

British Standard Methods of test for

Petroleum and its products

Part 49. Penetration of bituminous materials

(Identical with IP 49/83)

Méthodes d'essai du pétrole et des produits pétroliers
Partie 49. Pénétration des matières bitumineuses

Prüfmethoden für Erdöl und Erdölprodukte
Teil 49. Eindringen von bituminösen Materialien

Foreword

BS 2000 comprises a series of test methods for petroleum and its products that have been published previously by the Institute of Petroleum (IP) and have been accorded the status of British Standards. Each method should be read in conjunction with BS 2000 : Part 0 'General introduction', which gives details of the BSI/IP agreement for publication of the series, lists more than 60 methods scheduled to be published in this way in 1982 and 1983, and provides general information on safety precautions, sampling and other matters.

The numbering of the other Parts follows that of the corresponding methods published in 'IP Standards for petroleum and its products, Part 1 Methods for analysis and testing'. BS 2000 : Part 49 is thus identical with IP 49/83 which is itself identical with ASTM D5-73. Previous editions of this IP method were published in A5 format as BS 4691*, which has now been re-numbered and issued in the BS 2000 series.

This Part of BS 2000 incorporates the technical and editorial changes that have been made to the previous edition of IP 49, some of which result from publication of IP 49 as a joint ASTM/IP method. Provision has been made for testing materials having penetrations up to 500; the penetration apparatus and needle, the sample containers and the timing device have been specified more precisely; and thermometers have been specified for tests at 0 °C, 4 °C and 46.1 °C as well as at 25 °C. It is intended to issue further revisions when necessary in line with changes that may be made in subsequent editions of the IP method.

Caution. Attention is drawn to the safety precautions mentioned in Part 0 of this British Standard.

NOTE. The titles of the publications referred to in this Part of this British Standard are listed on the inside back cover.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

WITHDRAWN

*Now withdrawn.

Method (identical with IP 49/83)

1. SCOPE

1.1. This method describes a procedure for determining 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 tested using the special apparatus and modifications given in Section 7.3.

2. SUMMARY OF METHOD

2.1. 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.

3. DEFINITION OF PENETRATION

3.1. The penetration of a bituminous material is its consistency 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. APPARATUS

4.1. *Penetration Apparatus* - any apparatus which 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 is acceptable. The mass of the spindle shall be 47.5 ± 0.05 g. The total mass of the needle and spindle assembly shall be 50.0 ± 0.05 g. Masses 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 should be easily detached for checking its mass.

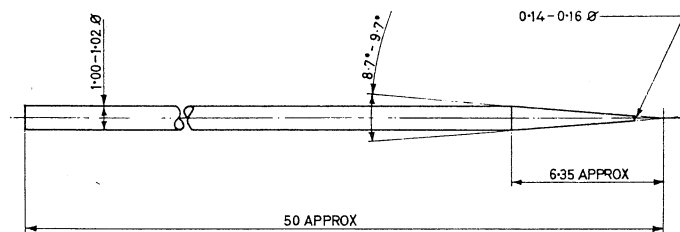


Fig. 1. Needle for penetration test.

4.2. Penetration Needle.

4.2.1. The needle, Fig. 1, shall be made from fully hardened and tempered stainless steel, American Iron and Steel Institute (AISI) grade 440-C or equivalent, Rockwell hardness (C 54 to 60). It shall be approx 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 from burrs. When surface texture is measured in accordance with American National Standard (ANSI) B46.1-1962 or British Standard 1134: Part 1, the surface roughness height of the tapered cone surface shall be 0.2 to 0.3 micrometres (8 to 12 microinches) 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 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

NOTE to 4.2.1. The term 'mass' is preferred to 'weight' in the context of lines 21 and 23.

¹ Under the standardization procedure of the Society this method is under the jurisdiction of the ASTM Committee D4 on Road and Paving Materials. In the IP this method is under the jurisdiction of the Standardization Committee.

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 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.

NOTE 1: There is no direct UK equivalent to steel of AISI grade 440-C, which is however available in the UK under that designation.

4.2.2. Needles used in testing materials for conformity with specifications shall be shown to have met the requirements of Section 4.2.1. when tested by a qualified agency.

NOTE 2: In the United States the manufacturer or commercial laboratories will certify the test needles for conformance to the permissible variations for a stated fee. In the United Kingdom the National Physical Laboratory will certify needles.

4.3. *Sample Container* – a metal or glass cylindrical, flat-bottomed container of essentially the following dimensions shall be used:

For penetrations below 200	Diameter 55 mm Internal depth 35 mm
For penetrations between 200 and 350	Diameter 70 mm Internal depth 45 mm

NOTE 3: In the USA 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. In the UK a container of 55 mm diameter and 57 mm depth can be used for penetrations between 200 and 350.

4.4. *Water Bath* – a bath having a capacity of at least 10 litres and capable of maintaining a temperature of $25 \pm 0.1^\circ\text{C}$ 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.

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

4.6. *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 sec or less and is accurate to within ± 0.1 sec for a 60 sec interval. An audible seconds counter adjusted to provide one beat each half-sec may also be used. The time for an 11 count interval must be 5 ± 0.1 sec. Any automatic timing device attached to penetrometer must be accurately calibrated to provide the desired test interval within ± 0.1 sec.

4.7. *Thermometers* – thermometers shall conform to the requirements prescribed in the Specifications for ASTM Thermometers (ASTM Designation: E1)² or in the specifications for IP Standard Thermometers³.

The following thermometers are required:

For tests at 25°C (77°F)	ASTM 17C or 17F IP 38C
For tests at 0°C (32°F) and 4°C (39.2°F)	ASTM 63C or 63F
For tests at 46.1°C (115°F)	ASTM 64C or 64F

The thermometer used for the water bath shall be calibrated periodically by the Method for Inspection, Test and Standardization of Etched-Stem Liquid-in-Glass Thermometers (ASTM Designation E77).²

5. PREPARATION OF TEST SPECIMEN

5.1. Heat the sample with care, stirring as soon as possible to prevent local over-heating, 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 (ASTM D36⁴–IP 58⁵) for tar pitch, 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.

²Annual Book of ASTM Standards, Part 44.

³Appears in Volume 2 of this publication.

⁴Annual Book of ASTM Standards, Part 15.

⁵Appears in this publication.

NOTES to 4.7. The detailed specifications for IP thermometers are given in BS 2000 : Part 0 : Addendum 1.

There is no direct IP or BS equivalent to ASTM E77 (current edition is E77-74, reapproved 1980, Standard method for verification and calibration of liquid-in-glass thermometers) but general guidance on thermometer calibration is given in BS 1041 : Section 2.1. See also ISO 386 and Annex D of BS 2000 : Part 0 : Addendum 1.

NOTE to 5.1. BS 2000 : Part 58 is identical with IP 58.