

SLOVENSKI STANDARD
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Bitumen and bituminous binders - Determination of the Fraass breaking point

Bitumen und bitumenhaltige Bindemittel - Bestimmung des Brechpunktes nach Fraaß

Bitumes et liants bitumineux - Détermination du point de fragilité Fraass
(standards.iteh.ai)**Ta slovenski standard je istoveten z: EN 12593:2007**

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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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English Version

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fragilité Fraass

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This European Standard was approved by CEN on 3 February 2007.

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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 CEN Management Centre has the same status as the official versions.

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

Management Centre: rue de Stassart, 36 B-1050 Brussels

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Foreword

This document (EN 12593:2007) has been prepared by Technical Committee CEN/TC 336 "Bituminous binders", the secretariat of which is held by AFNOR.

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of an identical text or by endorsement, at the latest by September 2007, and conflicting national standards shall be withdrawn at the latest by September 2007.

This document supersedes EN 12593:1999.

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

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1 Scope

This European Standard specifies a method for determining the Fraass breaking point which measures the brittleness of bitumen and bituminous binders at low temperatures.

WARNING — Use of this European Standard can involve hazardous materials, operations and equipment. This European Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this European Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2 Normative references

The following referenced standards are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced standard (including any amendments) applies.

EN 58, *Bitumen and bituminous binders - Sampling bituminous binders*

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

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

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

3 Terms and definitions

For the purposes of this document, the following term and definition applies.

3.1

Fraass breaking point

temperature, expressed in degrees Celsius, at which a film of bituminous binder of a specified and uniform thickness will break under defined loading conditions

4 Principle

A sample of bituminous binder is applied to a metal plate at an even thickness. This plate is submitted to a constant cooling rate and flexed repeatedly until the binder layer breaks; the temperature at which the first crack appears is reported as the Fraass breaking point

5 Apparatus

Usual laboratory apparatus and glassware, together with the following:

5.1 Plates, made of tempered spring steel with the following dimensions: $(41,00 \pm 0,05)$ mm long, $(20,0 \pm 0,2)$ mm wide and $(0,15 \pm 0,02)$ mm thick. The plates shall be kept flat and protected from corrosion when not in use. Any plate that becomes visibly curved or corroded shall be discarded.

5.2 Plate preparation equipment, used for application of the melted sample, and including:

5.2.1 Magnet block with a flat and smooth surface (Figure 1) holding one to three plates with a suitable cover (Figure 2).

5.2.2 Metal support with two distinct zones: one temperature regulated and controlled, the other one cooled by water circulation. The support shall be horizontal and include an air bubble level and level adjustment screws.

5.3 Fraass breaking apparatus, as shown in Figure 3, consisting of the parts described in 5.3.1 to 5.3.3.

NOTE Manual apparatus can be replaced by semi automatic or automatic apparatus reproducing the same conditions.

5.3.1 Bending apparatus, as shown in Figure 4. The clearance between the two tubes, when assembled so that one can move longitudinally within the other, shall not exceed 1 mm. The tubes shall be made of a material that is of low thermal expansion (linear expansion coefficient: $< 40 \times 10^{-6} \text{ 1/K}$) and a poor conductor of heat (thermal conductivity: $< 0,3 \text{ W/K x m}$).

The plate shall be held by two steel clips as shown in Figure 5, the upper clip being attached to the bottom end of the outer tube, and the lower clip being attached to the inner tube by means of a metal connecting piece. The clips shall be coplanar, parallel to the axis of the tube, and secured against twisting. The thermometer shall be mounted in such a way that the connecting piece does not act as a shield between the thermometer bulb and ambient temperature and that the reservoir of the thermometer is the same distance between the wall of the inner tube and the middle of the pre-bended test plate when at rest.

By rotating the crank handle (see Figure 3), which operates a mechanism consisting of a cone of hardened metal, as shown in Figure 6, and a setting screw, the inner tube may be moved up and down relative to the outer tube. Eleven rotations of the handle shall permit the initial distance between the upper and lower clip of $(40,0 \pm 0,1) \text{ mm}$ to be steadily reduced by $(3,5 \pm 0,1) \text{ mm}$.

A steel strut may be used to fix the initial bending of the steel test plate. The height of the strut is such that, when in place, the initial distance between the upper and the lower clips is $(40,0 \pm 0,1) \text{ mm}$.

Use of a semi automatic bending apparatus, from which the raising and lowering of the inner tube is controlled, for example, by a motor-driven cam disc, or of fully automatic apparatus, in which the reduction in temperature is controlled and the breaking point is automatically indicated, is permitted, provided that the test conditions specified in this standard are complied with.

5.3.2 Cooling apparatus, as shown in Figure 3, and comprising the inner test tube (5), the outer test tube (4) and the glass cylinder (1). The bungs (6), (7) and (8) shall be made of either rubber or cork. The bore (9) in the bung (7) may be used for introducing solid carbon dioxide. The cylinder (1) and the inner test tube (5) shall contain a small amount of drying agent. A transparent Dewar vessel having an inside diameter of $(75 \pm 5) \text{ mm}$ may be substituted for the outer test tube (4) and the cylinder (1).

NOTE 1 Care should be taken to ensure that all elements of the apparatus are vertical.

NOTE 2 Suitable bath liquids for the Dewar vessel are alcohols like ethanol, 1-propanol or 2-propanol.

5.3.3 Thermometer, 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 would be provided by the mercury stem thermometer, recognising and allowing for changed thermal response times compared with the mercury thermometer.

NOTE For this test method, in which decreasing temperatures are read during the test procedure, documented corrections should be determined in advance and applied to the observed readings.

5.4 Press, consisting of a base plate, a frame and two metal pressure blocks, measuring 100 mm x 72 mm x 25 mm (see Figure 7). The lower pressure block shall be mounted on an intermediate disk made of non-conducting material (see 5.5) and having the same dimensions as the pressure block.

A recess measuring 72 mm x 60 mm x 0,7 mm shall have been let into the pressure surface of the lower block. A metal plate shall be fixed at the vertex of the upper block with non-conducting material being used to separate the two. The vertex plate shall be fitted with a spindle that is turned by a handle. The thread of the spindle shall pass through the frame.

The depth of the notch shall be between 0,2 mm and 0,5 mm.

Electrical heating elements shall be fitted between the pressure blocks and the non-conducting material (see 5.5), and connected to a control device, by means of which the temperature of the pressure blocks can be varied. The pressure blocks shall be provided with bore holes into which the sensors of temperature measuring instruments shall be inserted.

5.5 Separating films, heat-proof, such as films made of cellulose hydrate, 0,05 mm thick, or silicone-coated paper.

5.6 Gripping pliers, for inserting the test plates between the clips. The ends of the gripping arms shall not be more than 8 mm wide and a block shall be used to prevent the ends approaching each other closer than 35 mm, to prevent excessive flexing of the test plates during insertion.

5.7 Balance, accurate to ± 5 mg.

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6 Sampling and sample preparation

6.1 General

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Take the test 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 12594 and EN 1425).

6.2 Coating test plates

Prepare three plates (5.1) by cleaning with a suitable and appropriate degreasing solvent, then drying and weighing to the nearest 0,01 g or tare with the same accuracy. For the repeatability and reproducibility of test results, it is essential that all tests are carried out using binder films of uniform thickness.

NOTE For instance solvents such as acetone, cyclohexane or methylene chloride can be used.

6.3 Melt application of sample

The plate shall be coated manually.

Weigh (410 ± 10) mg of bituminous binder on to the plate and place the plate on the magnet block (5.2.1).

NOTE 1 The sample can be in a heated (liquid) or unheated (normally solid) form as preferred.

The magnet block shall then be placed on the heating metal support (5.2.2) which is controlled at a temperature not exceeding the Ring and Ball softening point of the bituminous binder (see EN 1427) by more than 80 °C. For polymer modified bitumen, the temperature may not exceed 200 °C irrespective to the softening point.

As soon as the fluidity of the bituminous binder is sufficient, ensure a uniform distribution of the bituminous binder by manipulating the heating support.

NOTE 2 If necessary, a thin bladed instrument can be used (e.g. a rejected penetration needle) to assist in obtaining a uniform distribution.

If the remaining amount of bituminous binder is less than required, prepare a new plate. Allow the plate to rest for 1 min to 2 min to ensure that the entire plate is evenly coated with the bituminous binder and the surface is flat.

By carefully fanning with a flame, dispel any small air bubbles, which may have become entrapped, avoiding local overheating.

Move the magnet block supporting the plate to the cooling side of the support with the help of the cover.

NOTE 3 The total plate preparation should not take more than 10 min.

Leave the plates to lie horizontally on the cooled support at ambient temperature protected by a cover.

6.4 Press-application of sample

For binders with a Ring and Ball softening point (determined according to EN 1427) greater than 100 °C, the plate shall be laid centrally on a separating film measuring 100 mm x 100 mm. Approximately 2 g of binder shall be placed in the centre of the plate and covered with another separating film. The plate thus prepared shall be laid in the recess of the lower pressure block of the press, which has been heated in advance to about 20 °C above the softening point of sample under test (see Figure 8).

The upper pressure block shall be lowered by means of the threaded spindle until it rests on the lower block, and left in this position for 1 min. Then the upper block shall be raised again, and the coated test plate, after being left to cool to ambient temperature, shall be placed in ice water, with the separating films still in position. After approximately 2 min, the separating films shall be removed, and the sample trimmed to the edge of the metal plate with help of a sharp blade. The coated test plate shall then be weighed.

The binder quantity pressed on shall be (410 ± 10) mg. If the coated test plate fails the weight check, then another metal plate shall be coated according to the same procedure.

7 Procedure

7.1 Test conditions

Test the coated test plate 30 min to 240 min after coating starting at a temperature of at least 15 °C above the expected breaking point. Cool at 1 °C/min and bend at every degree Celsius starting at least 8 °C and not more than 12 °C above the anticipated breaking point.

NOTE If necessary, for a relatively high Fraass breaking point, the coated test plate can have a temperature above ambient to allow enough time to stabilize the cooling rate at 1 °C/min.

7.2 Measurement

Insert the coated test plate between the clips with the help of the gripping pliers (5.6). Take care when inserting the test plate to ensure that it bends gently enough for the binder film not to crack at this stage of the test. Should a crack in the film still occur, replace with another coated plate.

Mount the bending apparatus in the inner test tube of the cooling apparatus, and introduce the thermometer so that its bulb is located centrally behind the test plate inserted between the clips. Commence cooling at a rate of 1 °C/min. To achieve this, the space between the inner and the outer test tube shall be filled to a level of at least 100 mm with bath liquid (alcohol or any similar bath liquid), the temperature of which has been