Designation: D3104 – 14a (Reapproved 2018)

Standard Test Method for Softening Point of Pitches (Mettler Softening Point Method)[1]

This standard is issued under the fixed designation D3104; 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 having softening points in the range from 50 °C to 180 °C by this test method, and gives results comparable to those obtained by Test Method D2319 above 80 °C (176 °F).

1.2 The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

A314 Specification for Stainless Steel Billets and Bars for Forging

D2319 Test Method for Softening Point of Pitch (Cube-in-Air Method)

D4296 Practice for Sampling Pitch

3. Summary of Test Method

3.1 In this test method, the softening point is defined as the temperature at which pitch, suspended in a cylindrical cup with a 6.35 mm hole in the bottom, flows downward a distance of 19 mm to interrupt a light beam, as the sample is heated at a linear rate in air.

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 gradually softens or becomes less viscous. For this reason, the determination of the softening point must be made by an arbitrary, but closely defined, method if the test values are to be reproducible.

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

5. Apparatus

5.1 A METTLER TOLEDO dropping point cell[3] shall be used to determine pitch softening points by this test method. These commercially available instruments consist of a control unit with a digital temperature indicator, with furnace built in or attached, sample cartridges, and accessories. The control unit automatically regulates the heating rate of the furnace. The softening point is indicated on the readout, and the heating program stopped, when the sample flow triggers the softening point detection. A general view of the contents of a METTLER TOLEDO dropping point instrument is shown in Fig. 1 (old instrument) and Fig. 2 (new instrument).

5.1.1 *Integrated or Separate Control Unit*—The control unit shall provide a continuous, linear temperature increase from 25 °C to 250 °C at a rate of 2 °C/min. A digital readout shall indicate the temperature to 0.1 °C throughout.

5.1.2 *Integrated or Separate Furnace Unit*—The furnace unit shall be capable of heating one or two sample cup assemblies, as described in 5.1.3, at a linear rate of 2 °C ± 0.3 °C/min. It shall include a sensing system capable of detecting the softening point with a precision of 0.1 °C.

[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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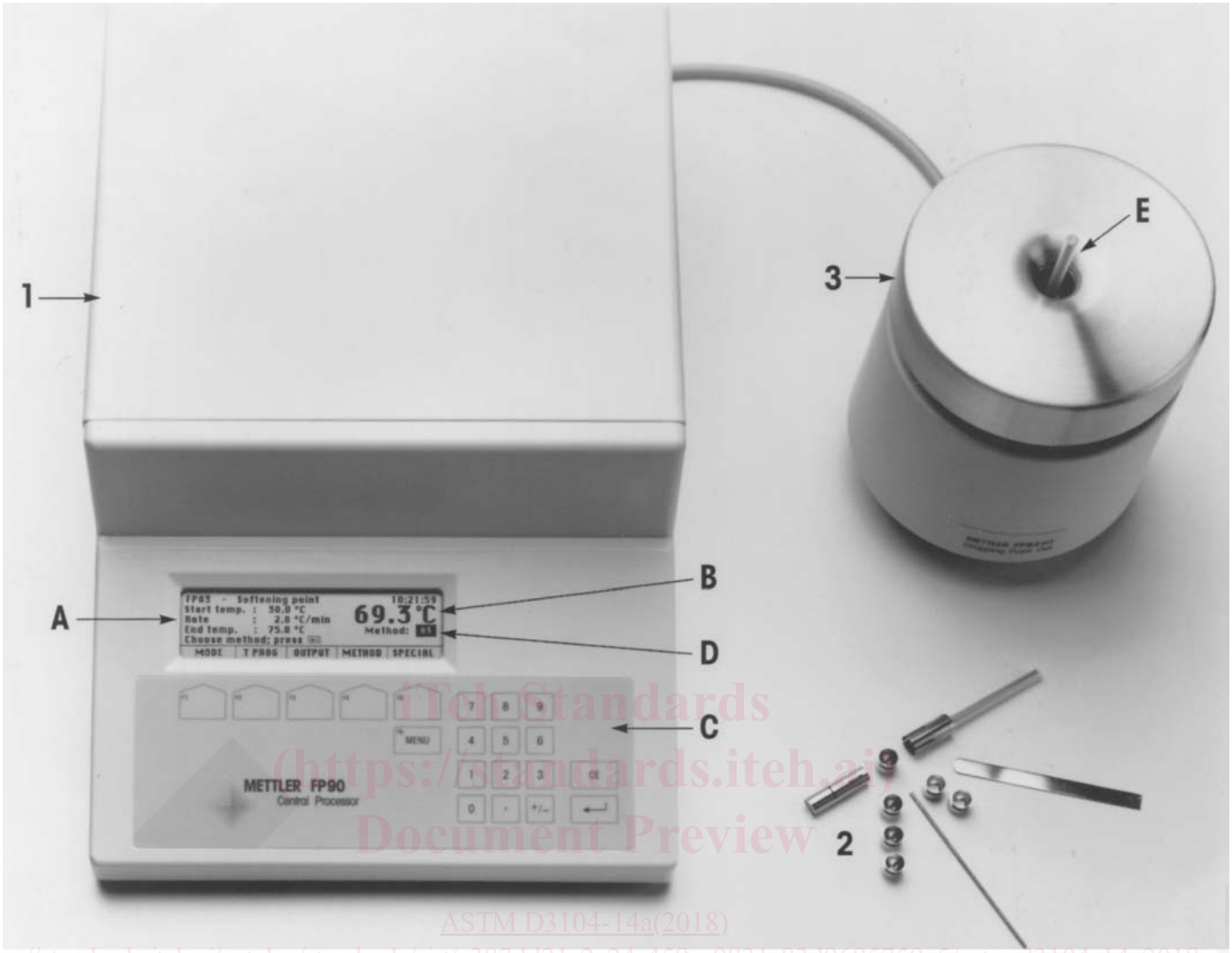
[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The sole source of supply of the apparatus known to the committee at this time is available from Mettler-Toledo, LLC., 1900 Polaris Pkwy, Columbus, OH 43240, www.mt.com. 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.



General View of the METTLER TOLEDO FP90/FP83HT

1 Control and evaluation unit
- A LCD with guide for operator
- B Temperature display
- C Keyboard with function keys F1 to F6
- D Selected method number

2 Cartridges with accessories

3 Measuring cell FP83HT
- E Sample holder

FIG. 1 General View of the METTLER TOLEDO FP90 Control Unit with Heater FP83HT

5.1.3 *Sample Cup Assembly*—A cup of chromium-plated brass, or of aluminum, or of stainless steel conforming to the requirements for Type 303 (UNS S30300) stainless steel as prescribed in Specification A314, with the dimensions shown in Fig. 3. It shall be placed in the assembly so that the pitch sample softening point will be detected when it has flowed down a distance of 19 mm.

6. Reagents

6.1 *Xylene,* industrial grade.

6.2 *Benzoic Acid.*

7. Calibration of the METTLER TOLEDO Apparatus

7.1 This step, required only occasionally, is designed to establish that the temperature indicated by the instrument is in agreement with a known standard. A special cup with a bottom orifice of 2.8 mm is used instead of the one prescribed for the testing of pitch.

7.2 *Reagent*—Use either analytical reagent or primary standard grade benzoic acid for the calibration. As this material is hygroscopic it must be stored in a tightly sealed container, and replaced with fresh material from a newly opened supply if hydration or other contamination is suspected.

FIG. 2 General View of the METTLER TOLEDO DP70

7.3 *Procedure—Old Instruments:*

7.3.1 *Filling the Sample Cup*—Place the cup on a clean, flat surface. Add a small amount of benzoic acid crystals and press down with a rod (approximately 4.5 mm in diameter). Check that the bottom orifice is completely filled. Refill and repeat the pressing step until the cup is filled with benzoic acid. Remove any crystals from the exterior of the cup.

7.3.2 *Heating*—Preheat the Mettler furnace to 121 °C, and maintain it at that temperature. Place the cartridge assembly containing the benzoic acid in position in the furnace, taking care that the slits for the light beam are properly positioned. Wait for temperature equilibration, that is, the furnace and the sample are in equilibrium at the preset temperature, but not less than the 30 s after inserting the cartridge, start the automatic heating cycle at 0.2 °C/min. The temperature will rise steadily at the correct rate until the drop point is reached, and then remain steady on the readout.

7.3.3 *Cleaning*—Immediately remove the cartridge assembly. Check to determine that the sample has passed through the light beam and no pre-triggering has occurred. If a malfunction is suspected, the entire procedure must be repeated. Inspect the apparatus carefully to ensure that no residue remains. Use a spatula shaped to the contour of the cup to remove most of the remaining acid from the cup and from the bottom of the cartridge. Wash the cup and cartridge in xylene, or other suitable solvent, to remove the last traces of the residue.

7.4 *Interpretation*—See 7.6.