SLOVENSKI STANDARD
SIST EN 13302:2003

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Bitumen in bitumenska veziva ï Določ anje viskoznosti bitumnov z rotacijskim viskozimetrom

Bitumen and bituminous binders - Determination of viscosity of bitumen using a rotating spindle apparatus

Bitumen und bitumenhaltige Bindemittel - Bestimmung der Viskosität von Bitumen mit einem Rotationsviskosimeter

Bitumes et liants bitumineux - Détermination de la viscosité a l'aide d'un viscosimetre rotatif

Ta slovenski standard je istoveten z: **EN 13302:2003**

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

Bitumen and bituminous binders - Determination of viscosity of bitumen using a rotating spindle apparatus

Bitumes et liants bitumineux - Détermination de la viscosité à l'aide d'un viscosimètre tournant

Bitumen und bitumenhaltige Bindemittel - Bestimmung der Viskosität von Bitumen mit einem Rotationsviskosimeter

This European Standard was approved by CEN on 17 January 2003.

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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 13302:2003) 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of an identical text or by endorsement, at the latest by October 2003, and conflicting national standards shall be withdrawn at the latest by October 2003.

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, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This European Standard specifies a method for the determination of the dynamic viscosity of bituminous binders at a range of temperatures by means of a coaxial viscometer. The typical range of application is from 50 °C to 250 °C using a rotating spindle apparatus.

The method can be used to measure the apparent viscosity of bitumen at application temperatures.

NOTE Some bitumens may exhibit non-Newtonian behaviour under the conditions of this method. Since non-Newtonian viscosity values are not unique material properties, but reflect the behaviour of the fluid and the measurement system, it should be recognised that measurements made by this method may not always predict performance under the conditions of use. Comparisons between non-Newtonian viscosities should be made only for measurements under similar conditions of shear stress and shear rate.

WARNING — The use of this standard may 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 to 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 (including amendments).

EN 58, *Sampling bituminous binders*.

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

3 Terms and definitions

For the purposes of this European Standard, the following terms and definitions apply.

3.1
shear stress
torque applied to turn the spindle at the set speed

3.2
shear rate
difference between the motions of a fluid near to the rotating spindle and near to the walls of the container at a given distance between rotating spindle and wall of the container

3.3
dynamic viscosity
ratio between the applied shear stress and the velocity gradient (the ratio of the shear stress exerted on a body to its corresponding shear rate)

NOTE It is the measure of the resistance to flow of the liquid. The SI unit of dynamic viscosity is the Pascal second (Pa.s). Millipascal second (mPa.s) is a frequently used sub-unit.

3.4
newtonian fluid
fluid having a viscosity that is independent of the rate of shear

NOTE The ratio of the shear stress to the rate of shear is the viscosity of the fluid. If this ratio is not constant the liquid is non-Newtonian and many fluids exhibit both Newtonian and non-Newtonian behaviour, depending on the temperature and the shear rate.

3.5
apparent viscosity
term used to characterise the resistance to flow of a Newtonian or non-Newtonian fluid

3.6
form factor
specific factor or factors to be applied for the individual equipment in order to obtain the actual viscosity from the readings, mainly due to the geometry of the apparatus

4 Principle

The torque applied to a spindle rotating in a special sample container containing the test sample measures the relative resistance of the spindle to rotation and provides a measure of the dynamic viscosity of the sample. It may be necessary to apply a form factor to yield the actual dynamic viscosity at the test temperature.

5 Apparatus

5.1 Rotating spindle viscometer

5.2 Appropriate spindles

5.3 Temperature control device, which may be an integrated unit with the viscometer, a water bath, an oil bath or an oven. The chosen device should be capable of controlling temperature of the test sample to ± 0,5 °C.

6 Sampling

The material under test shall be sampled in accordance with EN 58 and prepared in accordance with EN 12594.

7 Procedure

7.1 The bulk binder sample shall be placed in an oven maintained at a temperature of about 80 °C above the expected ring and ball softening point temperature or at a maximum of 200 °C which ever is the lower. The container shall be filled to at least 50 % of its volume and shall be covered with a loose lid to protect it against oxidation. The total heating time should be within 1h 15 min to 1h 45 min for samples of 100 ml to 499 ml and 1h 45 min to 2h 15 min for samples of 500 ml to 999 ml. The test sample shall be fluid, before transferring it to the sample container. Test samples shall be tested within 4 h after the start of their preparation.

NOTE Appropriate precautions to minimise oxidation of the samples should be taken, e.g. nitrogen blanketing.

7.2 Set the temperature control device to the desired test temperature and allow the apparatus to equilibrate.

7.3 Refer to the operating instructions for the calibration of the temperature control device if necessary.

7.4 Place the selected spindle in the sample container in the controlled temperature environment and wait until equilibrium temperature is obtained (about 1 h).

7.5 Remove the sample container and add the volume of sample specified for the spindle to be used. Do not overfill the sample container, as the sample volume is critical to meet the system calibration conditions. Put the loaded container back in the controlled temperature environment. Connect the spindle to the rotating spindle viscometer and lower the spindle into the sample to the depth specified by the manufacturer. Allow the system temperature to equilibrate (about 15 min to 30 min).

NOTE Appropriate precautions to minimise oxidation of the samples may be taken, e.g. nitrogen blanketing.

7.6 Switch the rotating spindle viscometer on and allow to stabilise. Take and record three readings at least 60 s apart for each test temperature required. Readings should be between 10 % and 90 % of the working range of the apparatus; if they are not, change one of the variables, i.e. spindle speed, resistive torque or spindle. When results are obtained from different working ranges, the one closest to 50 % of the working range shall be used as the result.

7.7 Where necessary, apply the form factor (see 3.6) to determine dynamic viscosity and calculate the arithmetic mean of the three values.

NOTE Limited reuse of sample is allowed for successive measurements at rising temperature levels.

8 Expression of results

Express the dynamic viscosity as the arithmetic mean of the three values, in Pa.s or mPa.s.

9 Precision

The precision of this method has not been determined. The following precision data are proposed until results of round robin tests are available.

9.1 Repeatability

The difference between two successive 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, differ by more than 5 % in only one case in twenty.

9.2 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, differ by more than 15 % of the mean in only one case in twenty.