Designation: D243/D243M – 14

Standard Test Method for Residue of Specified Penetration[1]

This standard is issued under the fixed designation D243/D243M; 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 is used to thermally reduce cutback asphalt, a road oil or a semisolid asphalt, having a penetration greater than 100, to a residue of specified penetration. It is primarily used with slow-curing cutback asphalt as specified in Specification D2026.

1.2 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.

1.3 **Warning—**Mercury has been designated by the United States Environmental Protection Agency (EPA) and many state agencies as a hazardous material that can cause central nervous system, kidney and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury containing products. See the applicable product Material Safety Data Sheet (MSDS) for details and EPA's website—http://www.epa.gov/mercury/—for additional information. Users should be aware that selling mercury, mercury-containing products, or both, into your state may be prohibited by state law.

1.4 *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]

D5 Test Method for Penetration of Bituminous Materials

D2026 Specification for Cutback Asphalt (Slow-Curing Type)

E1 Specification for ASTM Liquid-in-Glass Thermometers

E77 Test Method for Inspection and Verification of Thermometers

E220 Test Method for Calibration of Thermocouples By Comparison Techniques

E644 Test Methods for Testing Industrial Resistance Thermometers

E1137/E1137M Specification for Industrial Platinum Resistance Thermometers

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 thermometric device, 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, approximately 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 ± 5 mm through an opening 3 ± 2 mm larger in diameter than the container. It shall support the container 6 ± 2 mm above the hot plate, and with at least 6.4

[1] 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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[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.

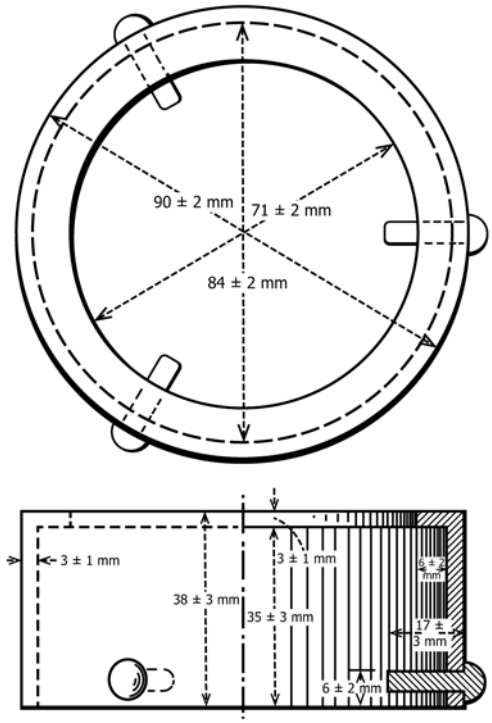

FIG. 1 Cast-Iron Air Bath

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.[3]

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*—A thermometer for measuring the temperature of the sample. The thermometer shall be one of the following:

5.1.4.1 An ASTM 11C or 11F liquid-in-glass thermometer which conforms to the requirements of Specification E1. The thermometer shall be standardized in accordance with one of the methods in Test Method E77.

5.1.4.2 A metal-sheathed thermocouple paired with a temperature readout. The overall sheath length shall be at least 50 mm [2 in.] greater than the immersion depth. The thermocouple system (probe and readout) shall be standardized in accordance with Test Method E220.

5.1.4.3 A platinum resistance thermometer (PRT) with a probe which conforms to the requirements of Specification E1137/E1137M. The PRT shall have a 3- or 4-wire configuration and the overall sheath length shall be at least 50 mm [2 in.] greater than the immersion depth. The PRT system (probe and readout) shall be standardized in accordance with Test Methods E644.

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 uniform mixture before the portion for testing is removed.

7. Procedure

7.1 Weigh a 100.00 ± 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 probe of the thermometric device in the sample equidistant from the sides of the container and with the bottom of the probe neither more than 6 mm [¼ in.] above nor touching the bottom of the container. The tip of the probe shall be completely immersed in the sample throughout the heating. An assembly of the apparatus is shown in Fig. 2.

7.2 Heat the sample as rapidly as possible without foaming to a temperature of 249°C [480°F] and during the evaporation, maintain the temperature between 249 and 260°C [480 and 500°F]. Stir the sample with the probe of the thermometric device from time to time to prevent local overheating and, to maintain a homogeneous sample. Stir into the sample all cakes of hardened asphalt 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 stirred 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 asphalt on the probe of the thermometric device that may be readily scraped

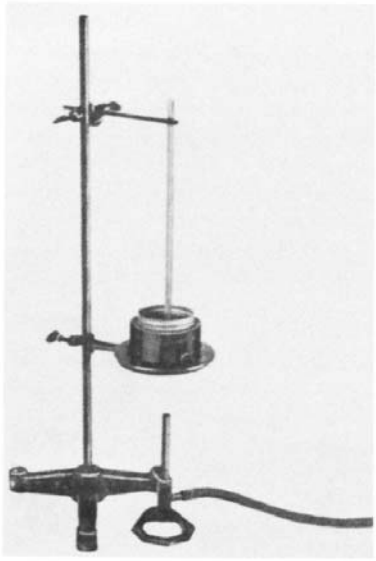

FIG. 2 Assembly of Apparatus

[3] A source of supply of the apparatus that meets the requirements of this test method is available from Humboldt Mfg. Co., 7300 Agatite Ave., Norridge, IL, 60706–4704, as part number H-2480. 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.

off; then remove the container from the air bath and cool and determine the mass of the residue. Determine the penetration of the residue in accordance with Test Method D5, 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 D5.

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 units of that desired, and its mass percentage 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; cutback; penetration; residue; road oil