with silicone oil or silicone grease. Form the pitch into truly shaped cubes either by pressing (7.2) or by pouring (7.4), the latter being preceded by melting (7.3) if the sample is solid. Use an excess of pitch in either case. Cool until firm and remove the surplus material 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. Then open the mold and

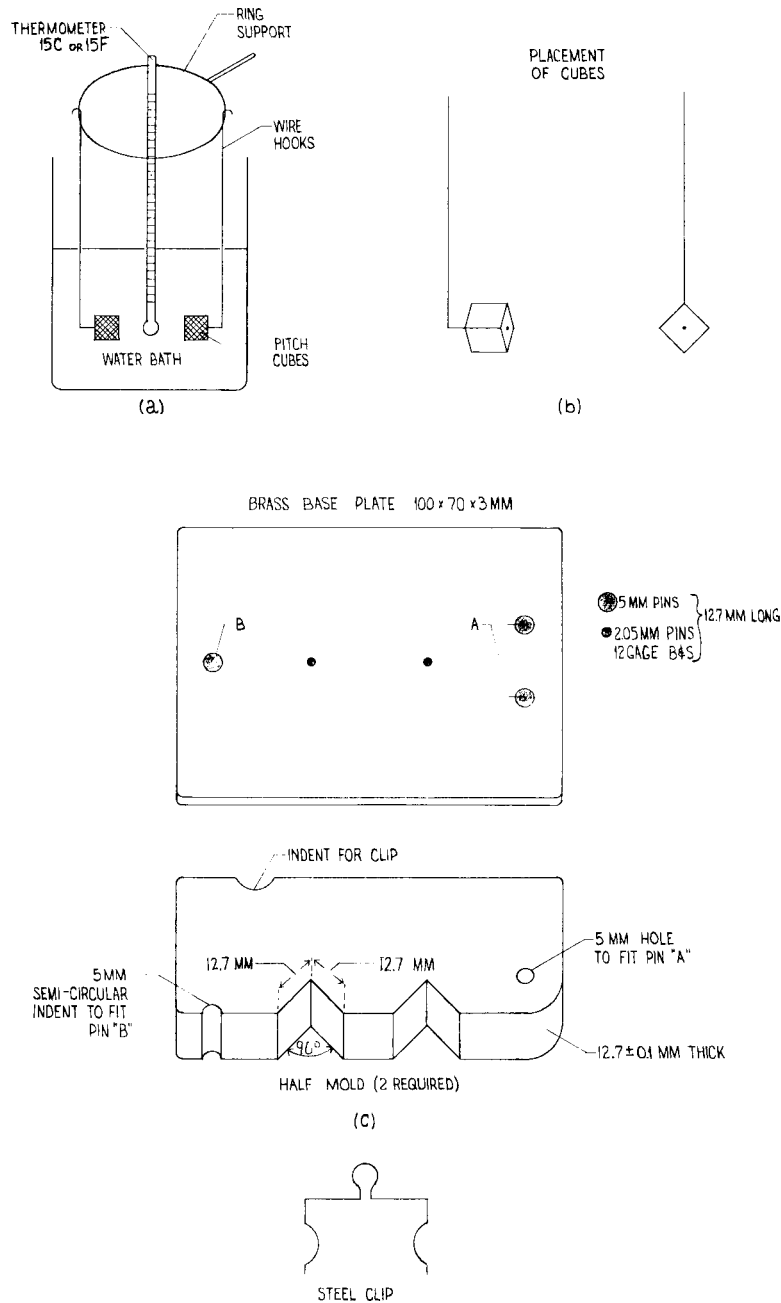


FIG. 1 Apparatus for Cube-in-Water Method

remove the cubes carefully to avoid distortion. Inspect each specimen for possible cracks or bubbles and reject any which are not perfect.

7.2 Pitches with softening points up to about 140°F (60°C) can usually be pressed into the mold by hand at, or slightly above, room temperature. If too soft, they may be pressed under water at about 40°F (5°C).

7.3 To melt a dry 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, 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 gently but thoroughly, avoiding the incorporation of air bubbles. The maximum temperature should not exceed the

expected softening point by more than 90°F (50°C). Any foam that forms must be skimmed off.

7.4 Pour a slight excess of the liquid pitch into the mold with the lip of the container close to the surface so as to minimize the entrainment of air bubbles. Underpouring past the blade of a spatula is helpful in this respect. Even small bubbles markedly affect the weight of the cube and the observed softening point. Cool the specimens in the mold until firm, under cold water if necessary; then trim and inspect the cubes as directed in 7.1.

8. Procedure

8.1 *Pitches Having Softening Points Between 110 and 176°F (43 and 80°C):*

8.1.1 Assemble the apparatus as shown in Fig. 1. Fill the container to a depth of about 100 mm with freshly boiled distilled water cooled to 60°F (15°C) (Note 1 and Note 2). Place two cubes of pitch on hooks as shown in Fig. 1, warming the hooks slightly before inserting them in the center holes if necessary. Suspend the specimens in the bath so that the lower edges are 1 in. (25 mm) above the bottom of the container. Position the thermometer so that the bulb is between the cubes and within 6 mm, but not touching either cube. The bottom of the bulb should be level with the bottom of the cubes. Allow the specimens to remain in the bath at 60°F (15°C) for 15 min before applying heat.

NOTE 1—The use of freshly boiled water is essential to prevent the formation of air bubbles on its surface which may retard the sinking of the cube.

NOTE 2—A sheet of filter paper that will sink when wet may be placed on the bottom of the container to prevent the pitch from sticking to the glass.

8.1.2 Apply heat in such a manner that the temperature of the water is raised 9°F (5°C)/min. The rate shall be uniform and shall not be averaged over the period of the test. The maximum permissible variation for any 1-min period, after the first three, shall be $\pm 1^\circ\text{F}$ (0.5°C). Reject all tests in which the rate of rise is outside these limits.

8.1.3 The softening point is the average of the temperatures at which the two cubes touch the bottom of the container. Estimate individual readings to the nearest 0.5°F (0.2°C).

Repeat the determination if the individual values differ by more than 2°F (1°C).

8.2 *Pitches Having Softening Points Below 110°F (43°C)*—Follow the procedures described in 8.1, but cool the water bath to 40°F (5°C). After immersing the specimens in the bath, maintain the temperature for at least 15 min before applying heat.

9. Report

9.1 Report the average value found in 8.1.3, rounded to the nearest 1°F (0.5°C) as the softening point (cube-in-water).

10. Precision

10.1 The following criteria shall be used for judging the acceptability of the results (95 % confidence level):

10.1.1 *Repeatability*—Duplicate values by the same operator shall not be considered suspect unless they differ by more than 3°F (1.5°C).

10.1.2 *Reproducibility*—The values reported by each of two laboratories shall not be considered suspect unless they differ by more than 5°F (3°C).

10.1.3 *Bias*—The procedures in Test Method D 61 for softening point has no bias because the value of softening point is defined only in the terms of this test method.

11. Keywords

11.1 cube-in-water; pitch; softening point

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