Designation: D5/D5M – 13

Standard Test Method for Penetration of Bituminous Materials[1]

This standard is issued under the fixed designation D5/D5M; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This test method covers determination of the penetration of semi-solid and solid bituminous materials.

1.2 The needles, containers and other conditions described in this test method provide for the determinations of penetrations up to 500.

NOTE 1—For guidance in preparing and testing emulsion residue specimens for this test method, please refer to Section 35 of Test Method D244.

1.3 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 nonconformance with the standard.

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]

D36 Test Method for Softening Point of Bitumen (Ring-and-Ball Apparatus)

D244 Test Methods and Practices for Emulsified Asphalts

E1 Specification for ASTM Liquid-in-Glass Thermometers

E77 Test Method for Inspection and Verification of Thermometers

E1137/E1137M Specification for Industrial Platinum Resistance Thermometers

E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids

2.2 *ANSI Standard:*

B46.1 Surface Texture[3]

2.3 *ISO Standard:*

ISO Standard 468 Surface Roughness—Parameters, Their Values and General Rules for Specifying Requirements[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *penetration, n*—consistency of a bituminous material expressed as the distance in tenths of a millimetre that a standard needle vertically penetrates a sample of the material under known conditions of loading, time, and temperature.

4. Summary of Test Method

4.1 The sample is melted (if starting at ambient temperature) and cooled under controlled conditions. The penetration is measured with a penetrometer by means of which a standard needle is applied to the sample under specific conditions.

5. Significance and Use

5.1 The penetration test is used as a measure of consistency. Higher values of penetration indicate softer consistency.

6. Apparatus

6.1 *Penetration Apparatus*—Any apparatus that permits the needle holder (spindle) to move vertically without measurable friction and is capable of indicating the depth of penetration to the nearest 0.1 mm, will be acceptable. The weight of the spindle shall be 47.5 ± 0.05 g. The total weight of the needle and spindle assembly shall be 50.0 ± 0.05 g. Weights of 50 ± 0.05 g and 100 ± 0.05 g shall also be provided for total loads of 100 and 200 g, as required for some conditions of the test. The surface on which the sample container rests shall be flat and the axis of the plunger shall be at approximately 90° to this surface. The apparatus shall have a leveling indicator. The spindle shall be easily detached for checking its weight.

[1] This test method is under the jurisdiction of ASTM Committee D04 on Road and Paving Materials and is the direct responsibility of Subcommittee D04.44 on Rheological 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.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

6.1.1 The leveling indicator shall be verified at least annually with a hand-held level.

6.2 *Penetration Needle:*

6.2.1 The needle (see Fig. 1) shall be made from fully hardened and tempered stainless steel, Grade 440-C or equal, HRC 54 to 60. The standard needle shall be approximately 50 mm [2 in.] in length, the long needle approximately 60 mm [2.4 in.]. The diameter of all needles shall be 1.00 to 1.02 mm [0.0394 to 0.0402 in.]. It shall be symmetrically tapered at one end by grinding to a cone having an angle between 8.7 and 9.7° over the entire cone length. The cone should be coaxial with the straight body of the needle. The total axial variation of the intersection between the conical and straight surfaces shall not be in excess of 0.2 mm [0.008 in.]. The truncated tip of the cone shall be within the diameter limits of 0.14 and 0.16 mm [0.0055 and 0.0063 in.] and square to the needle axis within 2°. The entire edge of the truncated surface at the tip shall be sharp and free of burrs. When the surface texture is measured in accordance with American National Standard B 46.1 or ISO 468 the surface roughness height, Ra, of the tapered cone shall be 0.2 to 0.3 µm [8 to 12 µin.] arithmetic average. The surface roughness height, Ra, of the needle shank shall be 0.025 to 0.125 µm [1 to 5 µin.]. The needle shall be mounted in a non-corroding metal ferrule. The ferrule shall be 3.2 ± 0.05 mm [0.126 ± 0.002 in.] in diameter and 38 ± 1 mm [1.50 ± 0.04 in.] in length. The exposed length of the standard needle shall be within the limits of 40 to 45 mm [1.57 to 1.77 in.], and the exposed length of the long needle shall be 50 to 55 mm [1.97 to 2.17 in.]. The needle shall be rigidly mounted in the ferrule. The run-out (total-indicator reading) of the needle tip and any portion of the needle relative to the ferrule axis shall not exceed 1 mm [0.04 in.]. The weight of the ferrule needle assembly shall be 2.50 ± 0.05 g. (A drill hole at the end of the ferrule or a flat on the side is permissible to control the weight.) Individual identification markings shall be placed on the ferrule of each needle; the same markings shall not be repeated by a manufacturer within a three-year period.

6.2.2 Needles used in testing materials for conformance to specifications shall be shown to have met the requirements of 6.2.1 when tested by a qualified agency.

6.3 *Sample Container*—A metal or glass cylindrical, flat-bottom container of essentially the following dimensions shall be used:

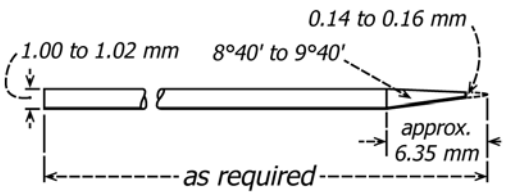

FIG. 1 Needle for Penetration Test

For penetrations below 40:	
Diameter, mm	33–50
Internal depth, mm	8-16
For penetrations below 200:	
Diameter, mm	55
Internal depth, mm	35
For penetrations between 200 and 350:	
Diameter, mm	55–75
Internal depth, mm	45–70
For penetrations 350 to 500	
Diameter, mm	55
Internal depth, mm	70

NOTE 2—For referee testing, the container for testing materials with penetrations below 40 shall be 55 × 35 mm.

6.4 *Water Bath*—A bath capable of maintaining a temperature of 25 ± 0.1°C [77 ± 0.2°F] or any other temperature of test within 0.1°C [0.2°F]. The bath shall have a perforated shelf supported in a position not less than 50 mm from the bottom and not less than 100 mm below the liquid level in the bath. If penetration tests are to be made in the bath itself, an additional shelf strong enough to support the penetrometer shall be provided. Brine may be used in the bath for determinations at low temperatures.

NOTE 3—The use of distilled water is recommended for the bath. Take care to avoid contamination of the bath water by surface active agents, release agents, or other chemicals; as their presence may affect the penetration values obtained.

6.5 *Transfer Dish*—When used, the transfer dish shall have a capacity of at least 350 mL and of sufficient depth of water to cover the large sample container. It shall be provided with some means for obtaining a firm bearing and preventing rocking of the container. A three-legged stand with three-point contact for the sample container is a convenient way of ensuring this.

6.6 *Timing Device*—For hand-operated-penetrometers any convenient timing device such as an electric timer, a stop watch, or other spring activated device may be used provided it is graduated in 0.1 s or less and is accurate to within ±0.1 s for a 60-s interval. An audible seconds counter adjusted to provide 1 beat each 0.5 s may also be used. The time for a 11-count interval shall be 5 ± 0.1 s. Any automatic timing device attached to a penetrometer shall be accurately calibrated to provide the desired test interval within ±0.1 s.

6.7 *Thermometers*—Calibrated liquid–in–glass thermometers of suitable range with subdivisions and maximum scale error of 0.1°C [0.2°F] or any other thermometric device of equal accuracy, precision and sensitivity shall be used. Thermometers shall conform to the requirements of Specification E1 or Specification E2251. Other thermometric devices shall conform to the requirements of Specification E1137/E1137M.

6.7.1 Suitable thermometers commonly used are:

ASTM Number	Range
17C or 17F	19 to 27°C [66 to 80°F]
63C or 63F	–8 to + 32°C [18 to 89°F]
64C or 64F	25 to 55°C [77 to 131°F]

6.7.2 The thermometer used for the water bath shall periodically be calibrated in accordance with Test Method E77. An

alternate thermometric device shall periodically be calibrated in accordance with Specification E1137/E1137M.

7. Preparation of Test Specimen

7.1 If the sample is not sufficiently fluid as received, heat the sample with care, stirring when possible to prevent local overheating, until it has become sufficiently fluid to pour. In no case should the temperature be raised to more than 60°C [140°F] above the expected softening point for tar pitch in accordance with Test Method D36, or to more than 90°C [194°F] above it for petroleum asphalt (bitumen). Heat samples for the minimum time necessary to ensure that they are sufficiently fluid. Stir to ensure that the sample is homogeneous. Avoid incorporating bubbles into the sample.

7.2 Pour the sample into the sample container to a depth such that, when cooled to the temperature of test, the depth of the sample is at least 120% of the depth to which the needle is expected to penetrate. Pour separate portions for each variation in test conditions. If the sample container is less than 65 mm in diameter and the expected penetration is greater than 200, pour three separate portions for each variation in test conditions.

NOTE 4—If sufficient material is available it is recommended to fill the sample container to near the brim.

7.3 Allow to cool in air at a temperature between 15 and 30°C (59 and 86°F) for 45 min to 1.5 h for the small (33 × 16 mm or less) container, 1 to 1.5 h for the medium (55 × 35 mm) container and 1.5 to 2 h for larger containers. Then place the samples together with the transfer dish, if used, in the water bath maintained at the prescribed temperature of test. Allow the small (33 × 16 mm or less) container to remain for 45 min to 1.5 h, the medium (55 × 35 mm) container to remain for 1 to 1.5 h and the larger containers to remain for 1.5 to 2 h.

NOTE 5—If conditions warrant, it is appropriate to loosely cover each container as a protection against dust. A convenient way of doing this is by covering with a lipped beaker.

8. Test Conditions

8.1 Where the conditions of test are not specifically mentioned, the temperature, load, and time are understood to be 25°C [77°F], 100 g, and 5 s, respectively. Other conditions may be used for special testing, such as the following:

Temperature, °C [°F]	Load, g	Time, s
0 [32]	200	60
4 [39.2]	200	60
45 [113]	50	5
46.1 [115]	50	5

In such cases the specific conditions of test shall be reported.

9. Procedure

9.1 Examine the needle holder and guide to establish the absence of water and other extraneous materials. If the penetration is expected to exceed 350 use a long needle, otherwise use a short needle. Clean a penetration needle with toluene or other suitable solvent, dry with a clean cloth, and insert the needle into the penetrometer. Unless otherwise specified place the 50-g weight above the needle, making the total weight 100 ± 0.1 g.

9.2 If tests are to be made with the penetrometer in the bath, place the sample container directly on the submerged stand of the penetrometer. Keep the sample container completely covered with water in the bath. If the tests are to be made with the penetrometer outside the bath, place the sample container in the transfer dish, cover the container completely with water from the constant temperature bath and place the transfer dish on the stand of the penetrometer.

9.3 Using the level indicator, ensure that the apparatus is level.

9.4 Either note the reading of the penetrometer dial or bring the pointer to zero. Position the needle by slowly lowering it until its tip just makes contact with the surface of the sample. This is accomplished by bringing the actual needle tip into contact with its image reflected on the surface of the sample from a properly placed source of light. Quickly release the needle holder for the specified period of time and adjust the instrument to measure the distance penetrated in tenths of a millimetre. If the container moves, ignore the result.

9.5 Make at least three determinations at points on the surface of the sample not less than 10 mm from the side of the container and not less than 10 mm apart. If the transfer dish is used, return the sample and transfer dish to the constant temperature bath between determinations. Use a clean needle for each determination. If the penetration is greater than 200, use at least three needles leaving them in the sample until the three determinations have been completed. If the sample container is less than 65 mm in diameter and the expected penetration is greater than 200, make one penetration in each of the three separate containers prepared as per 7.2.

NOTE 6—With a 55 mm container and a sample with expected penetration greater than 200, it is often not possible to position the needle holder for a third determination without bumping the other two in-place needles. For routine testing it is acceptable to use a single container for all three needle penetrations, moving the first two needles as necessary, provided that the difference between the highest and lowest penetration values does not exceed the value specified in 10.1.

10. Report

10.1 Report to nearest whole unit the average of three penetrations whose values do not differ by more than the following:

	0 to 49	50 to 149	150 to 249	250 to 500
Penetration				
Maximum difference between highest and lowest penetration	2	4	12	20

11. Precision and Bias

11.1 Precision estimates for this test were developed using the AMRL proficiency sample database, which includes paired-sample data representing approximately 16 000 repetitions of the penetration test at 25°C [77°F], and approximately 4000 repetitions of the penetration test at 4°C [39.2°F]. The materials for the database are conventional straight run and blended asphalts with penetration values ranging from 29 to 286 units measured at 25°C [77°F]. Analysis of this data indicates that precision of the test can be described with the following equations:

Symbols:	x = penetration test result (units) σ = standard deviation of penetration test result (units)
Single Operator Precision at 25°C [77°F]	if x < 60, then σ = 0.8 if x > 60, then σ = 0.8 + 0.03 [x–60]
Multilab Precision at 25°C [77°F]	if x < 60, then σ = 2.5 if x > 60, then σ = 2.5 + 0.05 [x–60]
Single Operator Precision at 4°C [39.2°F]	σ = 0.8 + 0.02 (x)
Multilab Precision at 4°C [39.2°F]	σ = 2.5 + 0.08 (x)

11.2 The acceptable range of two results (95 % confidence) can be determined by multiplying the standard deviation estimates given in 11.1 by a factor of 2.83 and rounding to th enearest whole number.

11.3 This test method has no bias because the values determined are defined only in terms of the test method.

12. Keywords

12.1 asphalt; bitumen; penetration