SLOVENSKI STANDARD
SIST EN 12592:2000

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Bitumen in bitumenska veziva - Določ evanje 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 12592:1999**

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 19 September 1999.

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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 Central Secretariat has the same status as the official versions.

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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

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of an identical text or by endorsement, at the latest by May 2000, and conflicting national standards shall be withdrawn at the latest by May 2000.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

This European Standard is based on ASTM D 2042-81.

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 referee tests.

NOTE 1 Bituminous binders will have varying solubilities in different solvents.

NOTE 2 For the purposes of this European Standard, the term "% (*m/m*)" is used to represent the mass fraction.

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 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 European Standard, the following definition applies.

3.1
solubility
portion of material which is soluble in a specific solvent.

4 Principle

A sample of bituminous binder is dissolved in a solvent and filtered through a layer of powdered glass in a sintered crucible. The insoluble material is 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, 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 P16 (see ISO 4793), effective diameter approximately 30 mm.

6.1.2 Powdered glass, borosilicated glass powder, particle diameter less than 80 µm.

6.1.3 Filter flask, 250 ml or 500 ml, heavy-wall, with 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, accurate to ± 2 mg, 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 with constant stirring 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 it may be performed at the laboratory air temperature. For referee tests, the flask and sample in solution shall be placed in a water bath maintained at 25 °C ± 2 °C for 1 h before filtering.

8.2 Preparation of crucible

Wash the powdered glass (6.1.2) and crucible (6.1.1) with the selected solvent (see clause 5) and filter under vacuum, handling the crucible at all times with tongs or disposable rubber gloves. Weigh 3,0 g ± 0,1 g of clean dried powdered glass into the crucible. Place the crucible with a uniform thickness of the powdered glass in the oven (6.2) at the temperature *T* (see 6.2) for 15 min, allow to cool in a desiccator and then determine the mass to the nearest 0,1 mg. Store in the desiccator until ready for use.

NOTE Washing of the powdered glass and crucible removes any solvent soluble material and allows any very small particles of powdered glass which can pass through the sinter disc to be removed before weighing.

8.3 Test method

Transfer approximately 2 g ± 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 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. Use a glass rod if necessary to remove any insoluble matter adhering to the container and tranfer it into the crucible. Rinse the glass rod and container and transfer it with crucible thoroughly.

Wash the insoluble matter in the crucible with solvent until the filtrate flows substantially colourless, then apply strong vacuum to remove the remaining solvent. Remove the crucible from the tube, 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 two weighings is not being 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 0,07 % mass.

NOTE In order to obtain precise results, the cooling time in the desiccator should be approximately the same (within ± 5 min) after all heatings. For example, if the empty crucible is weighed after a 30 min cooling in the desiccator, the crucible containing the insoluble matter is weighed after 30 min ± 5 min cooling in the desiccator. Either empty crucibles or crucibles containing insoluble matter that have remained in a desiccator overnight should be reheated in an oven for at least 30 min, then allowed to cool for the prescribed period before weighing.

9 Calculation

Calculate either the total percentage of insoluble matter (m_i) or the percentage of soluble matter (m_s), of the sample in the solvent as follows :

$$m_i = (A/B) \times 100 \ \% \ (m/m)$$

$$m_s = 100 - [(A/B) \times 100] \ \% \ (m/m)$$

where

A is the mass of insoluble material, in grams;
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 m_s, to the nearest 0,05 % *(m/m),* as the mean of the 2 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 % *(m/m)* 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 % *(m/m)* 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) the type and complete identification of the sample under test ;

b) a reference to this European Standard ;

c) the solvent used ;

d) the results obtained (see clause 10) ;

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

f) the date of the test.