SLOVENSKI STANDARD
SIST EN 17643:2022

01-september-2022

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

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

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

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

Ta slovenski standard je istoveten z: **EN 17643:2022**

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 17643:2022 **en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 17643

July 2022

ICS 75.140; 91.100.50

English Version

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Ref. No. EN 17643:2022 E

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European foreword

This document (EN 17643:2022) 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of an identical text or by endorsement, at the latest by January 2023, and conflicting national standards shall be withdrawn at the latest by January 2023.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

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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 characterize bitumen and bituminous binders and to assess the deformation behaviour at high service temperatures.

The test procedure described in this document covers the testing of paving grade bitumen or modified bitumen, as fresh (unused) binders, as well as binders after laboratory ageing conditioning (e.g. EN 12607-1, EN 14769), and also binders that have been 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. 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.

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 https://www.electropedia.org/

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

3.1
complex shear modulus
$|G^*|$
ratio of the amplitude of the shear stress to the amplitude of the shear strain in harmonic sinusoidal oscillation

Note 1 to entry: The (mathematical) real part of the complex shear modulus $|G^*|$ is G´. It is associated with the elastic part of material behaviour which represents energy stored during a shear cycle. The real part is the complex shear modulus multiplied with cosine of phase angle expressed in degrees.

Note 2 to entry: The (mathematical) imaginary part of the complex shear modulus $|G^*|$ is G´´. It is associated with the viscous part of material behaviour which represents energy dissipated during a shear cycle. The imaginary part is the complex shear modulus multiplied with sine of phase angle expressed in degrees.

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 shear stress or shear 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
temperature ramp
steadily increasing or decreasing test temperature

3.8
state of non-stationary temperature
non-constant temperature state within the test specimen as a result of applying a temperature ramp with a steady temperature rate

4 Principle

This standard 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 $\delta_{(BTSV)}$, without linear viscoelastic domain experimental validation.

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

NOTE Testing can also be done using strain-controlled mode; however, this is not within the scope of the BTSV method as standardized.

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. During this loading, the test temperature is steadily increased at a constant rate 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), as described in EN 14770, with either an integral temperature control system or temperature control attachments, capable of controlling the temperature over a minimum range of 20 °C to 90 °C with an accuracy of ± 0,1 °C throughout the test period. The rheometer shall be fitted with parallel plates with a diameter of 25,00 mm ± 0,05 mm, with a constant gap across the area of the plates. The temperature control system shall encompass both plates to avoid temperature gradients across the plates. When the test specimen is immersed in liquid other than water, ensure that the liquid does not affect the properties of the material being analysed. The rheometer shall operate in stress-controlled mode and be able to record data at intervals of 2,5 s.

NOTE 1 When liquid is used to immerse the test specimen, a water/glycol mixture has been found to be suitable. The proportions used depend on how low the temperature intended for testing is. Rheometers using radio frequency (RF) heating and/or liquid gas cooling or other heating/cooling systems can be used in accordance with the manufacturer's instructions.

Where the bottom plate nominally has the same diameter as the top plate, a visual check should be made to ensure the two plates are vertically aligned. If there is any doubt as to the alignment of the top and bottom plates, the manufacturer, or a qualified technician, should re-align the plate geometry.

NOTE 2 Apparatus with systems where the upper and lower plates have the same diameters make it easier to remove excess material from the inserted sample.

NOTE 3 For some binders, e.g. recovered from reclaimed asphalt, the temperature range can be adjusted, allowing higher testing temperatures. In these cases, the start temperature can be increased.

5.3 Moulds or sheet materials, for the preparation of the test specimens. The moulds or sheet material, where used, shall be of silicone or similar material, which does not adhere to the test specimen.

For a testing geometry with a diameter of 25 mm and a gap setting of 1 mm as for this standardized procedure, a mould with a cavity of approximately 18 mm in diameter and 2 mm deep may be used. In any case, the operator shall ensure adequate filling of the gap according to 8.2.

The use of grease or other anti-stick products should be avoided because they can affect the adherence of the specimen to the rheometer plates.

5.4 Oven, ventilated laboratory model, 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

Set up the rheometer in the sequence given in 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 temperature control system should be calibrated at regular intervals in accordance with the quality assurance procedure of the laboratory. The rheometer and temperature control system should be 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. Take note that external devices read the accurate temperature value only if they are calibrated correctly. A temperature verification procedure is described in Annex A.

6.2 Zero gap setting

For initialisation, the gap 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

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

7.2 Heating procedure for the preparation of the binder

Prepare the bituminous binder in accordance with EN 12594.

If the specification grade of the binder is known, the upper softening point limit may be used.

Avoid prolonged heating of the bulk binder sample, and use the heating periods given in EN 12594 as the maximum time prior to withdrawal of (a) sub-sample(s). Place the sample in the oven maintained at a temperature of (85 ± 5) °C above the expected softening point of the binder, or at 180 °C, whichever is the lower. For polymer modified binders, the temperature shall be in accordance with EN 12594.

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

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

Moulds or sheet materials may be used for all types of binders.

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 for safe handling and of sufficient volume, to prepare the required number of test specimens plus approximately 50 %.

Pour the binder into moulds or directly onto sheets. Care shall be taken that there are no air bubbles in the specimen. The moulds shall be stored at ambient temperature. If the ambient temperature is higher than 30 °C or the binder is very soft, specimens may 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 shall be maintained for all bituminous binders. For modified binders that exhibit phenomena such as crystallization (e.g. EVA modified binders), the minimum storage duration shall be increased to 12 h. A maximum delay of two weeks shall not be exceeded for all bituminous binders. The storage time shall 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 into the rheometer

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

When using moulds or sheet material, the specimens may be placed in the refrigerator but not below 5 °C, for a maximum time of 30 min prior to de-moulding. De-moulding and loading onto the rheometer should take place just after removal from the refrigerator.

Specimen installation shall be performed in conditions ensuring satisfactory adhesion of the test specimen to the rheometer plate, resetting rheologic history of the specimen by allowing sufficient molecular mobility and ensuring adequate filling of the gap (see 8.2). If the sample flows out of the gap before reaching gap setting and there is no adequate filling, the installation temperature shall be reduced.

For preheating, set the temperature of both plates to (80 ± 10) °C. The target temperature shall be held constant within a tolerance of ± 1 °C for a period of at least 5 min. If the upper plate has no heating, it can be warmed by contact with the lower plate and/or using a water bath. 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 specimen and set the trimming gap according to 8.2.

Alternative temperatures may be used for the temperature for both plates, provided that adhesion takes place between the binder and the plate, and that the binder is sufficiently fluid to allow the gap to be achieved. If binders exhibit low viscosity, it can be necessary to preheat the plates to a lower temperature.

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). The normal force should be monitored during gap setting and reach the key value of 1,0 N or below to continue the