SLOVENSKI STANDARD
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Bitumen in bitumenska veziva - Določanje temperature pri enakovrednem strižnem modulu z dinamičnim strižnim reometrom (DSR) - Preskus BTSV

Bitumen and bituminous binders - Determination of equi-shear modulus temperature using a Dynamic Shear Rheometer (DSR) - BTSV test

Bitumen und bitumenhaltige Bindemittel - Bestimmung der Äqui-Schermodultemperatur im Dynamischen Scherrheomter (DSR) - BTSV-Prüfung

Bitumes et liants bitumineux - Détermination de la température d'équi-module de rigidité à l'aide d'un rhéomètre à cisaillement dynamique (DSR) - Essai BTSV

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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 COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

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European foreword

This document (prEN 17643:2021) has been prepared by Technical Committee CEN/TC 336 “Bituminous binders”, the secretariat of which is held by AFNOR.

This document is currently submitted to the CEN Enquiry.

1 Scope

This document specifies the Binder Fast Characterization Test (for short: BTSV test, German: *Bitumen-Typisierungs-Schnell-Verfahren*). The test is conducted using a Dynamic Shear Rheometer (DSR). It is used to quickly characterize bitumen and bituminous binders and to assess the deformation behaviour at high service temperatures.

This document deals with the testing of fresh paving grade bitumen and modified bitumen, as conditioned in a laboratory ageing procedure (e.g. EN 12607-1, EN 14769) and also as recovered from asphalt mixtures. The test procedure in accordance with this document is not applicable for bituminous binders with particles larger than 250 µm (e.g. filler material, granulated rubber).

NOTE The test procedure has not been applied on bituminous binders recovered from bitumen emulsions yet.

The test determines the temperature and the associated phase angle at which a bituminous binder exhibits a defined complex shear modulus in stress-controlled oscillation mode at constant frequency and with continuous increase of the test temperature.

WARNING — The use of this document can involve hazardous materials, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this document to establish appropriate safety and health practices and to ensure that regulatory requirements are fulfilled prior to application of this document.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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*

EN 12597, *Bitumen and bituminous binders - Terminology*

EN 14770, *Bitumen and bituminous binders - Determination of complex shear modulus and phase angle - Dynamic Shear Rheometer (DSR)*

3 Terms and definitions

For the purposes of this document, the terms and definitions given EN 12597 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp

3.1
complex shear modulus
*G**
ratio of peak stress to the peak strain in harmonic sinusoidal oscillation

3.2
phase angle
δ
phase difference in degrees between stress and strain in harmonic oscillation

3.3
shear strain
γ
maximum deflection of the movable plate, measured at the outer edge, relative to the plate gap

Note 1 to entry: The shear strain is given as a percentage.

3.4
range of linear viscoelastic behaviour
range in which the complex shear modulus is independent of the stress or strain

Note 1 to entry: The procedure of BTSV-testing is such that the linear viscoelastic range does not need to be determined prior to testing.

3.5
isochrone
equation or curve on a graph representing the behaviour of a material at a constant frequency

3.6
temperature rate
continuous increase or decrease in the test temperature

3.7
state of non-stationary temperature
non constant temperature state within the test specimen as a result of applying a temperature ramp

4 Principle

This test procedure is used to determine the temperature (T_{BTSV}) at which a bituminous binder exhibits a complex shear modulus of 15 kPa under defined stress-controlled oscillation loading in a state of non-stationary temperature, and to determine the associated phase angle (δ_{BTSV}), without linear viscoelastic domain experimental validation.

The test shall be carried out in stress-controlled oscillation mode at a steadily increasing test temperature.

A parallel-plate system with a plate diameter of 25 mm and a plate gap of 1 mm is used.

The test specimen is continuously subjected to oscillating loading with a frequency of 1,59 Hz and a shear stress of (500 ± 5) Pa. During this loading, the test temperature is steadily increased at a rate of (1,20 ± 0,05) °C/min within a temperature range of 20 °C to 90 °C. The temperature range up to 30 °C serves to establish the temperature rate and is not taken into account in the evaluation.

5 Apparatus

5.1 General

Usual laboratory and glass apparatus, together with the following:

5.2 Dynamic Shear Rheometer (DSR)

A Dynamic Shear Rheometer as described in EN 14770 with parallel plates with a diameter of 25,00 mm ± 0,05 mm, with a constant gap across the entire area of the plates, and with temperature control systems capable of controlling the temperature over a minimum range of 20 °C to 90 °C with an accuracy of ± 0,1 °C shall be used throughout the test period. The temperature control system shall

include both plates in order to minimize the temperature difference between the plates. The rheometer shall operate in stress-controlled mode and be able to record data at intervals of 2,5 s.

NOTE Apparatus with systems where the upper and lower plates have the same diameters make it easier to remove excess material from the inserted sample.

5.3 Moulds for test specimen

Moulds of approximately 18 mm in diameter and 2 mm in depth, made of silicone or a similar material that does not adhere to the test specimen shall be used.

The use of greases or other products that serve as release agents is not permitted, as they can impair the adhesive ability of the test specimen on the rheometer plates.

5.4 Oven

Design with forced ventilation for laboratory operation, capable of being controlled at temperatures between 50 °C and 200 °C with an accuracy of ± 5 °C.

6 Preparation of rheometers

6.1 Set up

The rheometer shall be set up in accordance with the manufacturer's instructions. A parallel-plate-system with a plate diameter of 25 mm and a plate gap of 1 mm shall be selected.

The rheometer and the temperature control system should be checked and adjusted at regular intervals, and calibrated if necessary.

The rheometer and temperature control system should be calibrated at regular intervals in accordance with the quality assurance procedure of the laboratory. A suitable method is to calibrate the rheometer and temperature control system 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. Note shall be taken that external devices read the accurate temperature value only if they are calibrated correctly. A temperature verification procedure is described in Annex A.

The plates are to be prepared carefully by cleaning them with the aid of a suitable cleaning agent and a soft cleaning cloth or paper towel. Metallic materials or other materials that could damage the surfaces of the plates shall not be used, and it shall be ensured that the shaft of the upper plate is not bent.

6.2 Zero gap setting

For initialisation, the gaps between the plates need to be set to zero to give a reference for the gap change for the thermal expansion of the geometry. Prior to loading the first test specimen, the zero gap is set with both clean plates at ambient temperature.

NOTE For temperature control systems with minimized thermal gradients within the gap, the zero gap can be set at any temperature assuring thermal equilibrium of the geometry.

If the DSR has no gap compensation feature, the zero gap should be set at the mid-point of the temperature range to be tested.

7 Sample preparation

7.1 General

WARNING — This document involves handling of apparatus and binders at very high temperatures. Always wear protective gloves and eyewear when handling hot binders, and avoid contact with any exposed, unprotected skin.

The samples are to be taken in accordance with EN 58 and prepared in accordance with EN 12594.

7.2 Heating procedure for the preparation of the binder

Avoid prolonged heating of the bulk binder sample and use the heating periods of EN 12594 as the maximum time span prior to withdrawal of (a) sub-sample(s). For very large bulk samples, it is convenient to redistribute the binder in smaller bulk samples after heating and careful homogenization.

Place the sample in the oven maintained at a temperature that does not exceed 180 °C. For polymer-modified binders, the temperature shall be kept in accordance with EN 12594. 200 °C shall not be exceeded.

The sample shall not be reheated more than twice and shall be homogenized before preparing test specimen.

Reheating times for sub-samples shall conform to following requirements:

— < 50 g: max. 15 min
— 50 g to 100 g: max. 30 min
— 100 g to 500 g: max. 1 h
— 500 g to 1 kg: max. 2 h

7.3 Preparation of test specimens and conditioning

When the binder reaches the required temperature after the heating period, stir and mix with a spatula to ensure homogeneity (especially for polymer modified binders); or after the heating period, remove a sub-sample of convenient size for safe handling and of sufficient volume, to prepare the required number of test specimens plus approximately 50 %.

Pour the binder into moulds, avoiding air bubbles. The moulds shall be stored at ambient temperature. If the ambient temperature is higher than 30 °C, specimens can be cooled down for storage, but not below a temperature of 5 °C. All specimens shall be covered.

A new test specimen shall be used for each measurement, it is therefore advisable to produce several test specimens at once.

A minimum storage duration of 30 min before the de-moulding and testing procedure should be maintained for all bituminous binders that do not exhibit phenomena like crystallization (e.g. EVA modified binders). For these modified binders, the minimum storage duration is 12 h. A maximum delay of two weeks should not be exceeded for all bituminous binders. The storage time should be stated in the test report.

To avoid contamination of the surface of the test specimen by contact with skin, de-moulding and loading of the test specimen should be carried out using clean gloves.

8 Procedure

8.1 Placing the specimen onto the rheometer

To ensure bonding between the test specimen and the plates, the plates shall be preheated to (80 ± 10) °C. The target temperature shall be held constant for a duration of at least 5 min.

If binders exhibit low viscosity, it can be necessary to preheat the plates to a lower temperature.

If heating is carried out in a water bath, it shall be ensured that the plates are dry before the test specimen is loaded.

Load the test specimen is into the heated system.

8.2 Gap setting

The gap for 25 mm plate diameters is 1,0 mm.

Bring the test specimen to the gap of 1,05 mm at the specimen load temperature (see 8.1). Either the normal force should be limited to 1,0 N maximum during gap setting or a waiting time of at least 5 min needs to be maintained after gap setting.

Then, any excess binder shall be trimmed with a knife, a spatula or a special trimming tool. It has been found helpful to heat the trimming tool before trimming. After trimming, the opposite plate shall be raised or lowered to the set testing gap (1,00 ± 0,01) mm. Trimming at this stage is not permitted. If the test specimen does not cover the whole measuring plate (indicated by a slight bulging at the periphery of the test specimen), the rheometer plates shall be removed and re-prepared; and a fresh test specimen shall be prepared. The entire process should not take more than 10 min.

8.3 Test conditions

The plate gap of (1,00 ± 0,01) mm shall be kept constant during the entire duration of the test.

After the start temperature of 20 °C has been achieved, an equilibrium adjustment period of (15 ± 1) min shall be observed.

The test starts at a temperature of (20,0 ± 0,1) °C and ends when the complex shear modulus falls below the value of G^* = 14 kPa. During the test, the temperature shall increase steadily at a rate of (1,20 ± 0,05) °C/min.

The test specimen is to be subjected to a constant shear stress of (500 ± 5) Pa under oscillation at a frequency of f = 1,59 Hz in the parallel-plate system during the entire test duration.

8.4 Measurement

During the measurement, values of the complex shear modulus, phase angle and temperature shall be recorded at intervals of $t \leq 2{,}5$ s.

9 Expression of results

The temperature (T_{BTSV}) at which the complex shear modulus is (15,00 ± 0,05) kPa shall be determined from the measured values. The phase angle (δ_{BTSV}) measured at this temperature shall be determined (see Figure 1 for principle).

If there are no measured values within the range of G^* = (15,00 ± 0,05) kPa, $T_{(BTSV)}$ and $\delta_{(BTSV)}$ shall be calculated by interpolating between the nearby values; an exponential function shall be used for calculating (T_{BTSV}) and a linear function shall be used for calculating (δ_{BTSV}).

Two individual values of the temperature (T_{BTSV}) are compatible with one another if their difference $a \leq 0{,}5$ °C. The result (T_{BTSV}) shall be calculated as the arithmetic mean of two individual