



Designation: D3461 – 97(Reapproved 2007)

# Standard Test Method for Softening Point of Asphalt and Pitch (Mettler Cup-and-Ball Method)<sup>1</sup>

This standard is issued under the fixed designation D3461; 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 ( $\epsilon$ ) 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 asphalt and pitch in the range from 50 to 180°C by the cup-and-ball apparatus, and gives results comparable to those obtained by Test Method D36.

NOTE 1—If the softening point of asphalt by this Mettler cup-and-ball method fails to meet specified requirements, tests may be rerun using the Test Method D36 (ring-and-ball) softening point apparatus as a referee method.

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:*<sup>2</sup>

A314 Specification for Stainless Steel Billets and Bars for Forging (Withdrawn 2009)<sup>3</sup>

D36 Test Method for Softening Point of Bitumen (Ring-and-Ball Apparatus) (Withdrawn 2009)<sup>3</sup>

D140 Practice for Sampling Bituminous Materials (Withdrawn 2009)<sup>3</sup>

D4296 Practice for Sampling Pitch (Withdrawn 2009)<sup>3</sup>

## 3. Summary of Test Method

3.1 In this test method the softening point is defined as the temperature at which the specimen, suspended in a cylindrical cup with a 6.5-mm hole in the bottom and with a lead ball, 8 mm in diameter, centered on top of the sample in the cup,

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.05 on Properties of Fuels, Petroleum Coke and Carbon Material.

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<sup>2</sup> 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.

<sup>3</sup> The last approved version of this historical standard is referenced on www.astm.org.

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 Asphalt and pitch do not go through a solid-liquid phase change when heated, and therefore do not have true melting points. As the temperature is raised, they gradually soften or become 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 as one element in establishing the uniformity of shipments or sources of supply.

## 5. Apparatus

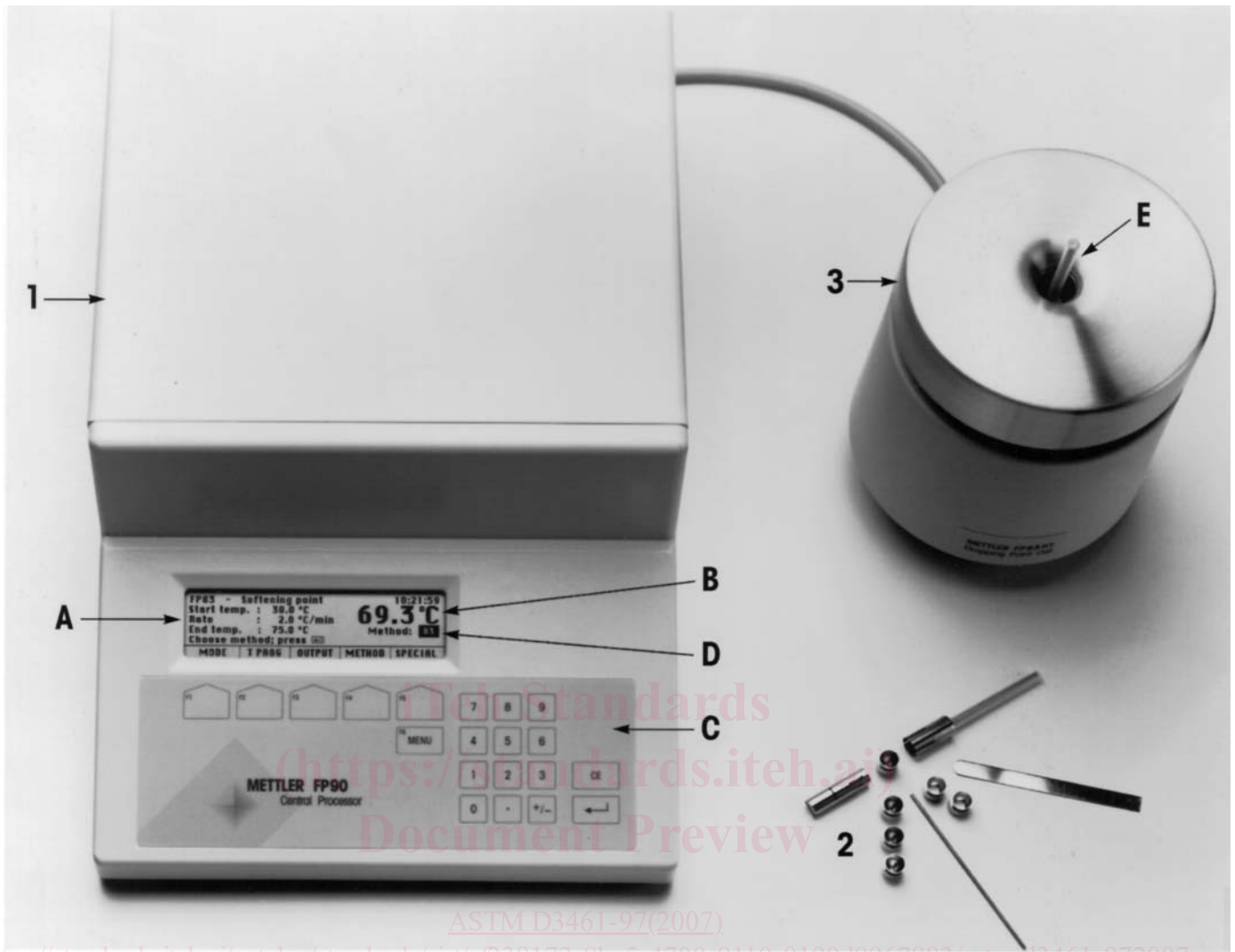
5.1 A Mettler dropping point cell<sup>4</sup> can be used to determine softening points by this test method. These commercially available instruments consist of a control unit with a digital temperature indicator, matched furnace, sample cartridges, and accessories. The control unit automatically regulates the heating rate of the furnace. The softening point is indicated on the readout, and the heating program stopped, when the sample flow triggers a photocell detector. A general view of the components of the Mettler apparatus is shown in Fig. 1.

5.1.1 *Control Unit*—This unit shall provide a continuous, linear temperature increase from 25 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 *Furnace Unit*—This unit shall be capable of heating a specimen cup assembly, as described in 5.1.3, at a linear rate of  $2 \pm 0.3$ °C/min. It shall include a sensing system capable of detecting the softening point with a precision of 0.1°C.

5.1.3 *Specimen Cup Assembly*—A cup of chromium-plated brass, or stainless steel conforming to the requirements for Type 303 (UNS 530300) as prescribed in Specification A314, with dimensions shown in Fig. 2. It shall be placed in the assembly so that the test specimen will flow down a distance of 19 mm before interrupting a light beam and stopping the heating program as the softening point is reached.

<sup>4</sup> Available from the Mettler Toledo, Inc., Balances and Instruments, 69 Princeton-Hightstown Rd., Hightstown, NJ 08520-0071.



General View of the METTLER TOLEDO FP90/FP83HT

- |                                        |                               |
|----------------------------------------|-------------------------------|
| 1 Control and evaluation unit          | 2 Cartridges with accessories |
| A LCD with guide for operator          | 3 Measuring cell FP83HT       |
| B Temperature display                  | E Sample holder               |
| C Keyboard with function keys F1 to F6 |                               |
| D Selected method number               |                               |

FIG. 1 General View of the Mettler FP-5/53

5.1.4 *Lead Ball*—A lead ball weighing  $3.20 \pm 0.10$  g. A32-caliber lead shot is suitable for this purpose.

## 6. Reagents

6.1 *Xylene*, industrial grade.

## 7. Calibration of Mettler 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 specimen testing.

7.2 *Reagent*—Use either analytical reagent or primary standard grade benzoic acid for this 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.

### 7.3 Procedure:

7.3.1 *Filling the Specimen Cup*—Place the cup on a clean, flat surface. Add a small amount of benzoic acid crystals and press down with a rod (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.

