Designation: D 1189 – 61 (Reapproved 1973)

Standard Test Method for VACUUM DISTILLATION OF LIQUID AND SEMI-SOLID ASPHALTIC MATERIALS TO OBTAIN A RESIDUE OF SPECIFIED PENETRATION[1]

This Standard is issued under the fixed designation D 1189; 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.

1. Scope

1.1 This method of test covers the determination, by vacuum distillation and with the elimination of oxidation effects, of the quantity of asphaltic residue of specified penetration. When the penetration of the residue is not otherwise stated, it shall be understood to be 100. The residue obtained is available for tests as desired.

NOTE 1—The test results on the residue may be affected by nonasphaltic materials present in the asphalt.

NOTE 2—This method is not intended to replace ASTM Method D 243, Test for Residue of Specified Penetration[2], which introduces oxidation effects.

2. Apparatus

2.1 *Flask*—A standard 500-ml Claisen distilling flask, constructed of heat-resistant glass,[3] as shown in Fig. 1 and conforming to the following dimensions:

Diameter of bulb, outside	101 ± 2.0 mm
Diameter of necks outside	25 ± 1.2 mm
Diameter of tubulature, outside	9 ± 0.5 mm
Height of flask, outside	268 ± 3.0 mm
Vertical distance from bottom of bulb, outside, to horizontal tangent at junction of necks, outside	126 ± 3 mm
Vertical distance from bottom of bulb, outside, to horizontal tangent at junction of neck and tubulature, inside	190 ± 3 mm
Length of tubulature	175 ± 3 mm
Angle of tubulature	75 ± 3 deg
Thickness of tubulature wall	0.035 to 0.045 in. (0.89 to 1.14 mm)

2.2 *Condenser Tube*—A suitable form of tapered glass condenser tube constructed of heat-resistant glass[3] and having the following dimensions:

Outside diameter of small end	12.5 ± 1.5 mm
Outside diameter of large end	28.5 ± 3.0 mm
Length	360.0 ± 4.0 mm
Length of tapered part	100.0 ± 5.0 mm

The condenser tube shall be as shown in Fig. 2, with a 75-deg angle smooth bend toward the outlet, and with approximately a 100-mm straight section at the outlet.

2.3 *Shield*—A galvanized iron shield, lined with ⅛-in. (3.2-mm) asbestos, fitted with transparent covered windows, of the form and dimensions shown in Fig. 3, to protect the flask from air currents and to prevent radiation. The cover (top) may be of Transite board made in two parts, or it may be of galvanized iron lined with ⅛-in. asbestos.

2.4 *Receiver*—Graduated cylinder, of uniform diameter, with a pressed or molded base and the lip on the top closed so as to make possible an airtight deal with a rubber stopper. The over-all height shall be not less than 248 mm (9¾ in.) nor more than 260 mm (10¼ in.). The cylinder shall be graduated in 1-ml divisions to contain 100 ml, and the graduated portion shall be not less than 177.8 mm (7 in.) nor more than 203.2 mm (8 in.) in length. Each fifth graduation shall be distinguished by a longer line, and the graduations shall be num-

[1] This method is under the jurisdiction of ASTM Committee D-4 on Road and Paving Materials and is the direct responsibility of Subcommittee D04.46 on Durability Tests.

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[2] *Annual Book of ASTM Standards*, Part 15.

[3] Borosilicate glass has been found satisfactory for this purpose.

bered from the bottom up at intervals of 10 ml. The graduations shall not be in error by more than 1 ml at any point on the scale. Immediately above the 100-ml graduation line, approximately 45 mm of chemically resistant glass tubing, 7 mm in outside diameter, shall be sealed in to provide a vacuum connection.

2.5 *Thermometer*—An ASTM High Distillation Thermometer having a range from 30 to 760 F (−2 to +400 C), as specified, and conforming to the requirements for Thermometer 8F or 8C, respectively, as prescribed in ASTM Specification E 1 for ASTM Thermometers.[4]

2.6 *Manometer*—A suitable laboratory manometer or gage for measuring the pressure of the system at the receiver to within 1 mm Hg.

2.7 *Vacuum*—Any suitable source of vacuum capable of reducing the absolute pressure within the system to approximately 4 mm Hg. A suitable vacuum regulator and a surge tank may be inserted in the vacuum line.

2.8 *Rubber Tubing, Stoppers, etc*—The rubber tubing shall be high grade laboratory tubing, having ¼-in. (6.4-mm) bore and ⅛-in. (3.2-mm) wall thickness. All stoppers must be well-fitting, neoprene stoppers. Ground-glass joints and connections may be used if desired.

2.9 *Heater*—The heating unit may consist of a Meker burner or a suitable electric heater.

2.10 *Safety Screen*—A safety screen that adequately shields the operator from the distillation apparatus.

3. Preparation of Sample

3.1 The sample, as received, shall be thoroughly stirred and agitated, warming, if necessary, to ensure a complete mixture before the portion for analysis is removed.

4. Apparatus Assembly

4.1 The flask shall be supported on a tripod or rings over two sheets of 20-mesh wire gauze, 150 mm square, as shown in Fig. 4. It shall be connected to the condenser tube by a right neoprene stopper joint. The thermometer shall be inserted through a stopper in the one neck (the vapor tube shall be closed with a neoprene stopper) with the bottom of the bulb 13 mm (½ in.) from the bottom of the flask. The manometer, vacuum source, etc. shall be connected into the system as shown in Fig. 4. To prevent air leakage into the system, ⅝-in. hose clamps may be used at all the rubber tubing glass connection points. All glass items should be well annealed and of uniform and heavy wall thickness in order to avoid danger of collapse under vacuum.

5. Procedure

5.1 Charge the tared flask with 200 ± 0.1 g of the material to be tested and insert the thermometer. Place the flask in the assembly, tighten the connections, and apply the vacuum. (If trouble is encountered in foaming, heating to 190 F (90 C) before applying the vacuum will be of assistance.) Apply heat slowly. Care must be exercised in applying the vacuum on some materials to prevent them from foaming over. In case of excess foaming, the vacuum may be adjusted to from 10 to 15 mm by opening the vacuum release cock slightly. As foaming subsides the vacuum may be gradually increased to maximum. In some cases it may be necessary to distill over a portion at the lower vacuum level.

5.2 Apply heat so as to bring over the distillate at a rate of between 50 and 75 drops per minute; however, the rate may be varied considerably without altering results, 1 h or less usually being sufficient for the distillation. The temperature shall not exceed 680 F (360 C) and it may be found necessary, in order not to exceed this temperature, to use additional insulation on the flask neck.

5.3 Four millimetres of mercury, absolute pressure, should be sufficient to reduce most materials to the desired residue. In some rare cases, however, it may be necessary to reduce the pressure below 4 mm Hg so that the material may be reduced to the desired penetration at a temperature not exceeding 680 F (360 C).

5.4 When it is estimated that the residue will have the desired penetration, discontinue the heat, tightly close the pinchcock immediately next to the receiver, and open the vacuum release into the vacuum manometer line, per-

[4] *Annual Book of ASTM Standards*, Parts 25 and 44.