SLOVENSKI STANDARD
SIST EN 12593:2000

01-julij-2000

Bitumen in bitumenska veziva - Določanje pretrgališča po Fraassu

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

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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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EUROPEAN STANDARD

NORME EUROPÉENNE

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English version

Bitumen and bituminous binders - Determination of the Fraass breaking point

Bitumes et liants bitumineux - Détermination du point de fragilité Fraass

Bitumen und bitumenhaltige Bindemittel - Bestimmung des Brechpunktes nach Fraaß

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

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Ref. No. EN 12593:1999 E

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of an identical text or by endorsement, at the latest by May 2000, and conflicting national standards shall be withdrawn at the latest by May 2000.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.

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1 Scope

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

WARNING The use of this standard can 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 when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

EN 58, *Sampling 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 12594, *Bitumen and bituminous binders - Preparation of test samples*.

EN ISO 3838, *Crude petroleum and liquid or solid petroleum products - Determination of density or relative density - Capillary-stoppered pyknometer and graduated bicapillary pyknometer methods (ISO 3838 :1983)*.

3 Terms and definitions

For the purposes of this European Standard, the following definition applies.

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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.

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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, and of the following dimensions: 41,00 mm ± 0,05 mm long, 20,0 mm ± 0,2 mm wide and 0,15 mm ± 0,02 mm thick. The plates shall be kept flat and protected from corrosion when not in use. Any plate which 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. Both apparatus improve the test performance.

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: 40×10^{-6} m/K) and a poor conductor of heat (thermal conductivity: < 0,3 W/K/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 thermometer bulb and ambient temperature.

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 mm ± 0,1 mm to be steadily reduced by 3,5 mm ± 0,1 mm.

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A steel strut may be used to fix the initial bending of 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 mm ± 0,1 mm.

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The use of semi automatic bending apparatus, in 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 and the indication of the breaking point by the bending of the plate are also controlled, 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 mm ± 5 mm may be substituted for the outer test tube (4) and the cylinder (1).

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

5.3.3 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. Therefore 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 the fact of 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 bridge 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 plate 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 bridge.
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 by less than 35 mm, to prevent excessive flexing of the test plates during insertion.

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5.7 Balance, accurate to ± 10 mg, readable to 1 mg.

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6 Sampling and sample preparation

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6.1 General

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 degreasing solvent (such as methylene chloride or acetone), then drying and weighing to the nearest 0,01 g. For the repeatability and reproducibility of test results, it is essential that all tests are carried out using binder films of uniform thickness.

6.3 Melt application of sample

In the case of binders having a Ring and Ball softening point (determined according to EN 1427) of less or equal to 100 ºC, the plate shall be coated manually.

Weigh on to the plate 0,40 × ρ_{25} g ± 0,01 g of bituminous binder where ρ_{25} is the density of the binder at 25 °C in grams per cubic centimetre in accordance with EN ISO 3838, 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 softening point Ring and Ball of the bituminous binder (see EN 1427) by more than 80 ºC.

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.

Reweigh the plate if a significant quantity of bitumen has adhered to any instrument used.

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.

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

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

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6.4 Press-application of sample

In the case of binders having a Ring and Ball softening point (determined according to EN 1427) of greater than 100 ºC, 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 ºC 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 about one minute. 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 0,41 $\times\rho_{25}$ g ± 0,01 g where ρ_{25} is the density of the binder at 25 °C in grams per cubic centimetre in accordance with EN ISO 3838. If the coated test plate fails the weight check, then another metal plate shall be coated according to the same procedure.

NOTE Different factors are given in the equations for calculating the required binder quantity, in 6.4 a factor of 0,41 and in 6.3 a factor of 0,40, because press-application of the binder, unlike melt application, does not produce an edge meniscus.

7 Procedure

7.1 Test conditions

Test the coated test plate 30 min to 240 min after coating at a temperature of at least 15 ºC above the expected breaking point. Cool at 1 ºC/min and bend at every degree Celsius starting at least 8 ºC and not more than 12 ºC 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 ºC/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 in such a way that its bulb is located centrally behind the test plate inserted between the clips. Commence cooling at a rate of 1 ºC/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 alcohol, the temperature of which has been adjusted to match the test plate temperature, the fall in temperature being produced by the addition of small quantities of solid carbon dioxide. The first three minutes are used to establish the fall in temperature at the rate specified. After an initial fall of 3 ºC, the temperature shall continue to fall by 1 ºC every 60 s ± 5 s. No variation shall exceed this maximum permissible variation of ± 5 s, nor shall the variation be averaged over the period of the test.

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Commence flexure of the test plate at a temperature of 10 °C ± 2 ºC above the expected breaking point position.

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Bend and stretch the test plate by turning the handle at a uniform rate of one revolution per second until it is arrested, examine the binder film for the appearance of the first crack and record the temperature at which it occured to the nearest 1 ºC, then, without pause, turn the handle backwards at the same speed.