SLOVENSKI STANDARD
SIST EN 13302:2010

01-junij-2010

Nadomešča:
SIST EN 13302:2003
SIST EN 13702-2:2004
SIST EN 14896:2006

Bitumen in bitumenska veziva - Določanje dinamične viskoznosti bitumenskih veziv z uporabo rotacijskega viskozimetra

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

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Bitumen und bitumenhaltige Bindemittel - Bestimmung der dynamischen Viskosität von bitumenhaltigem Bindemittel mit einem Viskosimeter mit rotierender Spindel

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

Ta slovenski standard je istoveten z: **EN 13302:2010**

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

SIST EN 13302:2010 **en,fr,de**

2003-01.Slovenski inštitut za standardizacijo. Razmnoževanje celote ali delov tega standarda ni dovoljeno.

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 13302

March 2010

ICS 75.140; 91.100.50

Supersedes EN 13302:2003, EN 13702-2:2003, EN 14896:2006

English Version

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

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

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This European Standard was approved by CEN on 23 January 2010.

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

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Foreword

This document (EN 13302:2010) 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of an identical text or by endorsement, at the latest by September 2010, and conflicting national standards shall be withdrawn at the latest by September 2010.

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.

This document supersedes EN 13302:2003, EN 13702-2:2003, EN 14896:2006.

This document is a merged document based on EN 13302:2003, EN 13702-2:2003 and EN 14896:2006. The reason for merging the three standards is that:

— the three standards did describe basically the same test method performed on three different types of bituminous materials originally developed by three different working groups almost parallel in time;

— minor insignificant differences in the three test methods were highly impractical for quality control/quality assurance in the laboratories using the same equipment for two or three of the mentioned standards.

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1 Scope

This European Standard specifies a method for the determination of the dynamic viscosity of a variety of bituminous binders: modified and unmodified bituminous binders, bituminous emulsions, cut-back and fluxed bituminous binders, by means of a rotating spindle apparatus (a coaxial viscometer).

Standard application temperatures are quoted, although the dynamic viscosity can be measured at other temperatures if required. Similarly, viscosity is quoted at standard rates of shear, although additional measures can be taken at varying shear rates if required.

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*

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3 Terms and definitions

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

3.1
shear stress
force acting tangentially to a surface divided by the area of the surface

NOTE Shear stress is expressed in Newton per square metre ($N \cdot m^{-2}$), kilogram per meter per square second ($kg \cdot m^{-1} \cdot s^{-2}$) or Pascal (Pa)

3.2
shear rate
velocity gradient in a flowing fluid perpendicular to the stress

NOTE 1 Shear rate is expressed in units per second (s^{-1}).

NOTE 2 The shear rate calculation depends upon the viscometer geometry. This should be mentioned by the viscometer manufacturer.

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

NOTE 1 Dynamic viscosity is expressed in Pascal second ($Pa \cdot s$). Millipascal second ($mPa \cdot s$) is a frequently used sub-unit.

NOTE 2 Dynamic viscosity is the measurement of the resistance to flow of a liquid.

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

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 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 (e.g. a cylinder) 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.

NOTE 1 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.

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NOTE 2 Unlike penetration grade bitumen, polymer modified bitumens (PMBs) do not show a straight line on the Heukelom-Diagram. This implies that in order to obtain information about the temperature susceptibility of PMBs, viscosity should be measured at different temperatures.

5 Apparatus

Usual laboratory equipment, together with the following:

5.1 Rotating spindle viscometer, dynamic viscosity measurements with rotational viscometers on the bituminous products addressed by this standard may cover wide ranges of shear rates and viscosities:

– Range of shear rate: $1\ s^{-1}$ to $10^4\ s^{-1}$

– Range of dynamic viscosities: 10^{-2} Pa · s to 10^6 Pa · s

In order to cover all product types mentioned in the Scope (Clause 1), under the most usual test conditions, this standards requires rotational viscometer(s) with sample containers and rotating spindles allowing following minimum capabilities:

– Range of shear rate: $1\ s^{-1}$ to $200\ s^{-1}$

– Range of dynamic viscosities: 10^{-2} Pa · s to 10^3 Pa · s

NOTE 1 In QA/QC, and in the case of rotating cylinders, the following two conditions give an indication of correct measuring geometry:

– Ratio of the radius: $R_2/R_1 \geq 1{,}1$ (reference to Figure 1);
– Difference between the radius $R_1 - R_2$ = 1 mm to 6 mm.

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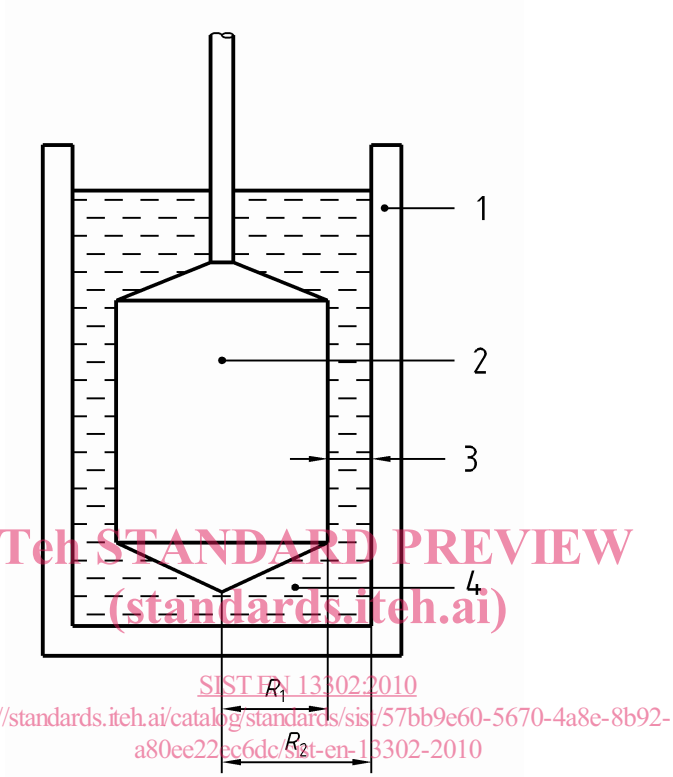
Range of temperature: 40 °C to 200 °C; for oxidised grades: 40 °C to 230 °C

NOTE 2 Typical test temperatures are 40 °C for bituminous emulsions, 60 °C to 90 °C for cut-backs and fluxed bitumens, 90 °C to 180 °C for unmodified or polymer modified bituminous binders.

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



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Key
1 Sample container
2 Spindle
3 Thickness of sample being measured
4 Sample under test
R_1 Inner radius (spindle)
R_2 Inner radius (sample container

Figure 1 — Rotational viscometer (principle)

5.2 Appropriate spindle(s), a spindle, or set of spindles, appropriate for the equipment, that will allow, for the materials to be tested and each specific test condition, dynamic viscosities to be measured with an accuracy close to ± 2 % and at least equal to ± 5 %. If achievable accuracy is not known from the information given by the manufacturer of the equipment, the spindles should be selected so as to obtain dynamic viscosity readings greater or equal to 20 % of the spindle working range for the chosen test conditions.

For low viscosity materials (typically, for viscosities below 100 mPa · s), it may be difficult to fulfil these requirements with existing equipment. Lower accuracy is then permitted but needs to be documented while indicating, in the test report, the actually achieved level of accuracy or, if not known, the position of the reading with regard to the working range of the used spindle (Clause 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 control of shear rate.

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5.4 Temperature control device, such as a water or oil jacket or any other appropriate device. The chosen device shall be capable of controlling temperature of the test sample to ± 0,5 °C.

6 Sampling

A sample from the material to be tested shall be taken in accordance with EN 58 and prepared in accordance with EN 12594.

7 Procedure

7.1 General

This paragraph is divided in several parts to cope with small deviations in procedure due to differences in the product types covered by this European Standard.

7.2 Bituminous binders (unmodified and modified)

7.2.1 Preparation of test sample

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 (in case of oxidised bitumen the maximum temperature is 230 °C). Do not overfill or underfill the sample container, as the sample volume is critical to meet the system calibration conditions. The container shall be covered with a loose lid to protect it against oxidation. The test sample shall be fluid before transferring it to the sample container. The total heating time should not exceed 1 h 45 min for samples of 100 ml to 499 ml and 2 h 15 min for samples of 500 ml to 999 ml. Test samples shall be tested within 4 h of the start of their preparation.

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7.2.2 Preheating

Set the temperature control device to the desired test temperature. Refer to the operating instructions for the calibration of the temperature control device if necessary.

Connect the selected spindle to the rotating spindle viscometer and lower it into an empty sample container placed into the temperature control device. Wait until equilibrium temperature is obtained (about 1 h). If, for practical reasons, the spindle cannot be preheated while being connected to the viscometer, it is admitted that it is connected at ambient temperature and lowered into the hot sample. Temperature equilibration time has then to be extended accordingly.

Additional sample containers may be preheated at the same temperature in a separate oven for at least 45 min.

Remove the sample container from the temperature control device or the oven and add the volume of sample specified for the spindle to be used. Do not overfill or underfill the sample container, as the sample volume is critical to meet the system calibration conditions. Avoid incorporation of air bubbles into the samples.

Place the loaded sample container into the temperature control device. Lower the spindle into the sample to the depth specified by the manufacturer.

Ensure the equipment is level by use of a levelling device (e.g. spirit level).

Allow the system to equilibrate at the test temperature.

Allow the moveable part to rotate during the pre-heating time at a very low shear rate. Maintain the temperature during the test at within ± 0,5 °C up to 100 °C and ± 1,0 °C above 100 °C.