SLOVENSKI STANDARD
SIST-TS CEN/TS 15325:2008

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Bitumen in bitumenska veziva - Določanje ničelne strižne viskoznosti z uporabo reometra s strižno silo po metodi lezenja

Bitumen and bituminous binders - Determination of Zero-Shear Viscosity (ZSV) using a Shear Stress Rheometer in creep mode

Bitumen und bitumenhaltige Bindemittel - Bestimmung der Null-Scherviskosität (ZSV) mit Hilfe eines Schubspannungs-Rheometers im Kriechmodus

Bitumes et liants bitumineux - Détermination de la viscosité a taux de cisaillement nul (ZSV) utilisant un rhéometre a contrainte de cisaillement en mode de fluage

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91.100.50	Veziva. Tesnilni materiali	Binders. Sealing materials

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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EUROPÄISCHES KOMITEE FÜR NORMUNG

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

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Contents

Page

Foreword

This document (CEN/TS 15325:2008) has been prepared by Technical Committee CEN/TC 336 “Bituminous binders”, the secretariat of which is held by AFNOR.

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1 Scope

This European standard describes the determination of Zero Shear Viscosity (ZSV), η_o, for bitumens and bituminous binders, preferably using test temperature domains in which 100 Pa.s < η_o < 50 000 Pa.s. The preferred test temperature is 60 °C but other temperatures for example, 45 °C or 50 °C could be used.

Under these conditions, ZSV (also referred to as the first Newtonian viscosity or absolute viscosity) is a suitable indicator to evaluate the partial contribution of the bituminous binder (including Polymer Modified Binders) to the rutting resistance of asphalt pavement layers.

This European standard describes the determination of ZSV using a Shear Stress Rheometer (SSR) in creep mode.

This method is applicable to unaged, aged and recovered bituminous binders including Polymer Modified Binders (PMBs).

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. Since this European standard involves handling apparatus and binders at high temperatures, always wear protective gloves and eye glasses when handling hot binder, and avoid contact with any exposed skin.

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 12594, *Bitumen and bituminous binders — Preparation of test samples*

3 Terms and definitions

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

3.1
creep test
rheological test in which constant stress is applied to a sample and the resulting deformation is then measured as a function of loading time

3.2
steady-state flow
state at which the rate of deformation reaches a constant value

3.3
steady-state viscosity (SSV)
ratio of the applied stress to the deformation rate under steady-state flow, in Pascal.seconds (Pa.s)

3.4
zero-shear viscosity (ZSV) or Newtonian dynamic viscosity (η_0)
constant value to which the SSV tends at low shear stress or shear rate values, measured in Pascal.seconds (Pa.s)

3.5
compliance (J)
ratio of a component of strain to a component of stress, in Pascal $^{-1}$ (Pa^{-1})

4 Apparatus

4.1 Small tools

— Spatula or any tool that can be used to trim the sample.

— Moulds and vials, for preparing the test specimens. The moulds, where used, shall be silicone or similar material which does not adhere to the test specimen. Vials, where used, shall be glass with a nominal capacity of 10 ml.

4.2 Oven, ventilated laboratory model, capable of being controlled between 50 °C and 200 °C with an accuracy of ± 5 °C.

4.3 Controlled-stress rheometer with temperature control

— rheometer that applies a constant stress to the specimen. It provides torsional or direct shear stress, depending on the specimen shape.

NOTE 1 Usually a microcomputer is connected to the rheometer for data acquisition and processing.

— temperature controller capable of maintaining a temperature in and around the specimen within ± 0,1 °C throughout the test period. The test temperature range depends on the controller and needs to include the operating temperature, generally 60 °C.

The rheometer and temperature control system shall be calibrated at regular intervals in accordance with the quality assurance procedure of the laboratory.

NOTE 2 A suitable method is that the rheometer and temperature control system is calibrated by a means traceable to a national standard. Also, it is advisable to verify the accuracy of the temperature control system by means of a certified temperature measuring device at regular intervals. Also note that external devices read the accurate temperature value only if they are calibrated correctly. A temperature verification procedure is described in Annex A.

NOTE 3 The temperature in the test sample may differ from the temperature read by the device if insufficient equilibration time is used.

4.4 Appropriate testing geometries

The parallel plate geometries are appropriate for all bituminous binders, with preferably a 25 mm diameter and with a 1 mm gap.

NOTE 1 Plates of different diameters and gaps between 0,5 mm and 2 mm can also be used, provided compliance effects of the instrument do not affect the results and the testing is carried out within the specified range of torque and angular deformation and within the linear region.

NOTE 2 Use of parallel plate geometry improves the precision of test method.

In any case, for Polymer Modified Bitumens, the smallest dimension of the sample geometry, d, must satisfy the following:

d > three times the size of the largest polymeric inclusions (usually, d $\geq$ 1 mm).

NOTE The selection of system geometry may affect the accuracy of results. The manufacturer may have determined the operational limits and this information may be available but, if not, it can be determined by running a test specimen over a range of test temperatures using all the test geometries likely to be used in practice.

4.5 Zero-gap setting

Carefully prepare the rheometer plates to receive the test specimen by cleaning with a suitable solvent and soft cleaning cloth or paper. Do not use metal or any other materials, which may damage the surfaces of the plates, and take care not to bend the shaft of the upper plate.

Set the geometry temperature to the required test temperature and wait for thermal equilibrium (10 min to 30 min).

Apply the manufacturer's procedure to reset the gap between the plates prior to loading the test specimen, with both plates at nominally the same temperature.

5 Specimen preparation

5.1 Test specimen preparation

Prepare the binder in accordance with EN 12594.

Two methods can be used:

— specimen preparation in a mould followed by attachment to a test plate (preferred method);

— loading directly into the plate gap.

In the latter case, pour sufficient binder from the vial onto the test geometry for there to be an excess appropriate to the measuring geometry chosen (proceed to sub-clause 5.2).

If using moulds, pour sufficient binder from the vial into the mould. To avoid successive sample heating, several specimens should be prepared at this stage. Discard any binder remaining in the vial.

Store the covered moulds or sheet material at ambient temperature before testing. Any specimen not tested within 7 days shall be discarded.

To minimise the effect of sample preparation, it is advised to pour the specimens 24 h before measuring.

Before testing, if necessary, place the specimens in a refrigerator (approximately 5 °C) to allow them to stiffen for proper, deformation-free release from the moulds. To avoid physical hardening, it is recommended not to leave the specimens in the cool chamber for longer than the time needed to obtain proper stiffness. The recommended time is approximately 10 min and shall not exceed 30 min.

Release the samples from the moulds. Wipe away any release agent that may have been used.

Attach the specimens to the clean, dry test plate.

5.2 Setting the gap and trimming the sample

After the specimen has been placed on one of the test plates as described above, bring the test specimen to the selected gap setting plus 0,05 mm.

Trim any excess binder with a knife or spatula. The tool may be heated on a hot plate or with a flame. After trimming, raise or lower the opposing plate to the set testing gap (± 0,01 mm). Do not trim at this stage. If the

test specimen does not cover the whole measuring plate (indicated by a slight bulging at the periphery of the test specimen), remove it and re-prepare the rheometer plates, and prepare a fresh test specimen.

6 Test execution

6.1 General

The experiment is conducted in two steps.

Step 1 is an optional stress sweep performed in conditions close to steady-state to gain some knowledge on the binder. In case of unknown binders, this step will shed some light on the non-Newtonian character of the binder and allow the proper stress selection for Step 2. In case of bitumen quality control (with a well-known binder), this step may be skipped.

In Step 2, the Zero Shear Viscosity value is determined at a given temperature and at a single (low) stress value. In no case, should Step 1 replace Step 2 in the determination of the ZSV because it is possible that not enough time has been spent in Step 1 to reach a steady state flow.

6.2 Step 1: stress sweep

The following sequence is applied to the same sample, without interruption between the consecutive creep tests:

Table 1 — Sequence applied with the same sample

Stress, Pa σ	Time, min t	Accumulated time, min. t'	Viscosity, Pa.s η
10	100	100	Computed during last 20 min
20	50	150	Computed during last 10 min
50	20	170	Computed during last 4 min
100	10	180	Computed during last 2 min
200	5	185	Computed during last 1 min
500	2	187	Computed during last 24 s
1000	1	188	Computed during last 12 s

NOTE The choice of these seven creep times has been selected to minimise the duration of the stress sweep. It is based on experience but does not guarantee a steady state flow. In other words, it is not certain that the measured properties are steady state viscosities (SSV). The obtained flow curve may thus only be an approximation of the real one.

The computed viscosity values can be plotted versus stress as shown for example in Figure 1 to Figure 3.

Figure 1 shows the flow curve of conventional 10/20 bitumen. The viscosity is almost independent of the applied shear stress. The binder presents a Newtonian character.

Figure 2 shows the flow curve of slightly modified bitumen (< 4 % of elastomer). The binder presents a higher non-Newtonian character when the viscosity plateaus at stresses below 50 Pa.

Figure 3 shows the flow curve of heavily modified bitumen (~ 7 % of polymer). The binder presents a non-Newtonian character and even the existence of the Newtonian plateau is not clear.