EXPERIMENT NO: 11

PENETRATION OF BITUMINOUS MATERIAL

AASHTO DESIGNATION: T 49-93′
(ASTM DESIGNATION: D 5-86)



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 Section 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 AASHTO standards:

T 53 Softening Point of Asphalt (Bitumen) and Tar in Ethylene Glycol (Ring-and-Ball)

2.2 ASTM Standards:

C 670 Practice for Preparing Precision Statements for Test Methods for Construction Materials
E1 Specification for ASTM Thermometers
E77 Method for Verification and Calibration of Liquid in Glass Thermometers.

2.3 ANSI Standard

B 46.1 Surface Texture
IP Standard Thermometers

3. DESCRIPTION OF TERM

3.1 The penetration of a bituminous material is the distance in tenths of a millimeter that a standard needle penetrates vertically into a sample of the material under fixed conditions of temperature, load and time.

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 permitting movements of the spindle without appreciable friction and which is accurately calibrated to yield results in accordance With the description of the term penetration (see section 3.1) will be acceptable. The surface on which the sample container rests shall be flat and the axis of the plunger shall be at approximately 90 degrees to this surface. The spindle shall be detachable without the use of special toots, for checking its mass. When the needle is mounted in a ferrule, the mass of the moving spindle shall be 47.5 ± 0.05 g. Regardless of the type of mounting of the needle, the total mass of the needle and spindle assembly shall be 50.0 ± 0.05 g. Weights of 50.0 ± 0.05 g and 100.0 ± 0.05 g shall be provided for total loads of 100 g and 200 g (0.9 N and 2 N), depending upon the conditions of test to be applied.

NOTE: 1- The detachability of the spindle prescribed here is intended to apply to penetration apparatus acquired after January 1, 1976. Penetration apparatus acquired before January 1, 1976, may conform either to this standard or to the previous standard (T. 49-74).

6.2 Needle- The needle, Figure 1, shall be made from fully 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 (0.039 to 0.040 in) in diameter. It shall be symmetrically tapered at one end to a cone whose angle shall be within the range of 8.7 to 9.7 deg over the entire length from full needle diameter, and whose axis shall be coincident with the needle axis within 0.0127 mm (0.005 in.) maximum run out (total indicator reading).

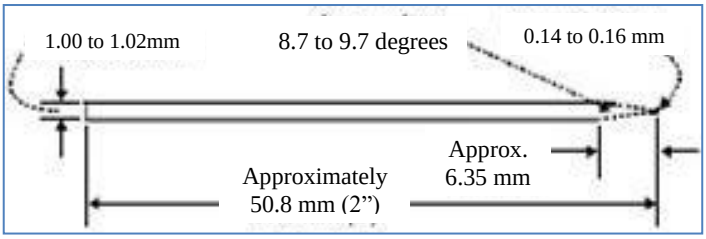


Figure 11.1: Needle for penetration test

After tapering, the point shall be ground off to a truncated cone, the smaller base of which shall be from 0.14 to 0.16 mm (0.0055 to 0.0063 in) in diameter. The truncation shall be square with the needle axis within 2 degree, and the edge shall be sharp and free from burrs.

6.2.1 When the surface texture of the tapered cone surface is measured in accordance with American National Standards Institute Standard B46.1, the surface roughness height shall be 0.2 to 0.3 μm (8 to 12 μ in.) arithmetic average.

6.2.2 The exposed length of the needle when mounted in the chuck of the penetration apparatus or in a ferrule shall be approximately 40 to 45 mm (1.57 to 1.77 in). When the needle is mounted in a ferrule, the ferrule shall be a cylindrical rod, 3.20 ± 0.05 mm (0.126 ± 0.002 in) in diameter and approximately 38 mm (1.5 in) long, made of stainless steel or brass, in which the needle shall be rigidly and coaxially mounted. The weight of the ferrule needle assembly shall be 2.50 ± 0.05 g. (A drill hole is permissible at the end of the ferrule to control 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 3year period.
NOTE 2- The manufacturer or commercial laboratories will certify the test needles for conformance to the permissible variations.

6.3 Container- A container, in which the sample is tested, made of metal or glass cylindrical in shape, and having a flat bottom. The container to be used for materials having a penetration of 200 or less shall have a nominal capacity of 3 oz (90 ml). Its inside dimensions shall be essentially as follows: 55 mm (2.17 in) in diameter and 35 mm (1.38 in) in depth. The container to be used for materials having a penetration over 200 shall have a nominal capacity of 6 oz (175 ml). Its inside dimensions shall be essentially as follows: 70 mm (2.75 in) in diameter and 45 mm (1.77 in) in depth.
NOTE 3- Containers known as tin boxes or as seamless ointment boxes may be obtained in dimensions conforming to the above requirements.

6.4 Water Bath - A water bath maintained at a temperature varying not more than 0.1°C (0.2°F) from the temperature of the test. The volume of water shall not be less than 10 liters. The bath shall have a perforated shelf supported in a position not less than 50 mm from the bottom of the bath

and not less than 100 mm below the liquid level in the bath. The water in the bath shall be substantially free from oil and slime or other organic growth. Brine may be used in the water bath for determinations at low temperatures. If penetration tests are to be made without removing the sample from the bath, a shelf strong enough to support the penetration apparatus shall be provided.

NOTE 4- The use of distilled, demineralized or deionized water is recommended for the bath. Care should be taken 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 for Container- When used; the transfer dish for the container shall be a cylinder with a flat bottom made of glass, metal or plastic. It shall be provided with some means which will ensure a firm bearing and prevent rocking of the container. It shall have a minimum inside diameter of 90 mm (3.5 in) and a minimum depth above the bottom bearing of 55 mm (2.17 in).
NOTE 5- A magnetic strip in the bottom of the transfer dish may be used to prevent the ointment tin from rocking.

6.6 Thermometers for Water Bath- 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.

The following thermometers conforming to the requirements of ASTM Specification E1, ASTM Thermometers are required:

6.6.1 For tests at 25°C (77°F) use an ASTM Saybolt Viscosity Thermometer 17°C (or 17°F) having a range of 19 to 27°C (66 to 80°F). The thermometer shall be immersed in the bath 150±15 mm.

6.6.2 For tests at 0°C (32°F) and 4°C (39.2°F) use ASTM Precision Thermometer 63°C (or 63°F) having a range of -8 to + 32°C (18 to 89°F). The thermometer shall be immersed in the bath 150±15 mm.

6.6.3 For tests at 46.1°C (115°F) use ASTM Precision Thermometer 64°C (or 64°F) having a range of 25 to 55°C (77 to 131°F). The thermometer shall be immersed in the bath 150 ± 15 mm.

6.6.4 Since the accuracy of the test results is dependent upon closely controlled temperature conditions, the thermometer used for the water bath should be calibrated by ASTM E77, Inspection, Test and standardization of Etched Stem Liquid-in-Glass thermometers.

6.7 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 second or less and is accurate to within ± 0.1 second for a 60 second interval. An audible second counter adjusted to provide 1 beat each 0.5 second may also be used. The time for a count interval must be 5 ± 0.1 second. Any automatic timing device attached to a penetrometer must be accurately calibrated to provide the desired test interval within ± 0.1 second.

6.8 Heater- An oven or hot plate, heated by electricity or gas, shall be provided for heating samples.

7. PREPARATION OF SAMPLE

7.1 Heat the sample with care to prevent local overheating until it has become fluid. Then with constant stirring, raise the temperature of the asphalt sample not more than 100°C or l80°F above its expected softening point or the tar pitch sample not more than 56°C or 100°F above its softening point determined in accordance with the Method of test for Softening Point of Bituminous Materials (Ring and Ball Method), T 53. Avoid the inclusion of air bubbles. To reach the pouring temperature, do not heat the softened sample more than 30 minutes.

Then pour it 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 separate samples for each variation in test conditions.

7.2 Loosely cover each container and its contents as a protection against dust, and allow to cool in an atmosphere at a temperature not higher than 30°C or 86°F and not lower than 20°C or 68°F for not less than $1\frac{1}{2}$ hours nor more than 2 hours when the sample is in a 175 ml (6 oz) container and for not less than 1 nor more than $1\frac{1}{2}$ hours when the sample is in a 90 ml (3 oz) container. Then place the sample in the water bath maintained at the prescribed temperature of test, along with the transfer dish if used, and allow it to remain for not less than $1\frac{1}{2}$ hours nor more than 2 hours when the sample is in the 175 ml (6 oz) container, and for not less than 1 nor more than $1\frac{1}{2}$ hours when the sample is in a 90 ml (3 oz) container.

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, 5 second, respectively.

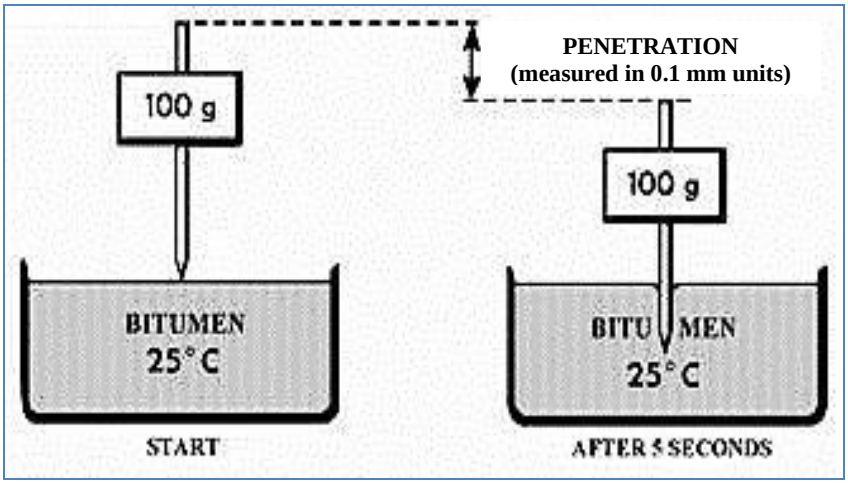


Figure 11.2: Penetration test

Other conditions of temperature, load and time may be used for special testing, such as:

Table 11.1: Temperature and Time for different loads of penetration needle assembly.

Temperature	Load, g	Time
0°C/(32°F)	200	60
4°C/(39.2°F)	200	60
46.1^0C/(115^0F)	50	5

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

9. PROCEDURES

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. Unless otherwise specified, place the 50 g weight above the needle, making the total load of 100 g $\pm$ 0.1g for the needle and attachment. If tests are made with the penetration apparatus mounted in the bath, place the sample container directly on the submerged stand of the penetration apparatus. If tests are made with the sample in the bath and the penetration apparatus outside the bath, place the containers on the shelf provided in the bath. In the above procedures the container shall be kept completely submerged during the complete test. If tests are made using the transfer dish with the penetration apparatus outside the bath, place the sample in a dish filled with water from the bath to a depth to cover completely the sample container. Then place the transfer dish containing the sample on the stand on the penetration apparatus and penetrate immediately. In each case, adjust the needle loaded with the specified weight to just make contact with the surface of the sample. Accomplish this by making contact of the actual needle point with its image reflected by the surface of the sample from a properly placed source of light (Note 8). Either note the reading of the dial or bring the pointer to zero. Then quickly release the needle for the specified period of time and adjust the instrument to measure the

distance penetrated. Observe the sample container as the needle is applied, and if any movement of the container is noted, ignore the result.
NOTE 6- For certain types of asphalt erratic results are sometimes obtained. When this occurs, pre-treat the needles by immersing them for 5 minutes in a 1 percent solution of olene acid prior to drying and running the test.
NOLL 7- For reference tests, penetrations at temperature other than 25^0C (77^0F) should be made without removing the sample from the bath.
NOTE 8- The positioning of the needle can be materially aided by using an illuminated methyl methacrylate rod.

9.2 Make at least three penetrations at points on the surface of the sample not less than 10 mm (3/8 in) from the side of the container and not less than 10 mm (3/8 in) apart. If the transfer dish is used, return the dish and sample to the water bath after each penetration. Before each test, clean the needle with a clean cloth moistened with toluene or other suitable solvent to remove all adhering bitumen, and then wipe with a clean dry cloth. For penetration values greater than 200, use at least three needles, leaving them in the sample until completion of the penetrations.

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 overall 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 Section 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:

Penetration under 100g load = (Penetration under 50g load) x 1.414

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

10. REPORT

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

Table 2: Limit of penetration values for different penetration range.

Penetration	0-49	50-149	150-249	≥ 250
Maximum difference between highest and lowest determinations	2	4	6	8

10.1.1 If the appropriate tolerance is exceeded, ignore all results and repeat the test.

11. PRECISION

11.1 Repeatability- Two results obtained by the same operator on the same

	Standard Deviation Within Laboratory	Standard Deviation Between Laboratory
Asphalt at 25^0C (77^0F) below 50 penetration	0.35 units	1.4 units
Asphalt at 25^0C (77^0F) 50 penetration and above	1.1 percent of their mean	2.8 percent of their mean
Tar pitches at 25^0C (77^0F)	5.2 percent of their mean	1.4 units

Sample in the same apparatus, and on different days should be considered suspect if they differby more than the following amounts:

Asphalt at 25^0C (77^0F) below 50 penetration	1 units
Asphalt at 25^0C (77^0F) 50 penetration and above	3 percent of their mean
Tar pitches at 25^0C (77^0F)	15 percent of their mean

For pitches estimates of precision are based on results from 2 pitches with penetration of 7 and24 estimates may not be applicable to appreciably harder or softer materials

11.2 Reproducibility- Two results obtained by different operators in different laboratories ondifferent days should be considered suspect if they differ by more than the following amounts:

Asphalt at 25^0C (77^0F) below 50 penetration	4 units
Asphalt at 25^0C (77^0F) 50 penetration and above	8 percent of their mean
Tar pitches at 25^0C (77^0F)	4 units*

NOTE: 9- Values show above was obtained by multiplying the estimates of the following population standard deviation (0'p) by 2 v 2.

These estimates of precision are based on the following data (see table entitled "Estimates of Precision"):

	Asphalt at below 50 penetration	Asphalt 50 penetration and above	Tar pitches
Samples	2	7	2
Laboratories	16	27	19
Replicates per sample	3	3	3
Degrees of freedom:			
Within laboratories	32	185	38
Between laboratories	14	89	17

* For definitions of terms and recommended used of precision indexes see ASTM RecommendedPractice E77, for Use of the Terms Precision and Accuracy as Applied to Measurement of a Property of a Material.