Designation: D61 – 15

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

This standard is issued under the fixed designation D61; 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). Pitches of higher softening point should be tested by Test Method D2319 or Test Method D3104.

1.2 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered 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]

D140 Practice for Sampling Bituminous Materials
D2319 Test Method for Softening Point of Pitch (Cube-in-Air Method)
D3104 Test Method for Softening Point of Pitches (Mettler Softening Point Method)
E1 Specification for ASTM Liquid-in-Glass Thermometers
E563 Practice for Preparation and Use of an Ice-Point Bath as a Reference Temperature
E1137 Specification for Industrial Platinum Resistance Thermometers
E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids
E2877 Guide for Digital Contact Thermometers

3. Terminology

3.1 *Definitions:*

3.1.1 *Digital Contact Thermometer (DCT), n*—an electronic device consisting of a digital display and associated temperature sensing probe.

3.1.1.1 *Discussion*—This device consists of a temperature sensor connected to a measuring instrument; this instrument measures the temperature-dependent quantity of the sensor, computes the temperature from the measured quantity, and provides a digital output or display, or both, of the temperature. The temperature sensing probe is in contact with the material whose temperature is being measured. This device is sometimes referred to as a *digital thermometer*.

NOTE 1—Portable electronic thermometers (PET) is an acronym sometimes used to refer to a subset of the devices covered by this definition.

4. Summary of Test Method

4.1 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.

5. Significance and Use

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

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

6. Apparatus

6.1 *Mold*—A mold suitable for forming two ½ in. (12.7 mm) cubes of pitch, having cylindrical core pins 12 gauge (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.)

6.2 *Hooks*—Two L-shaped hooks, made of 12 gauge (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.

6.3 *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).

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricantsand is the direct responsibility of Subcommittee D02.05 on Properties of Fuels, Petroleum Coke and Carbon Material.

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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.

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

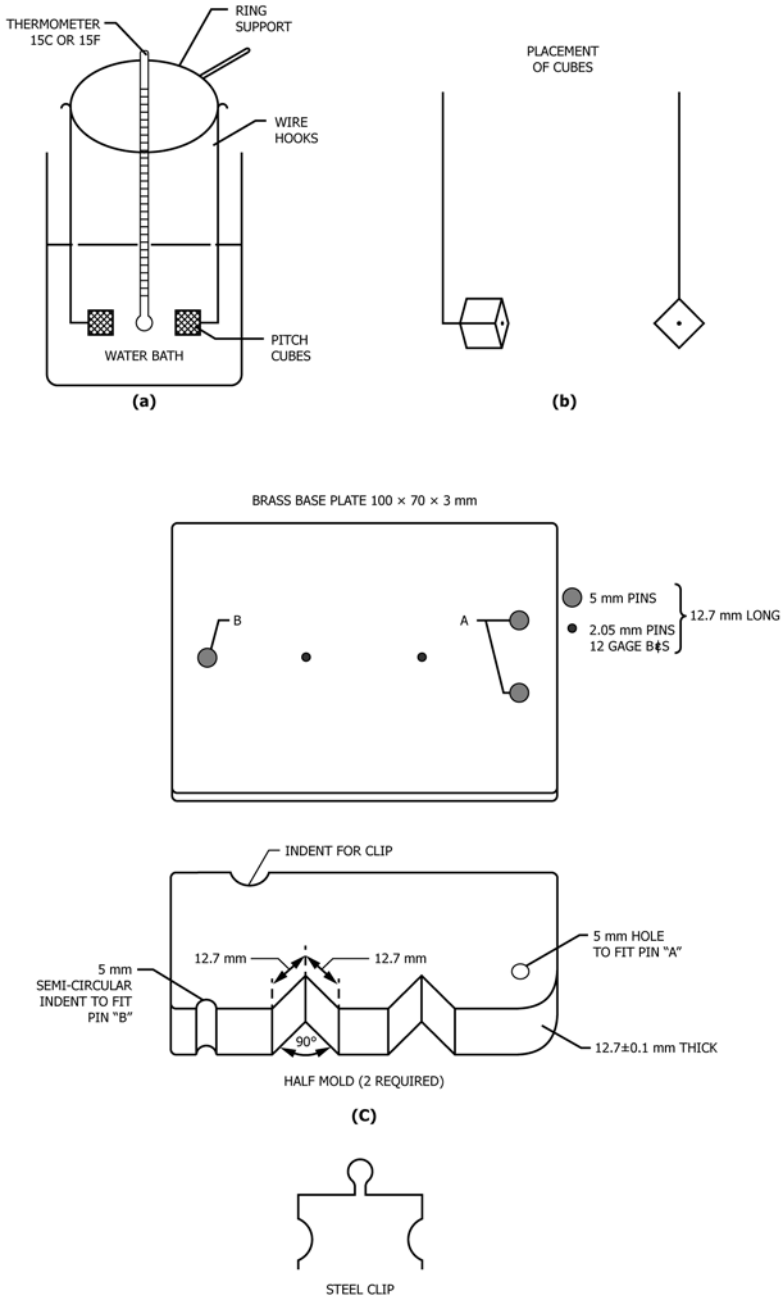

FIG. 1 Apparatus for Cube-in-Water Method

6.4 *Temperature Measuring Device*—Use either a calibrated DCT meeting the requirements described in 6.4.1 or liquid-in-glass thermometers described in 6.4.2. A DCT or calibrated liquid-in-glass thermometer shall be used as the thermometer for temperature measurement independent of the instrument's temperature control, and shall be located in the thermowell.

NOTE 2—The display device and sensor must be correctly paired. Incorrect pairing will result in temperature measurement errors and possibly irreversible damage to the electronics of the display.

6.4.1 *Digital Contact Thermometer Requirements:*

Criteria	Requirements
DCT	E2877 Class E, Minimum
Temperature range	–2 °C to 80 °C
Display resolution	0.1 °C
Sensor type	PRT, thermistor, Type E or T thermocouple
Sensor, sheath	Metal or glass
Sensor length	Less than 12 mm in length
Immersion depth	76 mm
Display accuracy	±0.2 °C for combined probe and sensor
Response time	Less than or equal to 30 s as defined in Specification E1137
Drift	Less than 0.2 °C per year
Calibration error	Less than 0.2 °C over the range of intended use
Calibration range	–2 °C to 80 °C
Calibration data	4 data points evenly distributed over the range and included in calibration report
Calibration report	From a calibration laboratory with demonstrated competency in temperature calibration which is traceable to a national calibration laboratory or metrology standards body

6.4.1.1 The DCT calibration drift shall be checked at least annually by either measuring the ice point or against a reference thermometer in a constant temperature bath at the prescribed immersion depth to ensure compliance with 6.4.1. With respect to an ice bath, Practice E563 provides guidance on the preparation and use of an ice bath. However, for this use, variance from the specific steps, such as water source, is permitted provided preparation is consistent. The basis for the variance is due to the ice bath reference being used for tracking change in calibration not verification.

NOTE 3—When a DCT's calibration drifts in one direction over several calibration checks, that is, ice point, it may be an indication of deterioration of the DCT.

6.4.2 An ASTM Low Softening Point Thermometer having a range from – 2 °C to 80 °C and conforming to the requirements for Thermometer 15 as described in Specification E1 or E2251.

6.4.2.1 *Calibration Check*—Verify thermometer at least annually against a reference thermometer in a constant temperature bath or an ice bath. The thermometer is to be inserted to its immersion depth. If using an ice bath, the ice point reading is to be taken within 60 min after the thermometer has been at test temperature for at least 3 min. If the corrected temperature reading deviates from the reference thermometer or the ice point, then repeat this calibration check. If the thermometer deviates from the reference value on two successive checks, then a full thermometer recalibration is needed.

6.4.2.2 *Recalibration*—A complete recalibration of the liquid-in-glass thermometer, while permitted, is not necessary in order to meet the accuracy ascribed to liquid-in-glass thermometer's design until the thermometers corrected measured temperature deviates from the reference thermometer or ice point by one scale division, or until five years has elapsed since the last full calibration.

6.5 *Heat Source:*

6.5.1 *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.

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

7. Sampling

7.1 Samples from shipments shall be taken in accordance with Practice D140 and shall be free of foreign substances. Thoroughly mix the sample before removing a representative portion for the determination or for dehydration.

7.2 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.

8. Test Specimens

8.1 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 (8.2) or by pouring (8.4), the latter being preceded by melting (8.3) 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 remove the cubes carefully to avoid distortion. Inspect each specimen for possible cracks or bubbles and reject any which are not perfect.

8.2 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).

8.3 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 shall be skimmed off.

8.4 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 8.1.

9. Procedure

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

9.1.1 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) (Notes 4 and 5). 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 4—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 5—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.

9.1.2 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 ±1 °F (0.5 °C). Reject all tests in which the rate of rise is outside these limits.

9.1.3 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).

9.2 *Pitches Having Softening Points Below 110 °F (43 °C)*—Follow the procedures described in 9.1, 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.

10. Report

10.1 Report the average value found in 9.1.3, rounded to the nearest 1 °F (0.5 °C) as the softening point (cube-in-water).

11. Precision and Bias

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

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

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

11.1.3 *Bias*—The procedure in Test Method D61 for softening point has no bias because the value of softening point is defined only in the terms of this test method.

12. Keywords

12.1 cube-in-water; pitch; softening point

SUMMARY OF CHANGES

Subcommittee D02.05 has identified the location of selected changes to this standard since the last issue (D61 – 75 (2010)) that may impact the use of this standard. (Approved July 1, 2015.)

(1) Revised Section 2.
(2) Added new Terminology Section 3.
(3) Revised 6.4.