Designation: D 5 – 86

Standard Test Method for Penetration of Bituminous Materials[1]

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 reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This method has been approved for use by agencies of the Department of Defense and for listing in the DoD Index of Specifications and Standards.

1. Scope

1.1 This test method covers determination of the penetration of semi-solid and solid bituminous materials. Materials having penetrations below 350 can be tested by the standard apparatus and procedure described. Materials having penetrations between 350 and 500 can be determined using the special apparatus and modifications given in 9.3.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and 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 Statements for Test Methods for Construction Materials[2]
D 2398 Test Method for Softening Point of Bitumen in Ethylene Glycol (Ring-and-Ball)[3]
E 1 Specification for ASTM Thermometers[4]
E 77 Method for Verification and Calibration of Liquid-in-Glass Thermometers[4]
2.2 *ANSI Standards:*
B 46.1 Surface Texture
2.3 *IP Standards:*
IP Standard Thermometers

3. Definition

3.1 *penetration*—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.

4. Summary of 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 full hardened and tempered stainless steel, Grade 440-C or equal, HRC 54 to 60. It shall be approximately 50 mm (2 in.) in length and 1.00 to 1.02 mm (0.0394 to 0.0402 in.) in diameter. 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 surface texture is measured in accordance with American National Standard B46.1 or the United Kingdom equivalent, the surface roughness height of the tapered cone surface shall be 0.2 to 0.3 μm (8 to 12 μin.) arithmetic average. The needle shall be mounted in a brass or stainless steel ferrule and the exposed length shall be within the limits of 40 and 45 mm (1.57 and

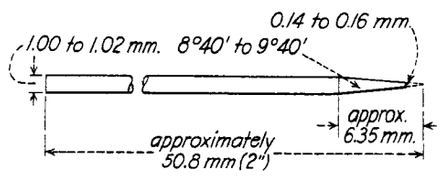

FIG. 1 Needle for Penetration Test

[1] 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.

Current edition approved April 25, 1986. Published May 1986. Originally published as D 5 – 59 T. Last previous edition D 5 – 83.

[2] *Annual Book of ASTM Standards*, Vol 04.03.

[3] Discontinued— see *1984 Annual Book of ASTM Standards*, Vol 04.04.

[4] *Annual Book of ASTM Standards*, Vol 14.01.

1.77 in.). The ferrule shall be 3.2 ± 0.05 mm (0.12 ± 0.003 in.) in diameter and 38 ± 1 mm (1.50 ± 0.04 in.) in length. 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.

NOTE 1—In the United States the manufacturer or commercial laboratories will certify the test needles for conformance to the permissible variations. In the United Kingdom the National Physical Laboratory will certify needles.

6.3 *Sample Container*—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	70
Internal depth, mm	45

NOTE 2—In the United States containers known as tin boxes or as seamless ointment boxes may be obtained in dimensions approximating the above requirements. A 3-oz container is used for penetrations below 200 and a 6-oz container is used for penetrations between 200 and 350.

6.4 *Water Bath*—A bath having a capacity of at least 10 litres 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 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 5-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 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 2398, or to more than 90°C above for 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 an atmosphere at a temperature between 15 and 30°C for 1 to 1½ h for the small container and 1½ to 2 h for the larger. 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 (3 oz) container to remain for 1 to 1½ h and the larger (6 oz) container to remain for 1½ 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
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 matter. Clean a penetration needle with toluene or other suitable solvent, dry with a clean cloth, and insert the needle in the penetrometer (Note 4). Unless otherwise specified place the 50-g weight above the needle, making the total moving load 100 ± 0.1 g. If tests are 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 the water in the bath. If the tests are 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. In either case, 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 by the surface of the sample from a properly placed source of light (Note 5). 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 4—For certain types of asphalts erratic results are sometimes obtained. When this occurs, the customary practice in the United Kingdom is to pretreat the needles by immersing them for 5 min in a 1 % solution of oleic acid prior to drying and running the test. This practice is not followed in the United States.

NOTE 5—The positioning of the needle can be materially aided by using an illuminated methyl methacrylate tube.

9.2 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.

NOTE 6—For referee tests, penetrations at temperatures other than 25°C (77°F) should be made without removing the sample from the bath.

9.3 The needles, containers, and other conditions described in this method provide for determinations of penetrations up to 350. However, the method may be used for direct determinations up to 500 provided special containers and needles are used. The container shall be at least 60 mm in depth. The over-all volume of material in the container should not exceed 125 mL to permit proper temperature adjustment of the sample.

9.3.1 Specially made needles for such determination shall meet all the requirements of 6.2 for dimensions and weight except that the minimum exposed length of the needle shall be 50 mm.

9.3.2 An approximation of the penetration of such high penetration materials may also be obtained by determining the penetration using the standard needle and 6-oz container but with a 50-g loading. The penetration is then calculated by multiplying the result for the 50-g load by the square root of 2. That is:

$$\text{Penetration under 100-g load} = \text{penetration under 50-g load} \times 1.414$$

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) 50 penetration and above, percent of their mean	1.1	3
Tar pitches at 77°F (25°C)[A] 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) 50 penetration and above, percent of their mean	2.8	8
Tar pitches at 77°F (25°C),[A] units	1.4	4

[A] 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.

The report of results obtained by this procedure shall indicate the basis of the test.

10. Report

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

Penetration	0 to 49	50 to 149	150 to 249	250
Maximum difference between highest and lowest determination	2	4	6	8

10.1.1 If the differences are exceeded repeat the test using the second sample.

10.1.2 If the appropriate tolerance is again exceeded, ignore all results and repeat the test completely.

11. Precision and Bias (Note 7)

11.1 Criteria for judging the acceptability of penetration results obtained by this method are given in Table 1. The figures given in Column 2 of Table 1 are the standard deviations or coefficients of variation that have been found to be appropriate for the materials and conditions of test described in Column 1. The figures given in Column 3 are the limits that should not be exceeded by the difference between the results of two properly conducted tests. The range of two acceptable results for single operation precision is often referred to as repeatability. The range for multilaboratory precision is often referred to as reproducibility. A "test result" is the average of acceptable penetrations as described in Section 10.

NOTE 7—For definition of terms and significance of the parameters indicated reference should be made to Practice C 670.