SLOVENSKI STANDARD
SIST EN 12593:2015

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Nadomešča:
SIST EN 12593:2007

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

Ta slovenski standard je istoveten z: EN 12593:2015

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

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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
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Contents

Page

Foreword

This document (EN 12593:2015) has been prepared by Technical Committee CEN/TC 336 “Bituminous binders”, the secretariat of which is held by AFNOR.

This document supersedes EN 12593:2007.

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of an identical text or by endorsement, at the latest by January 2016, and conflicting national standards shall be withdrawn at the latest by January 2016.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

There are three major changes in this revised standard compared to the former version.

— the standard has been revised due to a normative reference to mercury thermometers. The changes involve mainly the text in 5.3.3 and the present Annex A is now informative;

— regarding sample preparation the procedure for press-application of sample has been deleted, and the maximum sample preparation time of 10 min has been moved from an informative note to the normative text;

— the explanation for determination of Fraass breaking point in the former 7.3 and 7.4 have been editorial changed into the present 7.3 and a flow diagram in a new Figure 7.

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

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 documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (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 subjected 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 ± 0,05) mm long, (20,0 ± 0,2) mm wide and (0,15 ± 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 × 10^{-6} 1/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 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 ± 0,1) mm to be steadily reduced by (3,5 ± 0,1) 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 ± 0,1) 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 ± 5) mm may be substituted for the outer test tube (4) and the cylinder (1).

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

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

5.3.3 Temperature measuring device or thermometer, solid stem, as specified in Annex A.

A temperature measuring device (combining sensor and reading unit) shall:

- have a range from at least -40 °C to 40 °C
- be readable to 0,5 °C or less and
- have an accuracy of 0,5 °C or better.

Sensors based on platinum resistance thermometers have been found suitable but other principles are also allowed. The thermal response time of the sensor shall be comparable with the former used reference (see Annex A). The temperature measuring device shall be calibrated regularly.

Other temperature measuring devices including liquid in glass thermometers according to Annex A may be used. Even though the former temperature reference was stated to be a mercury stem thermometer, the now informative Annex A does not specify the use of mercury. It is important that the manufacturer of the temperature measuring device takes into account the complex conditions of declining temperature gradient during the test, and the device is calibrated against the results formerly achieved by the mercury stem thermometer if allowed by national regulations.

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 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.

5.5 Balance, accurate to ± 5 mg.

5.6 Calibration/Verification, all equipment shall be calibrated/verified at least once per year.

It is recommended to check monthly (in a regular use of the equipment) the temperature measuring device, the cooling rate and the bending detection device.

6 Sampling and sample preparation

6.1 General

Take the test sample in accordance with EN 58 and prepare it in accordance with EN 12594. Ensure that the sample is homogeneous and uncontaminated (see EN 1425).

6.2 Test plate preparation

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.

Due to the manner in which the steel plates are produced, they have a preferred direction to move when bended. This side is called the convex side of the plate shall be determined by gently bending the plate. Coating shall be done on the convex side.

At least 3 samples will need to be prepared, in case the expected breaking point is not correct.

6.3 Coating test plates

For binders with a Ring and Ball softening point (determined according to EN 1427) lower or equal than 100 ºC, 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 ºC for all binders except Polymer Modified Bitumens. For polymer modified bitumen according to

EN 14023, the temperature of the heating support should be between 180 °C and 200 °C irrespective of the softening point; the temperature shall not exceed 200 °C in any case.

As soon as the fluidity of the bituminous binder is sufficient, ensure a uniform distribution of the bituminous binder. This procedure shall be performed as soon as practicable after the plate has been placed on the heating metal support. The magnet block shall preferably be covered during heating.

NOTE 2 If necessary, a pre-heated 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 with the help of the cover or a metallic rod to the cooling side of the support. Protect the plates from dust with the help of the cover; ensure that it does not touch the plates.

The total plate preparation time shall not exceed 10 min.

Leave the plates to lie horizontally on the cooled support protected by a cover, until ambient temperature is reached.

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 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

7.2.1 Manual or semi-automatic apparatus

Insert the coated test plate between the clips with the help of the gripping pliers (5.4). 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 ºC/min. The first 3 min 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 ± 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.

To achieve cooling, 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 adjusted to match the test plate temperature, the fall in temperature being produced by the addition of small quantities of solid carbon dioxide.