SLOVENSKI STANDARD
SIST EN 14896:2006

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Bitumen in bitumenska veziva - Določanje dinamične viskoznosti bitumenskih emulzij ter rezanih in fluksiranih bitumenskih veziv - Metoda z rotacijskim vretenom

Bitumen and bituminous binders - Dynamic viscosity of bituminous emulsions, cut-back and fluxed bituminous binders - Rotating spindle viscometer method

Bitumen und bitumenhaltige Bindemittel - Dynamische Viskosität von Bitumenemulsionen, verschnittenen und gefluxten bitumenhaltigen Bindemitteln - Viskosimeter-Verfahren mit rotierender Spindel

Bitumes et liants bitumineux - Détermination de la viscosité dynamique des émulsions bitumineuses, des liants bitumineux fluidifiés et fluxés - Méthode du viscosimetre a mobile tournant

Ta slovenski standard je istoveten z: **EN 14896:2006**

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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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

Management Centre: rue de Stassart, 36 B-1050 Brussels

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Foreword

This document (EN 14896:2006) 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of an identical text or by endorsement, at the latest by March 2007, and conflicting national standards shall be withdrawn at the latest by March 2007.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of EU Construction Products Directive (89/106/EEC).

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, 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 the determination of the dynamic viscosity of bituminous emulsions, cut-back and fluxed bituminous binders, by means of a rotating spindle viscometer. There are standard application temperatures quoted, although the dynamic viscosity can be measured at other temperatures if required. Similarly, viscosity is quoted at standard rates of shear, although additional measurements can be taken at varying shear rates if required.

NOTE Some bituminous materials 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

The following referenced documents are indispensable for the application of this document. 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 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 rate
velocity gradient in a flowing fluid perpendicular to the stress, measured in units of s^{-1}

NOTE The shear rate calculation depends upon the viscosimeter geometry. This should be mentioned by the viscosimeter manufacturer.

3.2
shear stress
force acting tangentially to a surface divided by the area of the surface, measured in units of $N.\ m^{-2}$, $kg.\ m^{-1}.\ s^{-2}$ or Pa

3.3
dynamic viscosity
ratio between the applied shear stress and the shear rate

NOTE Dynamic viscosity is the measurement of the resistance to flow of the liquid. The SI unit of dynamic viscosity is the Pascal.second (Pa.s), the 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 shear rate is the viscosity of the fluid. If this ratio is not constant the liquid is non-Newtonian. Many fluids exhibit both Newtonian and non-Newtonian behaviour depending on the temperature and the shear rate.

3.5
form factor
specific factor or factors applied for 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

Usual laboratory equipment, together with the following:

5.1 Rotating spindle viscometer

Viscometer(s) with sample containers and rotating spindles allowing following minimum capabilities:

- range of shear rate: 2 s^{-1} to 50 s^{-1}
- range of dynamic viscosities: 10^{-2} Pa.s to 500 Pa.s
- range of temperature: 25 °C to 80 °C

For any given spindle and sample container combination, the operating instructions of the equipment must allow the operator to select the adequate rotation speed so as to obtain a desired shear rate.

5.2 Appropriate spindle

A spindle, appropriate for the equipment, and material to be tested, that allows the viscometer to work within an accuracy range of of ± 10%.

5.3 Sample container

Specific to the rotating spindle viscometer, that allows the equipment to be used in coaxial mode and therefore enable the shear rate control.

5.4 Temperature control device

The chosen device shall be capable of controlling the temperature of the test sample to ± 0,5 °C.

NOTE A thermoregulated bath or cell or a water jacket can be used as chosen device.

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 Bituminous emulsions

7.1.1 Manually agitate the bulk emulsion sample to ensure homogeneity without imparting excessive stress to the sample.

To maintain the integrity of the sample and reduce the chance of evaporation or skinning, ensure the sample is covered when not being agitated.

7.1.2 Switch on the temperature control system and set it to the required test temperature of 40 °C.

NOTE This method may be used at other temperatures but if so, this should be recorded in the test report (see Clause 10).

7.1.3 To enable determination of dynamic viscosity, transfer into the sample container an amount of the sample as defined by the viscosimeter set up instructions, and taking into account the spindle size. Cover the sample container with a suitable cover to reduce the level of evaporation and sample skinning.

7.1.4 Place the sample container with the sample into the test equipment, lower the spindle into the sample to the depth specified by the equipment manufacturer and allow it to reach equilibrium for a length of time such that the emulsion temperature is held steady at (40,0 ± 0,5) °C.

NOTE To accelerate temperature homogenisation, the spindle may be rotated at low speed during this phase.

7.1.5 Ensure the equipment is level.

NOTE A spirit level may be used.

7.1.6 To fully homogenize and break possible storage induced thixotropy effects, set the spindle rotating at a speed such that a shear rate of (50 ± 5) s^{-1} is achieved. Leave for (60 ± 5) seconds.

7.1.7 Set the spindle rotating at a speed such that a shear rate of (2 ± 0,2) s^{-1} is achieved. Allow to stabilize for a period of (30 ± 3) s, time at which the following are recorded:

- torque;
- viscosity;
- shear rate.

If the torque is outside an accuracy range of 10 %, (as defined by the manufacturer instructions), change the spindle for a more appropriate geometry and repeat sub-clauses 7.1.3 onwards.

For low viscosity emulsions (typically, for viscosities below 100 mPa.s), the available equipment and spindles may not allow them to reach the desired accuracy. In this case, viscosity may be measured at a higher shear rate. The actual shear rate employed must be recorded in the report (Clause 10).

NOTE The material may display some instability and therefore exhibit a changing viscosity with applied stress, so timing of the readings is very important. Any observations of unstable behaviour should be recorded.

7.1.8 If the material displays non-Newtonian behaviour, viscosities at other shear rates can be recorded, to characterise the behaviour. In such cases, the initial reading will be taken at a shear rate of 2 s^{-1} as from sub-clauses 7.1.3 onwards. The higher shear rate (typically 50 s^{-1} or higher) should then be employed. The shear rate should then be dropped to intermediate levels of shear and a final reading back at the initial shear rate of 2 s^{-1} obtained.

7.2 Cut-back and fluxed bituminous binders

7.2.1 The bulk binder sample shall be placed in an oven maintained at a temperature which should not exceed whichever is the lower of following temperatures:

— 80 °C above the estimated ring and ball softening point temperature;

— 140 °C for fluxed bituminous binders;

— 80 °C for cut-back bituminous binders.

The container shall be filled to at least 50% of its volume and shall be covered with a loose lid to protect it against evaporation and oxidation. Total heating time should be kept to a minimum to allow pouring the sample into the sample container. Test samples shall be tested within 4 h of the start of their preparation.

7.2.2 Manually agitate the bulk sample to ensure homogeneity without imparting excessive stress to the sample.

7.2.3 Switch on the temperature control system and set to it the required test temperature of 60 °C.

NOTE This method may be used at other temperatures but if so this should be recorded in the test report (see Clause 10).

7.2.4 To enable determination of dynamic viscosity, transfer into the sample container an amount of the sample as defined by the viscosimeter set up instructions, and taking into account the spindle size. Cover the sample container with a suitable cover to reduce the level of evaporation.

7.2.5 Place the sample container containing the sample into the test equipment, lower the spindle into the sample to the depth specified by the equipment manufacturer and allow it to reach equilibrium for a length of time such that the sample temperature is held steady at (60,0 ± 0,5) °C.

NOTE To accelerate temperature homogenisation, the spindle may be rotated at low speed during this phase.

7.2.6 Ensure the equipment is level.

NOTE A spirit level may be used.

7.2.7 To fully homogenize and break possible storage induced thixotropy effects, set the spindle rotating at a speed such that a shear rate of (50 ± 5) s^{-1} is achieved. Leave for (60 ± 5) seconds.

7.2.8 Set the spindle rotating at a speed such that a shear rate of (2 ± 0,2) s^{-1} is achieved. Allow to stabilize for a period of (30 ± 3) s, time at which the following are recorded:

— torque;

— viscosity;

— shear rate.

If the torque is outside an accuracy range of 10 %, (as defined by the manufacturers instructions) change the spindle for a more appropriate geometry and repeat sub-clauses 7.2.4. onwards.

For low viscosity products (typically, for viscosities below 100 mPa.s), the available equipment and spindles may be unable to reach the desired accuracy. In this case, viscosity may be measured at a higher shear rate. The actual shear rate employed must be recorded in the report (Clause 10).

7.2.9 If the material displays non Newtonian behaviour, viscosities at other shear rates can be recorded to characterise the behaviour. In such cases, the initial reading will be taken at a shear rate of 2 s^{-1} as from sub-