

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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National foreword

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This publication is not to be regarded as a British Standard.

It is being issued in the Draft for Development series of publications and is of a provisional nature. It should be applied on this provisional basis, so that information and experience of its practical application can be obtained.

Comments arising from the use of this Draft for Development are requested so that UK experience can be reported to the European organization responsible for its conversion to a European standard. A review of this publication will be initiated not later than three years after its publication by the European organization so that a decision can be taken on its status. Notification of the start of the review period will be made in an announcement in the appropriate issue of *Update Standards*.

According to the replies received by the end of the review period, the responsible BSI Committee will decide whether to support the conversion into a European Standard, to extend the life of the Technical Specification or to withdraw it. Comments should be sent to the Secretary of the responsible BSI Technical Committee at British Standards House, 389 Chiswick High Road, London W4 4AL.

The UK participation in its preparation was entrusted by Technical Committee B/510, Road materials, to Subcommittee B/510/19, Bitumen and related products.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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Amendments/corrigenda issued since publication

Date	Comments

English Version

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

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

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

This Technical Specification (CEN/TS) was approved by CEN on 23 March 2007 for provisional application.

The period of validity of this CEN/TS is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the CEN/TS can be converted into a European Standard.

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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^*

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

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).

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

where

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

$|\eta^*|$: 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.

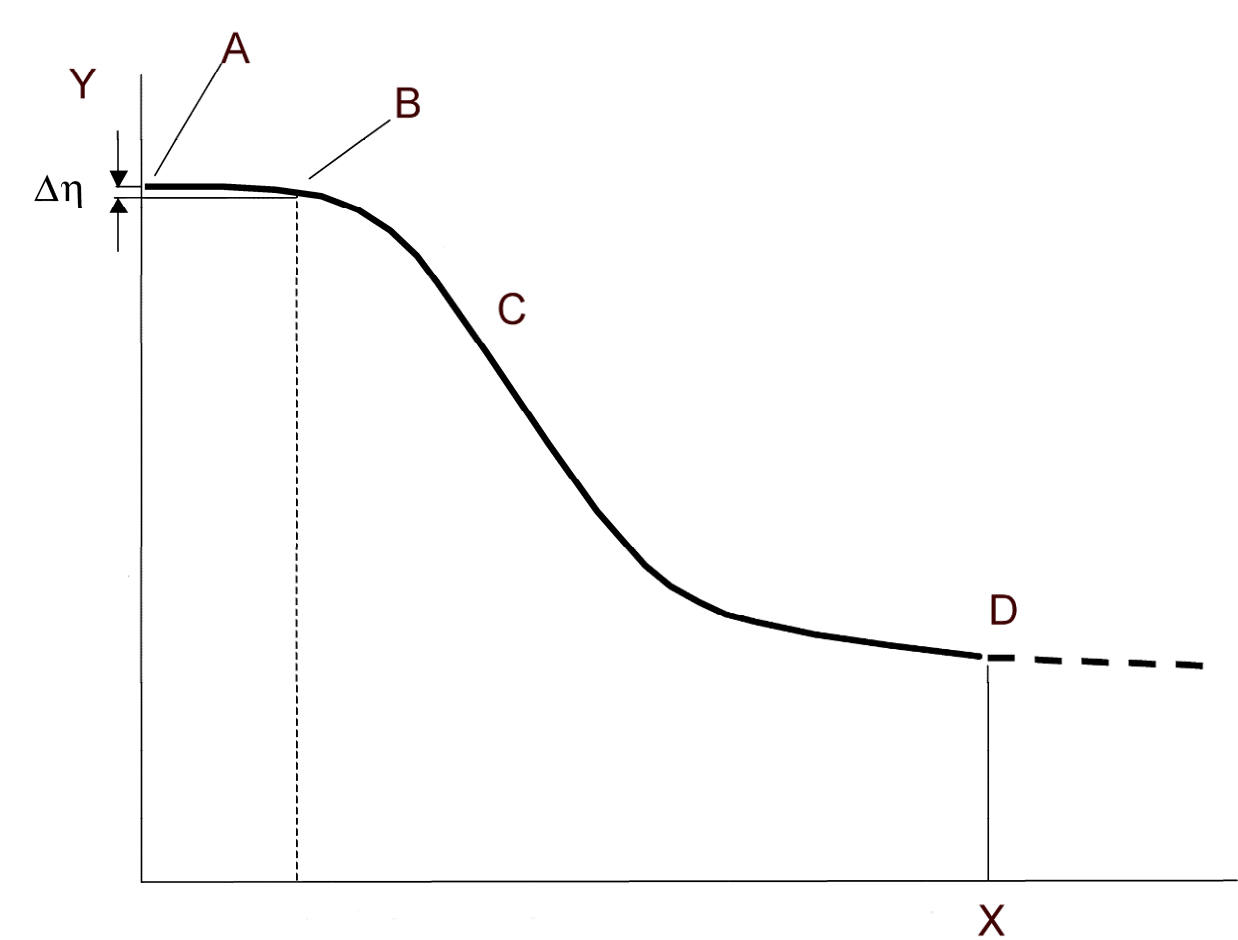
3.3

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 $\dot{\gamma}$ | A Zero shear viscosity η_0 (ZSV) |
| Y log dynamic viscosity η | 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:

$$\eta_0 = \eta_{\dot{\gamma}} + \Delta\eta \tag{5}$$

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. 0,0003 Hz) with an additional extrapolation to e.g. 0,0001 Hz. This will reveal a higher value of LSV (and likewise a higher value EVT2 than EVT1), 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.

$$\Delta T (^{\circ}\text{C}) = \text{EVT2} (^{\circ}\text{C}) - \text{EVT1} (^{\circ}\text{C}) \quad (6)$$

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 $\pm 0,1$ °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.