



## Standard Test Method for Residue of Specified Penetration<sup>1</sup>

This standard is issued under the fixed designation D 243; 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 is used to thermally reduce cut-back asphalt, a road oil or a semisolid asphalt, having a penetration greater than 100, to a residue of specified penetration.

1.2 The values stated in SI units are to be regarded as the standard. The inch-pound units given in parentheses are for information only.

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.* Specific precautionary statements are given in Note 2.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

D 5 Test Method for Penetration of Bituminous Materials<sup>2</sup>

E 1 Specification for ASTM Thermometers<sup>3</sup>

E 220 Test Method for Calibration of Thermocouples By Comparison Techniques<sup>4</sup>

### 3. Summary of Test Method

3.1 The material to be tested is rapidly heated to 249°C (480°F) and maintained at 249° to 260°C (480° to 500°F) during evaporation of the volatiles. Penetration of the residue is determined and if not within the specified limits, the evaporation procedure is repeated. Change in sample mass is used to calculate the percentage of residue having the specified penetration.

### 4. Significance and Use

4.1 This test method is used to determine the percentage of residue having a specified penetration at 100 g/5 s at 25°C (77°F). This test method provides a residue for quality control or for use in other tests as desired.

### 5. Apparatus

5.1 The apparatus shall consist of a container, heating bath, hot plate, and thermometer, with necessary accessory apparatus as follows:

5.1.1 *Container*—The container in which the sample is to be tested shall be a flat-bottom, cylindrical seamless tin box, 70 mm in diameter and 45 mm in depth.

NOTE 1—Containers known in the pharmaceutical industry as seamless “ointment boxes” may be obtained in dimensions conforming to the above requirements.

5.1.2 *Heating Bath*—The heating bath shall be a cast-iron air bath, or equivalent, permitting the immersion of the container to a depth of  $32 \pm 5$  mm through an opening  $3 \pm 2$  mm larger in diameter than the container. It shall support the container  $6 \pm 2$  mm above the hot plate, and with at least 6.4 mm free air space between the sides of the container and of the air bath below the opening. A suitable air bath is shown in Fig. 1.<sup>5</sup>

5.1.3 *Hot Plate*—The air bath shall be heated upon a suitably mounted hot plate, heated either electrically or by means of a gas flame. The plate shall be capable of maintaining the sample continuously at the required temperature, and apparatus necessary to fulfill this requirement, such as a rheostat or gas pressure regulator, shall be provided.

5.1.4 *Thermometer*—An ASTM Cleveland Open Flash Thermometer, having a range of  $-6$  to  $+400^\circ\text{C}$  or  $+20$  to  $+760^\circ\text{F}$  and conforming to the requirements for thermometer 11C or 11F, respectively, as prescribed in Specification E 1, or an equivalent thermometric device that has been calibrated in accordance with Test Method E 220.

5.1.5 A balance, readable to 0.01 g, for determining the mass of the asphalt sample and the mass of the residue.

### 6. Preparation of Sample

6.1 Thoroughly stir and agitate the sample as received to ensure a complete mixture before the portion for testing is removed.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D04 on Road and Paving Materials and is the direct responsibility of Subcommittee D04.46 on Durability and Distillation Tests.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 04.03.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 14.03.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 14.03.

<sup>5</sup> An air bath meeting the requirements of this test method is available from Humboldt Mfg. Co., 7300 Agatite Ave., Norridge, IL, 60706–4704, as part number H-2480.

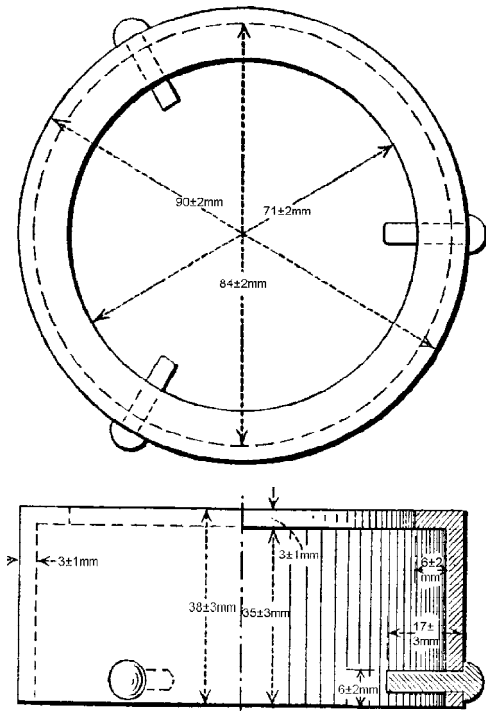


FIG. 1 Cast-Iron Air Bath

## 7. Procedure

7.1 Weigh a  $100.00 \pm 0.1$ -g sample of the material to be tested into a tared container; then place the container in the air bath in position to be heated. Support the thermometer in the sample equidistant from the sides of the container and with the bottom of the bulb neither more than 6 mm above nor touching the bottom of the container. The bulb shall be completely immersed in the sample throughout the heating. An assembly of the apparatus is shown in Fig. 2.

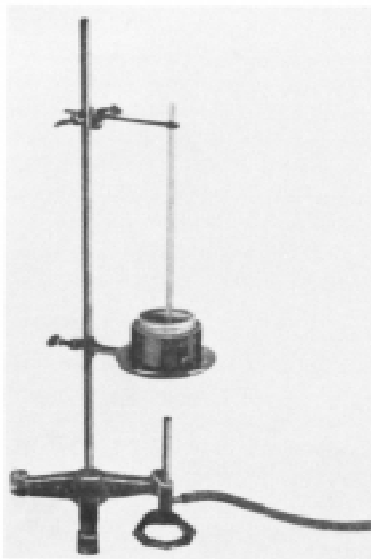


FIG. 2 Assembly of Apparatus

7.2 Heat the sample as rapidly as possible without foaming to a temperature of  $249^{\circ}\text{C}$  ( $480^{\circ}\text{F}$ ) and during the evaporation, maintain the temperature between  $249^{\circ}\text{C}$  ( $480^{\circ}\text{F}$ ) and  $260^{\circ}\text{C}$  ( $500^{\circ}\text{F}$ ). Stir the sample with the thermometer from time to time to prevent local overheating and, to maintain a homogeneous sample, flux into the sample all cakes of hardened bitumen that form at the sides of the container.

NOTE 2—**Precaution:** Certain types of road oil will readily form rings of hard asphalt at the side of the container. Take great care that this material is completely fluxed into the sample before the penetration of the residue is determined.

7.3 An experienced operator can judge approximately what percentage of residue should be obtained to secure the desired penetration. When it is supposed that the residue will show the required penetration, return to the container the bitumen on the thermometer that may be readily scraped off; then remove the container from the air bath and cool and weigh it. Determine the penetration of the residue in accordance with Test Method D 5, except use the container in which evaporation was conducted, as specified in this test method, instead of using the smaller container specified in Test Method D 5.

7.4 It frequently is necessary to make several trials before a residue of the required penetration is obtained. If it is determined to be greater than that required, remove all water from the container and the surface of the sample, and repeat the heating and determination of penetration as before. Ordinarily, a residue shall be considered as satisfactorily obtained when its penetration is within 15 dmm of that desired, and its percentage by weight of the original sample shall be calculated. When it is necessary to determine more precisely the percentage of residue having the specified penetration, such a percentage shall be calculated by interpolation between percentages of two residues, one having a penetration greater and one having a penetration lower than that specified.

## 8. Report

8.1 Report the results as follows:

8.1.1 Percentage of residue of—penetration (determined—) stating, first, the specified penetration, and second, the penetration actually determined for the sample tested or calculated by interpolation.

## 9. Precision and Bias

9.1 With care and proper attention to details, duplicate determinations by this test method should not differ from each other by more than 1.0 % with the same operator nor more than 2.5 % between different laboratories.

9.2 The precision of tests on the residue after heating, other than penetration, has not been determined. The nature of this test method makes it impractical to develop a precision statement.

9.3 *Bias*—The procedure in this test method has no bias because the property being measured is defined only in terms of this test method.

## 10. Keywords

10.1 asphalt; cut-back; penetration; residue; road oil

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