



SLOVENSKI STANDARD
SIST EN 1426:2000

01-julij-2000

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Bitumen and bituminous binders - Determination of needle penetration

Bitumen und bitumenhaltige Bindemittel - Bestimmung der Nadelpenetration

Bitumes et liants bitumineux - Détermination de la pénétrabilité a l'aiguille

Ta slovenski standard je istoveten z: **EN 1426:1999**

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ICS:

75.140	Voski, bitumni in drugi naftni proizvodi	Waxes, bituminous materials and other petroleum products
91.100.50	Veziva. Tesnilni materiali	Binders. Sealing materials

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en

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ICS 75.140; 91.100.50

English version

Bitumen and bituminous binders - Determination of needle penetration

Bitumes et liants bitumineux - Détermination de la pénétrabilité à l'aiguille

Bitumen und bitumenhaltige Bindemittel - Bestimmung der Nadelpenetration

This European Standard was approved by CEN on 5 September 1999.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

Foreword

This European Standard has been prepared by Technical Committee CEN/TC 19 "Petroleum products, lubricants and related products", the secretariat of which is held by NNI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by April 2000, and conflicting national standards shall be withdrawn at the latest by April 2000.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

In this standard, annex A is normative and annex B is informative.

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1 Scope

This European Standard specifies a method for determining the consistency of bitumen and bituminous binders. A normal procedure is described for penetrations up to $500 \times 0,1$ mm, but for penetrations above this value, different operating parameters are necessary.

WARNING. The use of 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 the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

EN 58, *Sampling of bituminous binders*

EN 1425, *Bitumen and bituminous binders- Characterization of perceptible properties*

EN 1427, *Bitumen and bituminous binders- Determination of softening point- Ring and Ball method*

EN 10088-3, *Stainless steels – Part 3 : Technical delivery conditions for semi-finished products, bars, rods and sections for general purposes*

EN 12594, *Bitumen and bituminous binders- Preparation of test samples*

prEN 12597:1996, *Petroleum products - Bitumen and bituminous binders - Terminology*

ISO 6508, *Metallic materials - Hardness test - Rockwell test (scales A ; B ; C ; D ; E ; F ; G ; H ; K).*

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3 Definitions

For the purposes of this European Standard, the definitions given in prEN 12597:1996 and the following apply.

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3.1

penetration

consistency, expressed as the distance in tenths of a millimetre that a standard needle will penetrate vertically into a sample of the material under specified conditions of temperature, load and duration of loading.

4 Principle

The penetration of a standard needle into a conditioned test sample is measured. For penetrations up to $500 \times 0,1$ mm the operating parameters are a test temperature of $25\text{ }^{\circ}\text{C}$, an applied load of 100 g, and a duration of loading of 5 s. For penetrations above $500 \times 0,1$ mm the test temperature is reduced to $15\text{ }^{\circ}\text{C}$ but the operating parameters of the applied load and the duration of loading remain unchanged.

5 Apparatus

Usual laboratory apparatus and glassware, together with the following.

5.1 Penetrometer ; an apparatus that permits a needle holder to move vertically without measurable friction and that enables the penetration of the needle to be determined to the nearest 0,1 mm. The needle holder shall be readily detachable from the apparatus and shall have a mass of $47,50\text{ g} \pm 0,05\text{ g}$. A weight of $50,00\text{ g} \pm 0,05\text{ g}$ suitable for attachment to the needle holder shall be provided if not already fixed to the needle holder. The stand upon which the specimen container, or transfer dish, rests shall be flat and horizontal. The 50 g weight shall be fixed underneath the support (see figure 1).

NOTE 1 : An example of a suitable penetrometer is shown in figure 1. To facilitate levelling, the penetrometer should be provided with level adjustment screws.

NOTE 2 : Equipment that controls the penetration time automatically can also be used. Such equipment should be checked regularly for correct penetration time (see 5.6).

5.2 Penetration needle, (see figure 2), made from fully hardened, tempered and polished stainless steel of type X105CrMo17 (1.4125), conforming to EN 10088-3, taking into account that it is not necessary to comply with the minimum content of molybdenum specified in that standard, and of Rockwell hardness C54 to C60 determined in accordance with ISO 6508. The cylindrical body of the needle shall have a diameter of 1,00 mm to 1,02 mm and one end shall be symmetrically tapered by grinding to a cone having an angle of $9^{\circ} 10' \pm 30'$ over the entire cone length. The cone shall be co-axial with the cylindrical body of the needle ; the total axial variation of the intersection between the conical and cylindrical surfaces shall not exceed 0,2 mm. The conical tip of the needle shall be ground square to the axis of the needle within 2° until the diameter at the tip is between 0,14 mm and 0,16 mm. The edge of the ground tip shall be sharp and free from burrs.

For penetrations up to $350 \times 0,1$ mm the length of the needle shall be approximately 50 mm. For penetrations between $350 \times 0,1$ mm and $500 \times 0,1$ mm, use needles that conform to the requirements given for mass and dimensions, but which are longer in length such that the ferrule into which the needle is fixed does not penetrate the material under test.

The needle shall be rigidly mounted in a brass or stainless steel ferrule with 5 mm to 10 mm of the needle inside the ferrule. The run-out of the needle tip or any part of the needle relative to the ferrule axis shall not exceed 1 mm. The ferrule shall be $3,20\text{ mm} \pm 0,05\text{ mm}$ in diameter and $38\text{ mm} \pm 1\text{ mm}$ in length.

The mass of the ferrule needle assembly shall be $2,50\text{ g} \pm 0,05\text{ g}$.

NOTE 1 A drill hole at the end of the ferrule or a flat on the side is possible to control the mass.

Individual identification marking shall be engraved or stamped on the ferrule of each needle ; the same marking shall not be repeated by the manufacturer within a 3 year period.

The needle shall be inspected visually for corrosion and shape and the masses of the needle and spindle checked regularly.

NOTE 2 : The mass of the needle plus needle holder together with the additional mass of the weight provides a moving load of 100 g.

NOTE 3 : It is important that initial and continued compliance with the stringent requirements for the needle should be maintained. Certification of compliance can be obtained through the needle supplier or a qualified agency.

NOTE 4 : For information concerning tolerances of the needle (figure 2), see ISO 286-1, ISO 1829, ISO 1101 and ISO 1302.

5.3 Test sample container, metal or glass, cylindrical, flat-bottomed. The internal depth of the container shall be at least 10 mm greater than the expected penetration, and not less than 35 mm. Its internal diameter shall be at least 55 mm and shall not exceed 70 mm.

NOTE 1 : Recommended dimensions of the test sample container are given in table 1 :

Table 1 : Recommended dimensions of the test sample container

Penetration (0,1 mm)	Internal depth (mm)	Internal diameter (mm)
penetration < 200	35	55
200 ≤ penetration < 350	45	70
350 ≤ penetration < 500	60	70

If insufficient binder is available (e.g. if it was obtained by extraction of a bituminous mix core), place a metal (e.g. aluminium) ring in the test sample container. In order to fulfil the requirements of 7.4.2, use rings with an outer diameter of 53 mm ± 1 mm, and an inner diameter of 36 mm ± 1 mm. For penetrations lower than 100 × 0,1 mm the height of the ring shall be 20 mm ± 1 mm. For penetrations from 100 × 0,1 mm up to 200 × 0,1 mm the height of the ring shall be 30 mm ± 1 mm.

NOTE 2: The precision of the penetration values determined using the metal ring may be different from those given in clause 9.

5.4 Water bath, with a capacity of at least 10 l, and capable of maintaining the temperature of the test sample 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.

NOTE : The use of distilled or deionized water is recommended for the bath. Care should be taken to avoid contamination of the bath water by surface active agents or any other material which may affect the penetration values.

5.5 Transfer dish , for tests outside the water bath. The dish shall have a capacity of at least 350 ml and shall be deep enough to ensure that the test sample container is completely covered with water.

The bottom of the transfer dish shall be so constructed that the dish cannot be rocked when it is placed on the stand of the penetrometer. Similarly the surface on which the test sample container rests shall be made so that the test sample container cannot rock during penetration of the test sample.

NOTE : For temperatures lower than 25 °C, the capacity of the transfer dish should be 1,5 l.

5.6 Timing device, accurate to within 0,1 s.

5.7 Thermometers, solid stem, as specified in annex A.

Other temperature measuring devices may be used instead of mercury stem thermometers. However, the mercury stem thermometer is the reference device and any alternative device employed shall be calibrated so as to provide the same readings as a mercury stem thermometer, recognising and allowing for change of thermal response times compared with the mercury thermometer.

NOTE: When measuring and controlling nominally constant temperatures, as in the method described, alternative devices can indicate greater cyclic variations than mercury thermometers, to an extent depending on the cycle time of heating and the power of the controlled heat input.

6 Sampling

6.1 Take the laboratory sample in accordance with EN 58, taking all necessary safety precautions, and ensuring that the test sample is representative of the laboratory sample from which it is taken. Ensure that the laboratory sample is homogeneous and is not contaminated (see EN 1425).

6.2 Remove a minimum of 100 g of the laboratory sample, if necessary using a warmed knife, and transfer it to a suitable container (see EN 12594).

6.3 Raise the material to the required temperature, i.e. 80 °C to 90 °C above the expected softening point (see EN 1427) and fill one clean (free of dust, grease, rust, etc...) test sample container (5.3). For polymer modified bitumen, the temperature may not exceed 200 °C irrespective of the softening point. Fill the container to a depth such that, when the test sample is cooled to the test temperature, the depth is at least 10 mm greater than the depth to which the needle is expected to penetrate.

6.4 Immediately after filling, loosely cover the test sample container with a container such as a lipped beaker of suitable size.

NOTE 1 : This assists in the elimination of any air bubbles and it is a convenient way of providing protection against dust.

Allow the test sample to cool at an ambient temperature between 15 °C and 30 °C ; for test samples less than or equal to 45 mm deep, cool for 60 min to 90 min and for test samples more than 45 mm to 60 mm deep, cool for 90 min to 120 min provided that the volume of the test sample does not exceed 180 ml; for test samples of more than 180 ml, cool for 60 min to 90 min for each 100 ml of test sample. Then place the test sample in the constant temperature bath for a similar period to that used for cooling, and commence the test.

NOTE 2 : For measurement at temperatures lower than 25 °C, longer cooling times can be required.

6.5 When the conditions are not specified, for penetration values below $500 \times 0,1$ mm, the temperature, total applied load (see 5.2 note 2) and duration of loading shall be 25 °C, 100 g and 5 s, respectively. For penetration values above $500 \times 0,1$ mm, the test shall be carried out at conditions of 15 °C, 100 g and 5 s, respectively.

When other conditions are used, the details shall be reported in full.

NOTE : In this case, the precision can be different from that stated in 9.

7 Procedure

7.1 Preparation of the needle holder and needles

Examine the needle holder and its guide to check that it is free of water and other extraneous matter. Ensure that the needle holder moves freely in its guide. Clean a penetration needle with toluene or other suitable solvent, dry with a clean cloth and insert the needle into the needle holder. Unless otherwise specified, add the 50 g weight and ensure that the total applied load is $100,0 \text{ g} \pm 0,10 \text{ g}$.

NOTE : Erratic results may be obtained with bitumens from certain sources. In such cases pre-treat the needles by immersing for 5 min in a 1% solution of oleic acid in toluene prior to drying.

7.2 Tests in the constant temperature bath

If tests are made with the penetrometer in the bath, place the test sample container directly on the submerged shelf of the penetrometer. Keep the test sample container completely covered with the water in the bath.

7.3 Tests outside the constant temperature bath

If tests are made with the penetrometer outside the bath, place the sample container in the transfer dish which has been kept in the bath with the test samples, cover the test sample container completely with water from the constant temperature bath, and place the transfer dish on the stand of the penetrometer.

For referee tests, make penetrations at temperatures other than 25 °C without removing the test samples from the bath unless the transfer dish is separately controlled so that the correct temperature is ensured.

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7.4 Determination of the penetration

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7.4.1 First determinations

With the test sample container in position, slowly lower the needle until its tip just makes contact with its image reflected by the surface of the test sample. Ensure that the zero position of the needle is noted, then quickly release the needle holder for the specified period of time. If the container moves during the determination, discontinue the test. Adjust the instrument as necessary to determine the penetration of the needle into the sample in tenths of a millimetre.

NOTE : The positioning of the needle can be aided materially by good illumination, e.g. by the use of an illuminated methyl methacrylate tube or a lens.