SLOVENSKI STANDARD
SIST EN 12592:2007

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Nadomeġļa:
SIST EN 12592:2000

Bitumen in bitumenska veziva - Doloļanje topnosti

Bitumen and bituminous binders - Determination of solubility

Bitumen und bitumenhaltige Bindemittel - Bestimmung der Löslichkeit

Bitumes et liants bitumineux - Détermination de la solubilité

Ta slovenski standard je istoveten z: EN 12592:2007

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 solubility

Bitumes et liants bitumineux - Détermination de la solubilité

Bitumen und bitumenhaltige Bindemittel - Bestimmung der Löslichkeit

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

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

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Foreword

This document (EN 12592:2007) 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of an identical text or by endorsement, at the latest by September 2007, and conflicting national standards shall be withdrawn at the latest by September 2007.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 12592:1999.

This European standard is based on ASTM D 2042-01.

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.

1 Scope

This European Standard specifies a method for determining the degree of solubility of bituminous binders having little or no mineral matter other than recovered bituminous binders from asphalt mixes, in a specific solvent. Toluene is used as the solvent for reference tests.

NOTE Bituminous binders will have varying solubility in different solvents.

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 European Standards are indispensable for the application of this European Standard. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced European 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 12594, *Bitumen and bituminous binders - Preparation of test samples*

ISO 4793, *Laboratory sintered (fritted) filters - Porosity grading, classification and designation*

ISO 5272, *Toluene for industrial use - Specifications*

ISO 5280, *Xylene for industrial use – Specification*

3 Terms and definitions

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

3.1
solubility
portion of material that is soluble in a specific solvent

4 Principle

A sample of bituminous binder is dissolved in a solvent. The solution (with the dissolved sample) is filtered through a layer of powdered glass in a sintered crucible. The insoluble material is then washed, dried, and weighed.

5 Reagents and materials

NOTE Examples of specific solvents are given in 5.1 and 5.2.

5.1 Toluene, conforming to ISO 5272.

5.2 Xylene, conforming to ISO 5280.

6 Apparatus

Usual laboratory apparatus and glassware, together with the following:

6.1 Filtering apparatus,

NOTE Assembled as illustrated in Figure 1. Details of the component parts are as given in 6.1.1 to 6.1.5.

6.1.1 Sintered glass crucible, porosity P4 (see ISO 4793), effective diameter approximately 30 mm.

6.1.2 Powdered glass, borosilicate glass powder, particle diameter approximately between 50 µm and 80 µm.

NOTE Instead of powdered glass, glass fibre or filter grade 934 or equivalent may also be used. However, the use of powdered glass is the reference method. When using glass wool or filter, a Gooch crucible may also be used.

6.1.3 Filter flask, 250 ml or 500 ml, heavy-wall, with a side tube.

6.1.4 Filter tube, 40 mm to 42 mm inside diameter.

6.1.5 Rubber tubing or adaptor, for holding the crucible on the filter tube.

NOTE Other suitable assemblies permitting vacuum filtration with a crucible can be used.

6.2 Oven, capable of maintaining a temperature, *T*, of 15 °C to 25 °C above the boiling temperature of the selected solvent.

6.3 Balance, readable to 0,1 mg.

7 Sampling

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 homogenous and not contaminated (see EN 1425). If the sample contains water, heat a representative portion of between 100 g and 200 g at a temperature not exceeding 130 °C stirring constantly until the binder ceases to foam.

8 Procedure

8.1 Test conditions

Normally the temperature at which the test is run is not critical and may be performed at the laboratory air temperature. For reference tests, the flask and sample in solution shall be placed in a water bath maintained at (25 ± 2) °C for 1 h before filtering.

NOTE Crystallised paraffins may disturb the analysis of the samples with high wax content. To avoid this, use the higher test temperature of 40 °C.

8.2 Preparation of crucible

Weigh (3,0 ± 0,1) g of clean dried powdered glass into the crucible. Place the crucible in the filter tube (6.1.4). Wash the powdered glass (6.1.2) with a small portion of the selected solvent (see clause 5) and filter carefully into the flask with or without a light vacuum, as necessary. Repeat the washing process several times. Place

the crucible on a heater or steam bath for 30 min before placing in the oven (6.2) at the temperature *T* (see 6.2) for at least 20 min. Cool in a desiccator for 25 min to 35 min and determine the mass to the nearest 0,1 mg. Repeat the drying and weighing until constant mass is obtained, i.e. the difference between the two weights shall not exceed 0,5 mg.

8.3 Test method

Transfer (2 ± 1) g of the dry sample, if necessary, using a warmed knife, into a weighed Erlenmeyer flask or any other suitable container (EN 12594).

Determine the mass of the container and sample to the nearest 1 mg. Add 100 ml of the selected solvent to the container in small portions with continuous agitation until all lumps disappear and no undissolved sample adheres to the container. Stopper or cover the container and set aside for at least 15 min.

Place the previously prepared and weighed crucible in the filter tube (6.1.4). Wet the powdered glass with a small portion of solvent and filter the solution carefully into the flask through the powdered glass of the crucible with or without a light vacuum as necessary. If the insoluble matter is appreciable, retain as much of it as possible in the container until the solution has drained through the filter mat. Wash the container with a small amount of solvent and, using a stream of solvent from a wash bottle, transfer all insoluble matter to the crucible. If necessary use a glass rod to remove any insoluble matter adhering to the container and transfer it into the crucible. Thoroughly rinse the glass rod and container and transfer it with the crucible.

Wash the insoluble matter in the crucible with solvent until the filtrate flows substantially colourless, then apply strong vacuum to remove the remaining solvent or place the crucible into a vacuum desiccator. Remove the crucible from the tube or desiccator, wash the bottom free of any dissolved matter, and place the crucible on a heater or a steam bath for 30 min. Place in an oven at the temperature *T* (see 6.2) for at least 20 min. Cool in a desiccator for 25 min to 35 min and determine the mass to the nearest 0,1 mg. Repeat the drying and weighing until constant mass is obtained, i.e. the difference between the two weights is not more than 0,5 mg.

Repeat the determinations, making at least two valid determinations. Two solubility determinations shall be considered valid if they do not differ by more than 0,2 % mass.

9 Calculation

Calculate either the total mass percentage of insoluble matter (x_i) or the mass percentage of soluble matter (x_s), of the sample in the solvent as follows:

$$x_i = \left(m_A / m_B \right) \times 100 \quad (1)$$

$$x_s = 100 - \left[\left(m_A / m_B \right) \times 100 \right] \quad (2)$$

where

x_i is the percentage of insoluble matter, in percent;

x_s is the percentage of soluble matter, in percent;

m_A is the mass of insoluble material, in grams;

m_B is the mass of the dry sample, in grams.

10 Expression of results

Express the solubility as the percentage of mass of soluble material x_s, to the nearest 0,05 %, as the mean of the two valid determinations.

11 Precision

11.1 Repeatability

The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed 0,10 % absolute in only one case in twenty.

11.2 Reproducibility

The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed 0,15 % absolute in only one case in twenty.

NOTE These precision data are not automatically applicable to modified bitumen and for modified bitumen they should only be used for guidance, until criteria data are available.

12 Test report

The test report shall contain at least the following information:

a) type and complete identification of the sample under test;

b) reference to this European Standard;

c) solvent used;

d) results obtained (see Clause 10);

e) any deviation, by agreement or otherwise, from the procedure specified;

f) date of the test;

g) type of auxiliary filtrating medium (glass powder, glass fibre, filter) if used.