



# Standard Test Method for Penetration of Bituminous Materials<sup>1</sup>

This standard is issued under the fixed designation D 5; 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 reappraisal. A superscript epsilon (ε) indicates an editorial change since the last revision or reappraisal.

*This standard has been approved for use by agencies of the 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.

1.3 The values stated in SI units are to be considered 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:

C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials<sup>2</sup>

D 36 Test Method for Softening Point of Bitumen (Ring-and-Ball Apparatus)<sup>3</sup>

E 1 Specification for ASTM Thermometers<sup>4</sup>

E 77 Test Method for Inspection and Verification of Liquid-in-Glass Thermometers<sup>4</sup>

### 2.2 ANSI Standard:

B46.1 Surface Texture<sup>5</sup>

### 2.3 ISO Standard:

ISO Standard 468 Surface Roughness—Parameters, Their Values and General Rules for Specifying Requirements<sup>5</sup>

## 3. Terminology

### 3.1 Definitions:

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

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-4 on Road and Paving Materials and is the direct responsibility of Subcommittee D04.44 on Rheological Tests.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 04.02.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 04.04.

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

<sup>5</sup> Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

TABLE 1 Precision Criteria

| Material  | Standard Deviation or Coefficient of Variation (Is) or (Is %) | Acceptable Range of Two Test Results (d2s) or (d2s %) |
|---|---|---|
| Single-operator precision:  |   |   |
| Asphalts at 77°F (25°C) below 50 penetration, units                     | 0.35  | 1   |
| Asphalts at 77°F (25°C) 60 penetration and above, percent of their mean | 1.4   | 4   |
| Tar pitches at 77°F (25°C) <sup>A</sup> percent of their mean           | 5.2   | 15  |
| Multilaboratory precision:  |   |   |
| Asphalts at 77°F (25°C) below 50 penetration, units                     | 1.4   | 4   |
| Asphalts at 77°F (25°C) 60 penetration and above, percent of their mean | 3.8   | 11  |
| Tar pitches at 77°F (25°C) <sup>A</sup> units                           | 1.4   | 4   |

<sup>A</sup>Estimates of precision for tar pitches are based on results from 2 pitches with penetration of 7 and 24. Estimates may not be applicable to appreciably harder or softer materials.

## 4. Summary of Test Method

4.1 The sample is melted 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 g 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 spindle shall be easily detached for checking its weight.

### 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,

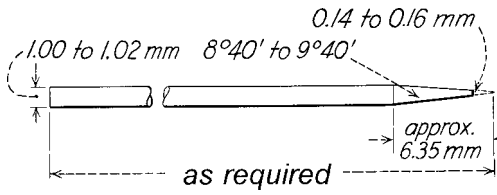


FIG. 1 Needle for Penetration Test

HRC 54 to 60. The standard needle shall be approximately 50 mm (2 in.) in length, the long needle approximately 60 mm (24 in.).<sup>6</sup> 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 2.19 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 3-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*<sup>7</sup>—A metal or glass cylindrical, flat-bottom container of essentially the following dimensions shall be used:

|                                       |    |
|---------------------------------------|----|
| For penetrations below 200:           |    |
| Diameter, mm                          | 55 |
| Internal depth, mm                    | 35 |
| For penetrations between 200 and 350: |    |
| Diameter, mm                          | 55 |
| Internal depth, mm                    | 70 |

6.4 *Water Bath*—A bath having a capacity of at least 10 L and capable of maintaining a temperature of 25 ± 0.1°C or any

other temperature of test within 0.1°C. 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 1—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 must be 5 ± 0.1 s. Any automatic timing device attached to a penetrometer must 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 E 1.

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 E 77.

## 7. Preparation of Test Specimen

7.1 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 above the expected softening point for tar pitch in accordance with Test Method D 36, or to more than 90°C above it for petroleum asphalt (bitumen). Do not heat samples for more than 30 min. 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 10 mm greater than the depth to which the needle is expected to penetrate. Pour two separate portions for each variation in test conditions.

7.3 Loosely cover each container as a protection against dust (a convenient way of doing this is by covering with a lipped beaker) and allow to cool in air at a temperature between 15 and 30°C for 1 to 1.5 h for the small container and 1.5 to 2

<sup>6</sup> Long needles are available from Stanhope-Seta, Park Close, Englefield Green, Egham, Surrey, U.K. TW20 OXD.

<sup>7</sup> Sample Containers are available from Ellisco Inc., 6301 Eastern Ave., Baltimore MD, 21224 and Freund Can Co., 155 West 84th St., Chicago IL, 60620-1298.

h for the taller. Then place the two samples together with the transfer dish, if used, in the water bath maintained at the prescribed temperature of test. Allow the smaller container to remain for 1 to 1.5 h and the taller (6 oz) container to remain for 1.5 to 2 h.

## 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 (Note 2). 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.

NOTE 2—For referee tests, penetrations at temperatures other than 25°C (77°F) should be made without removing the sample from the bath.

9.3 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 (Note 3). Either note the reading of the penetrometer dial or bring the pointer to zero. 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.

NOTE 3—The positioning of the needle can be materially aided by using an illuminated poly-methyl methacrylate tube.

9.4 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.

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

## 11. Precision and Bias

11.1 Use the following criteria for judging the acceptability of penetration results for asphalt at 25°C. The precision at other temperatures is being determined.

11.1.1 *Single Operator Precision*—The single operator coefficient of variation has been found to be 1.4 % for penetrations above 60, and the single operator standard deviation has been found to be 0.35 % for penetrations below 50. Therefore, the results of two properly conducted tests by the same operator on the same material of any penetration, using the same equipment, should not differ from each other by more than 4 % of their mean, or 1 unit, whichever is larger.

11.1.2 *Multilaboratory Precision*—The multilaboratory coefficient of variation has been found to be 3.8 % for penetrations above 60, and the multilaboratory standard deviation has been found to be 1.4 for penetrations below 50. Therefore, the results of two properly conducted tests on the same material of any penetration, in two different laboratories, should not differ from each other by more than 11 % of their mean, or 4 units, whichever is larger.

NOTE 4—These values represent, respectively, the d1s (or d1s %) and d2s (or d2s %) limits as described in Practice C 670.

11.1.3 *Bias*—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

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