SLOVENSKI STANDARD
SIST-TS CEN/TS 15324:2008

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Bitumen in bitumenska veziva - Določanje ravnotežne temperature viskoznosti (temperatura ekviviskoznosti) na osnovi nizke strižne viskoznosti z uporabo dinamičnega strižnega reometra na nizko frekvenčno osciloskopski način

Bitumen and bituminous binders - Determination of equiviscous temperature based on Low Shear Viscosity using a Dynamic Shear Rheometer in low frequency oscillation mode

Bitumen und bitumenhaltige Bindemittel - Bestimmung der Äquiviskositätstemperatur basierend auf niedriger Scherviskosität mit Hilfe eines dynamischen Scher-Rheometers in niederfrequentem Schwingungsmodus

Bitumes et liants bitumineux - Détermination de la température d'équiviscosité basée sur la mesure de la viscosité a faible taux de cisaillement utilisant un rhéometre a cisaillement dynamique (DSR) en mode oscillatoire a basse fréquence

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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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Bitumen and bituminous binders - Determination of equiviscous temperature based on Low Shear Viscosity using a Dynamic Shear Rheometer in low frequency oscillation mode

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Bitumen und bitumenhaltige Bindemittel - Bestimmung der Äquiviskositätstemperatur basierend auf Viskosität bei niedriger Schergeschwindigkeit mit Hilfe eines dynamischen Scher-Rheometers in niederfrequentem Schwingungsmodus

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Foreword

This document (CEN/TS 15324:2008) 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 document describes the determination of the EquiViscous Temperature (EVT) of bitumen or bituminous binder samples, based on a defined, practice related Low Shear Viscosity (LSV), using a Dynamic Shear Rheometer (DSR) in low frequency oscillation mode.

The EquiViscous Temperature (EVT) measured by this binder test is seen as a performance indicator for the partial contribution of the bituminous binder to the rutting resistance of the compacted asphalt mixture under service conditions at elevated pavement temperatures.

The test method described in this document is applicable to unaged, aged and recovered bituminous binders including Polymer Modified Binders (PMBs).

WARNING — 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 determine the applicability of regulatory limitations prior to use. Since this document 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 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 12594, *Bitumen and bituminous binders – Preparation of test samples*

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results – Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

3 Terms and definitions

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

3.1
complex shear modulus G*

$$G^* = \frac{\tau^*}{\gamma^*} = |G^*| e^{i\delta} = |G^*| \cos\delta + i|G^*| \sin\delta = G' + iG'' \quad (i^2=-1) \tag{1}$$

where

— G* is the complex shear modulus, expressed in Pascal (Pa);

— τ^* is the harmonic, sinusoidal shear stress, expressed in Pascal (Pa);

— γ^* is the harmonic, sinusoidal shear strain;

— $|G^*|$ is the norm of the complex shear modulus, ratio of peak stress to peak strain in harmonic, sinusoidal oscillation;

— δ is the phase angle of the complex shear modulus, shift between stress and strain in harmonic, sinusoidal oscillation;

— G' is the real part of the complex shear modulus (storage modulus);

— G" is the imaginary part of the complex shear modulus (loss modulus)

3.2
complex viscosity η*

$$\eta^* = \frac{\tau^*}{\dot{\gamma}^*} = \frac{G^*}{i\omega} = \frac{G''}{\omega} - i\frac{G'}{\omega} = \eta' - i\eta'' \quad (2)$$

where

— η* is the complex viscosity, expressed in Pascal. second (Pa.s);

— τ^* is the harmonic, sinusoidal shear stress, expressed in Pascal (Pa);

— $\dot{\gamma}^*$ is the harmonic, sinusoidal shear strain rate, expressed in second^{-1} (1/s);

— η' is the real part of the complex viscosity (dynamic viscosity);

— η" is the imaginary part of the complex viscosity;

— ω is the angular frequency, expressed in radian/second (rad/s).

$$\eta_{\dot{\gamma}} = \left|\eta^*\right| = \frac{\left|G^*\right|}{\omega} \quad (3)$$

where

$\eta_{\dot{\gamma}}$: viscosity (calculated from complex modulus, measured at low shear rate) in Pa.s;

– $\left|\eta^*\right|$: norm of complex viscosity, ratio of peak shear stress to peak shear rate in harmonic sinusoidal oscillation, in (Pa · s);

$$\omega\,(\text{rad/s}) = 6{,}28318\,f\,(\text{Hz}) \quad (4)$$

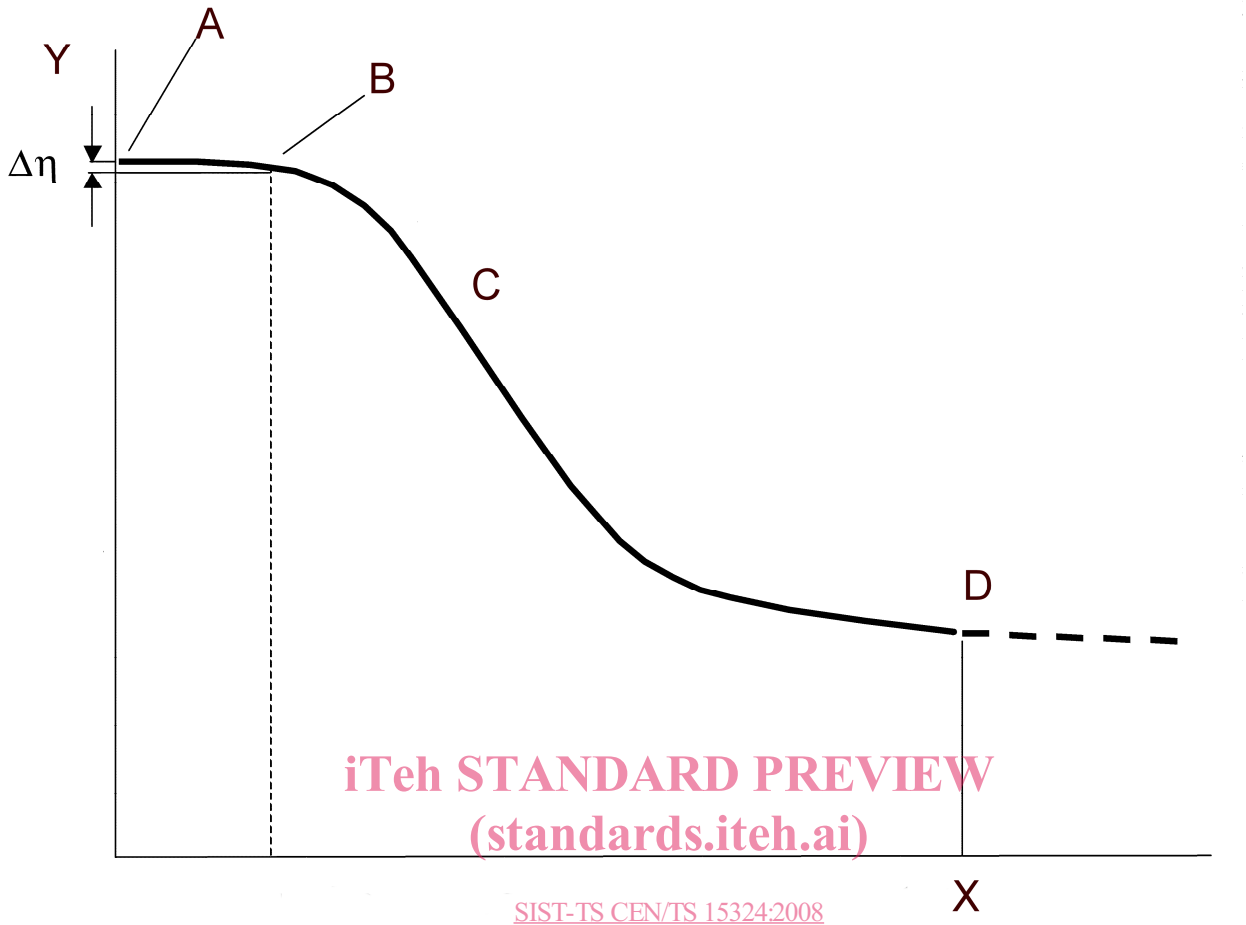
where

– f : frequency of sinusoidal oscillation, in Hz.

3.3
Zero Shear Viscosity (ZSV), η_0
dynamic viscosity of bitumen depends on the shear stress or shear strain rate level (i.e. non-Newtonian substance)

The shear thinning or pseudo-plastic behaviour is characterised by decreasing viscosity with increasing shear stress or shear rate between two well defined values: "Zero Shear Viscosity" η_0 at zero and "Limiting Viscosity" η_∞ at infinitely high shear stress or shear rate (Figure 1).

The dynamic viscosity in the intermediate domain between η_0 and η_∞ is called "Apparent Viscosity" because it depends on the (incident) loading conditions



Key

X log shear stress τ , or log shear rate $\dot{\gamma}$

Y log dynamic viscosity η

A Zero shear viscosity η_0 (ZSV)

B Low shear viscosity $\eta_{\dot{\gamma}_1}$ (LSV)

C apparent viscosity η

D limiting viscosity η_∞

Figure 1 — Definition of the Zero Shear Viscosity (ZSV), Low Shear Viscosity (LSV) and increase in viscosity $\Delta\eta$ with decreasing shear stress or shear rate

3.4

Low Shear Viscosity (LSV) $\eta_{\dot{\gamma}}$

dynamic viscosity at low shear stress or shear rate, where “low” means close to zero (see Figure 1). The concept of LSV is introduced because it can be measured directly: it is derived from the complex modulus measured with DSR in oscillation mode at low frequencies in combination with low strain amplitudes (see Equation (3)). As seen in Figure 1, LSV is smaller than ZSV:

$$\eta_0 = \eta_{\dot{\gamma}} + \Delta\eta \qquad (5)$$

The lower the shear stress or shear rate at which LSV is measured, the closer the value will approximate the ZSV

3.5

EquiViscous Temperature (EVT) related to Low Shear Viscosity (LSV)

temperature at which a bitumen sample exhibits a given LSV (e.g. 2 kPa·s), at a given shear stress or shear rate

4 Principle

4.1 General

The EquiViscous Temperature, based on a defined Low Shear Viscosity, indicates the rutting susceptibility of the binder. EVT can only be used for comparing or ranking binders when the EVT is based on the same LSV, measured at the same shear stress or shear rate.

The test is performed in two steps.

4.2 Test part 1: temperature sweep

A temperature sweep is carried out to determine the equiviscous temperature value EVT1 related to a defined Low Shear Viscosity $\eta_{\dot{\gamma}_1}$ (e.g. 2 kPa.s) at a low frequency (e.g. 0,01 Hz) and a low strain amplitude (e.g. 0,1). The procedure is described in sub-clause 7.1.

4.3 Test part 2: frequency sweep

A frequency sweep at test temperature EVT1 (determined in test part 1) is carried out from a higher frequency (e.g. 1 Hz) to a lower frequency (e.g. 0,0 003 Hz) with an additional extrapolation to e.g. 0,0001 Hz. This will reveal a higher value of LSV (and likewise a higher value ETV2 than ETV1), which is a closer approximation to ZSV.

The difference ΔT between EVT2 and EVT1 shall be calculated from the increase of LSV, as explained in sub-clause 7.2.

$$\Delta T\,(°C) = EVT2\,(°C) - EVT1\,(°C) \qquad (6)$$

For routine testing purposes, e.g. quality control, a significant simplification of the test procedure is possible by measuring only EVT1 by a temperature sweep at a low frequency in the magnitude of f = 0,01 Hz.

Particularly for PMBs, test part 2 can reveal a significant increase in EVT. Therefore, EVT2 shall be measured by carrying out a frequency sweep to lower frequency.

All measurements shall be carried out in the linear viscoelastic region (see Clause 7).

NOTE It is recommended to systematically apply the same values of LSV, shear strain and frequency, as recommended by the notes in Clause 7 of this standard. Only then will it be possible to use the EVT for ranking binders.

5 Apparatus

Usual laboratory apparatus and glassware, together with the following:

5.1 Dynamic shear rheometer (DSR), with either an integral temperature control system or temperature control attachments, capable of controlling the temperature over a minimum range of 5 °C to 85 °C with an accuracy of ± 0,1 °C throughout the test period. The rheometer shall be fitted with parallel plates, 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 be able to determine $|G^*|$ in the range of 10 Pa to 10 MPa (± 2 %).

For rheometers using an air bearing, and to avoid damage, the air supply to the bearing shall be switched on before the instrument is switched on. When not in use, the spindle shall be secured.

Make a visual check to ensure the two plates are vertically aligned. If there is any doubt as to the alignment, the manufacturer or a qualified technician shall re-align the plate geometry.

The rheometer and temperature control system shall be calibrated at regular time intervals.

NOTE 1 When liquid is used to immerse the test specimen, a water/glycol mixture has been found to be suitable.

NOTE 2 For bituminous binders it is recommended to use a plate diameter of 25 mm and a gap of 1 mm. Plates of different diameters and gaps between 0,5 and 2 mm can also be used, provided compliance effects of the instrument do not affect the results and the testing is done within the specified range of torque and angular deformation and within the linear region (see sub-clause 7.1, NOTE 5). This should be valid at any applied temperature and frequency.

NOTE 3 It is recommended that the rheometer and temperature control system are 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, such as a type P thermocouple. Also note that external devices read the accurate temperature value only if they are calibrated correctly.

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

5.3 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 rheometer and specimen

6.1 Rheometer set-up

Set up the rheometer in the sequence given in the manufacturer's instructions, including the procedure for selecting and setting the parallel plate geometry and gap. It is essential that the operational limits of stiffness for the selected geometry are determined (see also sub-clause 5.1, NOTE 2).

Select the appropriate oscillation package, if applicable, from the software menu.

Carefully prepare the rheometer plates 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.

6.2 Gap setting

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

The effective gap will be affected by the actual temperature in the geometry. If the DSR has no automatic gap compensation feature, the method of correcting gap changes for temperatures different from the gap setting temperature shall be reported.

NOTE 1 According to sub-clause 5.1, NOTE 2, a gap of 1 mm is recommended.

NOTE 2 If the DSR has an automatic gap compensation feature, the gap may be set at any temperature within the range covered. If the DSR has no gap compensation feature, it is recommended that the gap is set at a number of different mid-point temperatures not exceeding 15 °C intervals within the range tested.

6.3 Specimen preparation

Prepare the binder sample in accordance with EN 12594.

Two methods can be used for preparing the test specimens:

— specimen preparation in a mould (preferred method);

— directly pouring onto the test plate.

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. Discard any binder remaining in the vial. If preferred, weigh the required quantity of binder directly on to the approximate centre of the measuring geometry being used. Proceed to sub-clause 6.4.

If using moulds, pour sufficient binder into the mould. To avoid successive sample heating, several specimens may 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 any longer than the time needed to obtain proper stiffness. The recommended time is approximately 10 min and shall not exceed 30 min.

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

Attach the specimen to the clean, dry test plate.

6.4 Setting the gap and trimming the specimen

After the specimen has been loaded into the rheometer as described above, bring the rheometer to the selected gap setting + 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.

NOTE Good bonding of the specimen to the plates is a prerequisite for successful testing. The bonding depends on the temperature of the plates at the start of the test. If the test temperature is lower than 40 °C, it is necessary to briefly increase the temperature without exceeding the presumed softening point to ensure the binder adheres to the test plates.

7 Procedure

7.1 Test Part 1, temperature sweep: determination of EVT1

Set up the rheometer to test in the oscillatory mode (see NOTE 1). The temperature sweep shall run from lower temperature to higher temperature, monitoring at least two full cycles at each test temperature (see NOTE 2 and NOTE 3). Allow sufficient time to reach thermal equilibrium at each step (see NOTE 4). The applied strain has to be within the linear viscoelastic region (see NOTE 5).

For every temperature increment, calculate the Low Shear Viscosity $\eta_{\dot{\gamma}}$ according to Equation (3) for both cycles. Calculate the mean value from $\eta_{\dot{\gamma}}$ (cycle 1) and $\eta_{\dot{\gamma}}$ (cycle 2). Plot the mean values of $\eta_{\dot{\gamma}}$ versus the temperature, T in a log $\eta_{\dot{\gamma}}$ versus temperature plot (Figure A.1 and Figure A.3). Determine parameters a and b of Equation (7) by linear regression using all data points.

$$\log \eta_{\dot{\gamma}} = -a * T(°C) + b \qquad (7)$$