DRAFT FOR DEVELOPMENT

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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

ICS 91.100.50

BSI
British Standards

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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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.

CEN members are required to announce the existence of this CEN/TS in the same way as for an EN and to make the CEN/TS available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the CEN/TS) until the final decision about the possible conversion of the CEN/TS into an EN is reached.

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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*

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

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;

— $\left|G^*\right|$ 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);

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

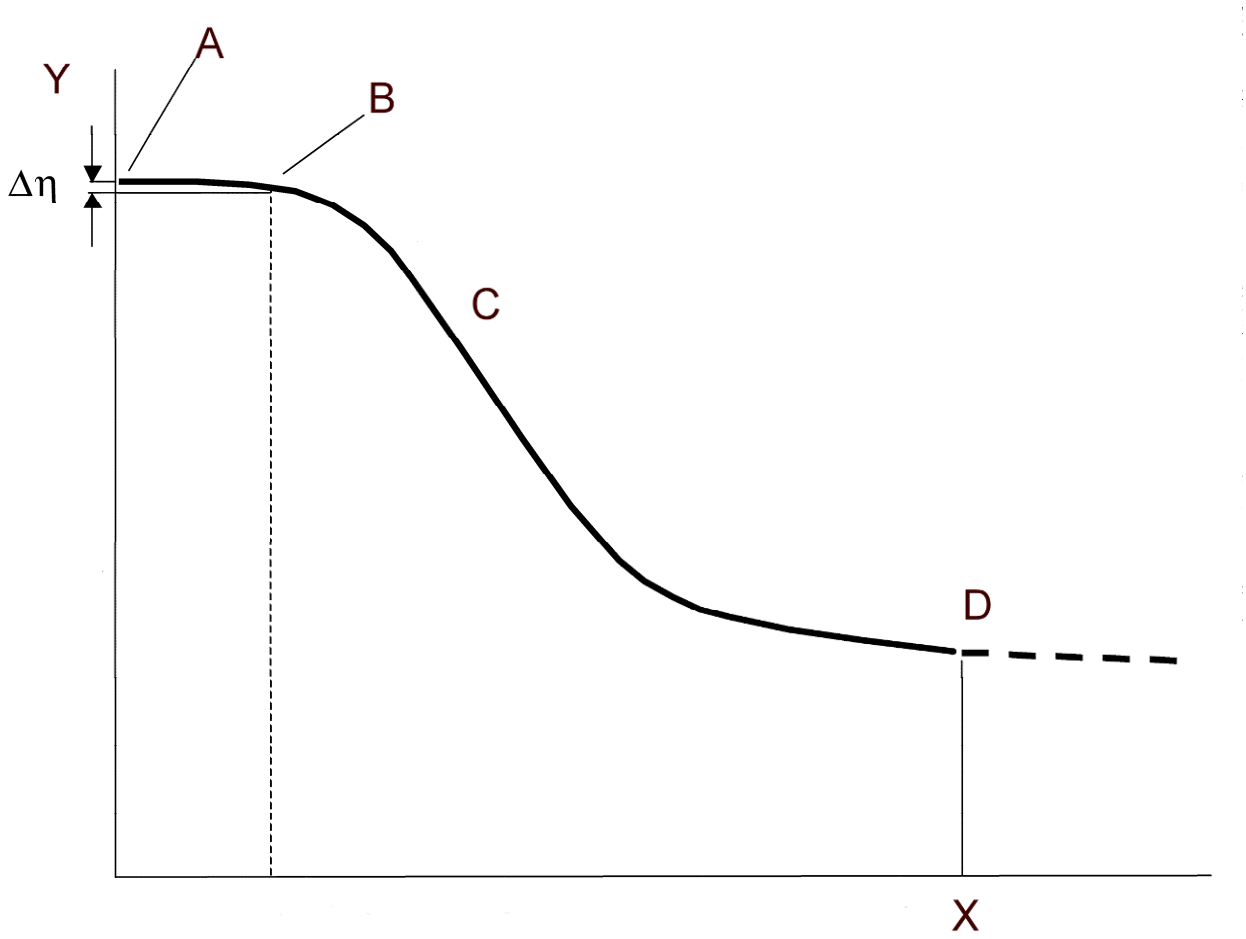
where

$-f$: 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.

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

where

a and b are positive constants.

Calculate the temperature EVT1, corresponding to a chosen (performance related) $\eta_{\dot{\gamma}_1}$ value (see NOTE 6):

$$EVT1 = \frac{\left(\log \eta_{\dot{\gamma}} - b\right)}{-a} \quad (8)$$

NOTE 1 It is recommended to do the temperature sweep at a frequency of 0,01 Hz and a strain amplitude of 0,1. This corresponds to a shear rate of 0,0 063 sec^{-1}.

NOTE 2 It is recommended to do a measurement at each temperature increment of 1°C

NOTE 3 It is recommended that the temperature sweep covers the viscosity range of 5 kPa·s to 0,8 kPa·s. For routine tests like quality control, this interval may be reduced.

NOTE 4 It is possible to specify a single thermal equilibration time that is valid for DSR's produced by different manufactures. The design (fluid bath or air oven) of the environmental control system and the starting temperature dictate the time required to reach the test temperature.

NOTE 5 Generally a strain amplitude of $\gamma \approx 0,1$ is within the linear viscoelastic region. However, for some PMBs, the linear domain may be less. Publications report that a correlation exist between the maximum strain value γ_{max} of the linear viscoelastic region and the value of the complex modulus at the considered temperature. Control the strain within 20 % of the target value calculated by Equation (9).

$$\gamma_{\max} = \frac{12,0}{\left(G^*\right)^{0,29}} \quad (9)$$

where

γ_{max} is the shear strain in percent (%);

G* is the complex shear modulus, expressed in kiloPascal (kPa).

If the nature of the test sample is unknown or the polymer concentration of a PMB sample exceeds a level that is typical for standard paving PMB, the region of the linear viscoelastic behaviour has to be experimentally determined. This can be done also in the DSR equipment by an amplitude sweep with an extra part of the sample material at a test temperature near EVT1. During the amplitude sweep the complex modulus has to be determined as a function of the applied strain. The horizontal part of the curve represents the linear viscoelastic region. The strain target should be a value not higher than two-third of the maximum strain value within the linear viscoelastic region.

NOTE 6 $\eta_{\dot{\gamma}_1}$ = 2,0 kPa·s is recommended. With the test conditions recommended in NOTE 1, the result is then the equiviscous temperature EVT1 for a LSV of 2,0 kPa·s at a shear rate of 0,0 063 sec^{-1}.

7.2 Test Part 2, frequency sweep: determination of EVT2 and ΔT

According to Equation (6),

$$EVT2\,(°C) = EVT1\,(°C) + \Delta\Delta\,(°C) \quad (10)$$

To find the temperature adjustment ΔT(°C), a frequency sweep shall be carried out at the test temperature EVT1 (°C) found in Test Part 1 (The temperature adjustment ΔT (°C) is demonstrated in Figure 1.).

Table 1 contains a typical frequency sequence which proved to be appropriate for the execution of the frequency sweep.

Table 1 — Typical frequency sequence

	1	2	3	4	5	6
Frequency f (Hz)	1	0,3	0,1	0,03	0,01	0,003

Set the temperature controller of the DSR to EVT1 as the test temperature. Initiate the testing immediately after reaching thermal equilibrium to minimise the effect of molecular association (steric hardening) that can cause an increase in modulus if the specimen is held at elevated temperature for a prolonged period of time.

Carry out the frequency sweep running from the higher frequency to the lower (see NOTE 1).

Calculate η according to Equation (3) for each frequency step.

Plot the results as η versus the logarithm of the frequency. Fit the data points with the following mathematical equation:

$$\eta = -c * \log(f) + d \tag{11}$$

Where:

c and *d* are positive constant parameters.

Extrapolate the curve and calculate the viscosity related to a frequency close to zero. Thus find $\eta_{\dot{\gamma}_2}$ (or LSV2), the LSV at a shear rate close to zero (see NOTE 2, Figure A .2 and Figure A.4).

Calculate ΔT by:

$$\Delta T = \frac{\Delta \log(\eta)}{-a} = \frac{\log \eta_{\dot{\gamma}_1} - \log \eta_{\dot{\gamma}_2}}{-a} = \frac{\log \frac{\eta_{\dot{\gamma}_1}}{\eta_{\dot{\gamma}_2}}}{-a} \tag{12}$$

Physically, ΔT is the increase in the EquiViscous Temperature that would be obtained, if the temperature sweep in test part 1 were carried out at a frequency of 0,0 001 Hz (instead of the recommended frequency of 0,01 Hz).

NOTE 1 A shear strain of 0,1 is recommended.

NOTE 2 It is recommended to do the extrapolation to a frequency of 0,0 001 Hz. With a shear strain of 0,1 as recommended in NOTE 1, $\eta_{\dot{\gamma}_2}$ (or LSV2) is the viscosity at a temperature EVT1 and a very low shear rate of 0,000 063 s^{-1}.

NOTE 3 In case $\eta_{\dot{\gamma}_2}$ (as the result of Test part 2) is lower than $\eta_{\dot{\gamma}_1}$ (equiviscosity used in Test part 1), an error or failure must have occurred (sample preparation, test procedure, data acquisition, calculation etc.).

8 Expression of results

8.1 General

Two results are considered valid if they do not differ by more than 1,0 °C of their mean.

8.2 Test part 1

Express the equiviscous temperature EVT1 in °C (to the nearest 0,1 °C) as the average of two valid determinations. Report the LSV1 (in Pa·s) to which this EVT1 is related, as well as the applied frequency and shear strain (see NOTE in sub-clause 4.3).

8.3 Test Part 2

Express the equiviscous temperature EVT2 in °C (to the nearest 0,1 °C) as the average of two valid determinations. Also express the temperature adjustment ΔT in °C (to the nearest 0,1 °C) as the average of two valid determinations. Report the frequency at which LSV2 is determined, as well as the shear strain.

9 Precision

Two Round Robin tests were carried out in 2003 in which 15 laboratories participated. Five binders were studied, including 2 pure bitumen and 3 PMBs. Two repeated tests were carried out for each binder and by each laboratory. This produced sufficient data to estimate the repeatability and reproducibility of the test method according to ISO 5725-2.

The results are summarized in Table 2 for EVT1 (Test Part 1) and in Table 3 for EVT2 (Test Part 2).

Table 2 — Repeatability and reproducibility standard deviation of EVT1 (at 2 000 Pa.s)

Binder	Bitumen A	Bitumen B	PMB 1	PMB 2	PMB 3
General mean (°C)	44,7	69,3	60,8	67,5	60,6
Repeatability standard deviation (°C)	0,7	0,7	0,7	1,0	0,6
Repeatability limit (°C)	1,9	2,0	2,0	2,8	1,6
Reproducibility standard deviation (°C)	0,7	1,0	1,5	1,6	1,9
Reproducibility limit (°C)	2,0	2,8	4,3	4,6	5,3

Table 3 — Repeatability and reproducibility standard deviation of EVT2

Binder	Bitumen A	Bitumen B	PMB 1	PMB 2	PMB 3
General mean (°C)	45,4	70,9	63,1	71,6	66,7
Repeatability standard deviation (°C)	0,6	0,8	0,7	0,7	0,7
Repeatability limit (°C)	1,7	2,2	1,9	2,0	2,1
Reproducibility standard deviation (°C)	1,0	1,8	2,2	2,5	2,3
Reproducibility limit (°C)	2,9	5,1	6,0	7,0	6,3

10 Test reports

The test report shall contain at least the following information.

For Test Part 1:

a) type of equipment;

b) type and complete identification of the sample under test;

c) reference to this European standard;

d) date of the test;

e) test plate diameter, to the nearest 0,1 mm and gap, to the nearest 1 μm;

f) target strain **γ**, to the nearest 0,001;

g) target frequency f, to the nearest 0,001 Hz;

h) chosen value $\eta_{\dot{\gamma}_1}$ (i.e. LSV1), to the nearest 1 Pa.s;

i) temperature EVT1, to the nearest 0,1 °C;

j) plots according to Figure A.1 and Figure A.3.

NOTE 1 For gaining experience with this new test method it is recommended to add the primary test results according to Table A.1 and Table A.3, including phase angle **δ** to the test reports.

If Test Part 2 has been carried out, the test report shall also contain the following information:

k) minimum and maximum frequency f of the frequency sweep, to the nearest 0,001 Hz;

l) target strain **γ**, to the nearest 0,001;

m) frequency at which $\eta_{\dot{\gamma}_2}$ (i.e. LSV2) is determined by extrapolation;

n) viscosity $\eta_{\dot{\gamma}_2}$ (i.e. LSV2), to the nearest 1 Pa.s;

o) temperature EVT2, to the nearest 0,1 °C;

p) plots according to Figure A.2 (and Figure A.4).

NOTE 2 For gaining experience with this new test method it is recommended to add the primary test results according to Table A.2 and Table A.4 including phase angle **δ** to the test reports.

For both test parts, the test report shall contain the following information:

q) any deviation, by agreement or otherwise, from the procedure specified in this standard.

Annex A
(informative)

Test examples

A.1 General

Two test examples are shown to demonstrate the procedure. Although valid according to the present standard, the test conditions used in these examples are not the values recommended by the standard.

A.2 Test example 1: bitumen 1 (paving grade bitumen)

A.2.1 General

Rheometer type: XYZ123

Test set-up: 25 mm parallel plates, 1mm gap.

A.2.2 Test Part 1: determination of EVT1 (for $\eta_{\dot{\gamma}}$ = 2,0 kPa.s)

Test conditions: f = 0,0159 Hz, γ = 0,1

A temperature sweep is performed from 30 °C up to 55 °C (see Table A.1).

Table A.1 — Temperature sweep at f = 0,0159 Hz, γ = 0,1

Temperature	| G* |	η
°C	Pa	Pa.s
30,0	3 074,6	30746,0
31,0	2 562,7	25 627,0
32,0	2 110,3	21 103,0
33,0	1 737,5	17 375,0
34,0	1 436,7	14 367,0
35,0	1 194,8	11 948,0
36,0	986,8	9 868,2
37,0	816,9	8 169,4
38,0	681,2	6 812,4
39,0	566,3	5 663,2
40,0	473,3	4 733,3
41,0	392,3	3 922,9
42,0	327,5	3 275,2
43,0	273,4	2 734,3
44,0	228,1	2 281,4
45,0	190,3	1 902,5
46,0	161,4	1 614,0
47,0	136,0	1 359,6
48,0	112,8	1 127,6
49,0	96,7	966,7
50,0	82,0	819,6
51,0	69,3	692,6
52,0	58,2	581,8
53,0	49,7	497,1
54,0	42,2	421,9
55,0	36,6	366,5

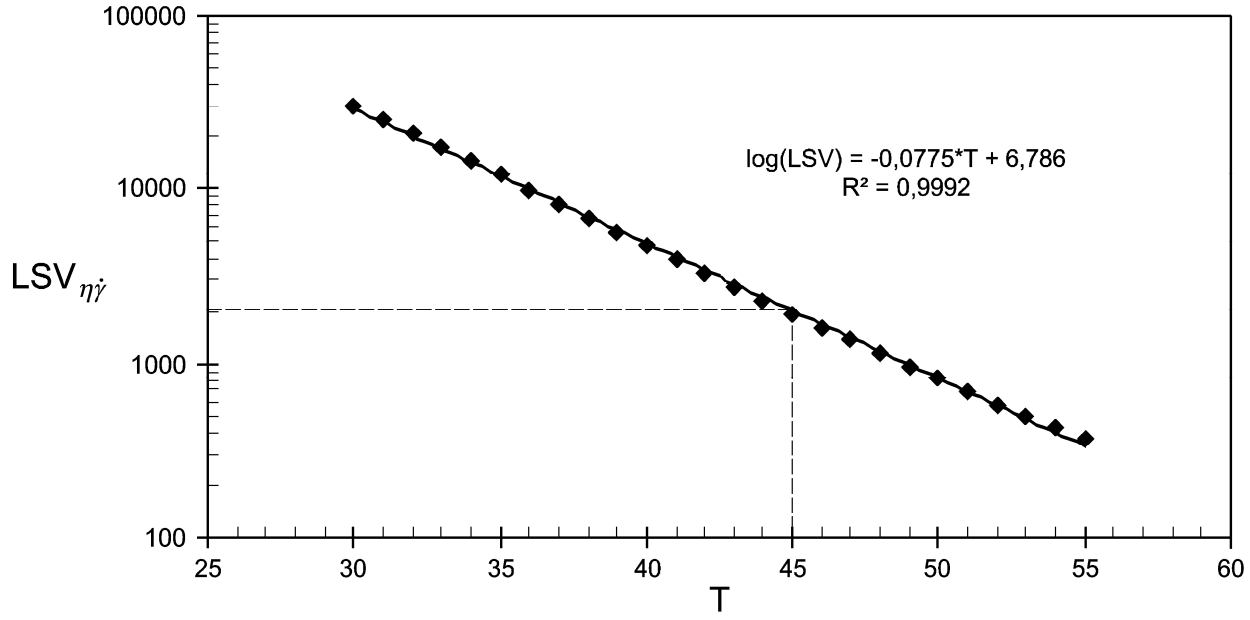
Figure A.1 shows the graph of $\log(\eta_{\dot{\gamma}})$ versus temperature, fitted by linear regression (see Equation 7):

$$\log \eta_{\dot{\gamma}} = -a * T + b = -0{,}0775.T + 6{,}786 \quad \text{(A.1)}$$

EVT1, corresponding to 2,0 kPa.s, is calculated as follows (see Equation 8):

$$EVT1 = \frac{\left(\log(2.10^{3}) - 6{,}786\right)}{-0{,}0775} = 45{,}0°C \quad \text{(A.2)}$$

NOTE EVT1 is determined as the average result of at least two temperature sweeps. For brevity, only one temperature sweep is shown in this example.



Key

X Temperature, °C

Y Low shear viscosity, Pa.s

Figure A.1 — Temperature sweep for determination of EVT1 (for $\eta_{\dot{\gamma}}$ = 2,0 kPa.s)

A.2.3 Test Part 2: determination of ΔT and EVT2

Test conditions: test temperature = EVT1 = 45 °C, γ = 0,1

A frequency sweep is performed from 1,596 Hz down to 0,008 Hz. The data are given in Table A.2.

Table A.2 — Frequency sweep at EVT1 = 45 °C and γ = 0,1

f	|G*|	η
Hz	Pa	Pa.s
1,596	16 481	1648,1
0,796	8 890,6	1 778,12
0,159	1 955,8	1 955,8
0,080	1 014,2	2 028,4
0,016	208,65	2 086,5
0,008	104,51	2 090,2

Figure A.2 shows the graph of $\eta_{\dot{\gamma}}$ versus log(f), fitted by linear regression (see Equation 11):

$$\eta_{\dot{\gamma}} = -c * \log(f) + d = -188{,}95 \times \log(f) + 1751{,}9 \quad \text{(A.3)}$$

Using this equation, $\eta_{\dot{\gamma}2}$ and $\eta_{\dot{\gamma}1}$ are calculated:

$$\eta_{\dot{\gamma}_2} = \eta_{\dot{\gamma}} \text{ at } 0{,}0001 \text{ Hz} = 2\,508 \text{ Pa.s} \quad \text{(A.4)}$$

$$\eta_{\dot{\gamma}_1} = \eta_{\dot{\gamma}} \text{ at } 0{,}01 \text{ Hz} = 2\,130 \text{ Pa.s} \quad \text{(A.5)}$$

ΔT is calculated using Equation (12):

$$\Delta T = \frac{\log \frac{2130}{2508}}{-0{,}0775} \quad \text{(A.6)}$$

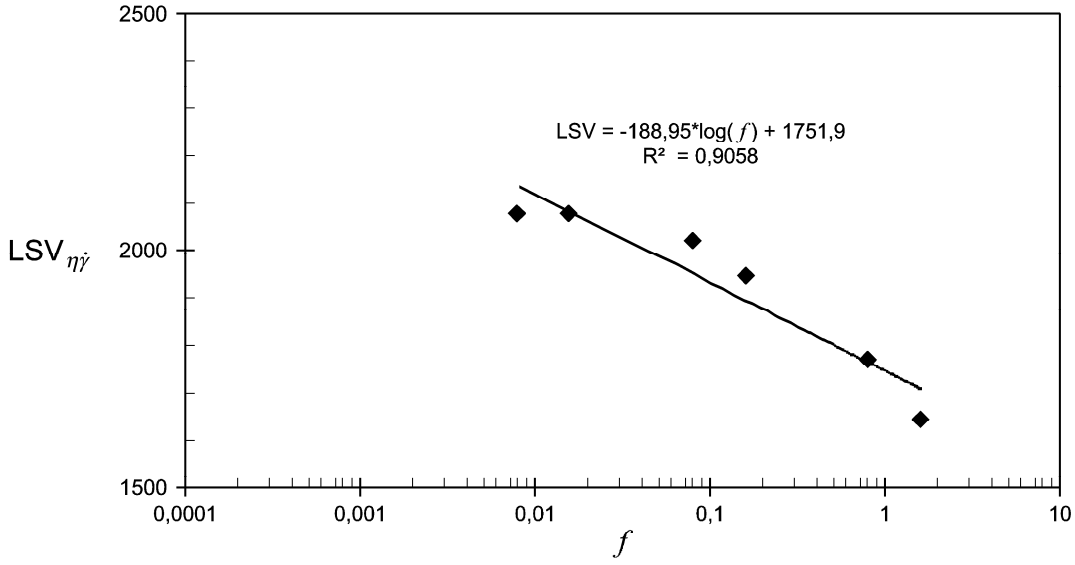
where

-0,0 775 is the slope of the log($\eta_{\dot{\gamma}}$) versus temperature curve (see Figure A.1).

For this example, ΔT= 0,9 °C

Finally, EVT2 is calculated according to Equation 10, and expressed with one decimal digit:

$$EVT2 = EVT1 + \Delta T = 45{,}9\ ^{\circ}C \quad \text{(A.7)}$$



Key

X Radial frequency, (rad/sec) Hz

Y Low shear viscosity, Pa.s

Figure A.2 — Frequency sweep for extrapolation to f=0,0 001 Hz (≈ZSV)

NOTE ΔT is determined as the average result of at least two frequency sweeps. For brevity, only one frequency sweep is shown in this example.

A.3 Test example 2: bitumen (PMB)

A.3.1 General

Rheometer type: XYZ123

Test set-up: 25 mm parallel plates, 1mm gap.

A.3.2 Test Part 1: Determination of EVT1 (for $\eta_{\dot{\gamma}}$ = 2,0 kPa·s)

Test conditions: f = 0,0159 Hz, γ = 0,1

A temperature sweep is performed from 55 °C up to 80 °C (see Table A.3).

Table A.3 — Temperature sweep at f = 0,0159 Hz, γ = 0,1

Temperature	G*	η
°C	Pa	Pa.s
55,0	974,4	9 793,8
56,0	873,9	8 739,4
57,0	756,4	7 564,1
58,0	664,2	6 641,6
59,0	586,2	5 862,3
60,0	512,5	5 124,9
61,0	447,3	4 472,5
62,0	393,0	3 929,9
63,0	344,7	3 447,1
64,0	301,2	3 012,1
65,0	265,1	2 651,4
66,0	232,4	2 323,6
67,0	203,6	2 036,1
68,0	178,7	1 786,8
69,0	157,1	1 570,5
70,0	138,1	1 380,7
71,0	122,6	1 225,6
72,0	108,3	1 083,4
73,0	95,2	952,3
74,0	84,6	845,8
75,0	74,9	749,5
76,0	66,3	663,2
77,0	58,6	586,1
78,0	51,9	519,4
79,0	45,9	459,0
80,0	40,6	405,7

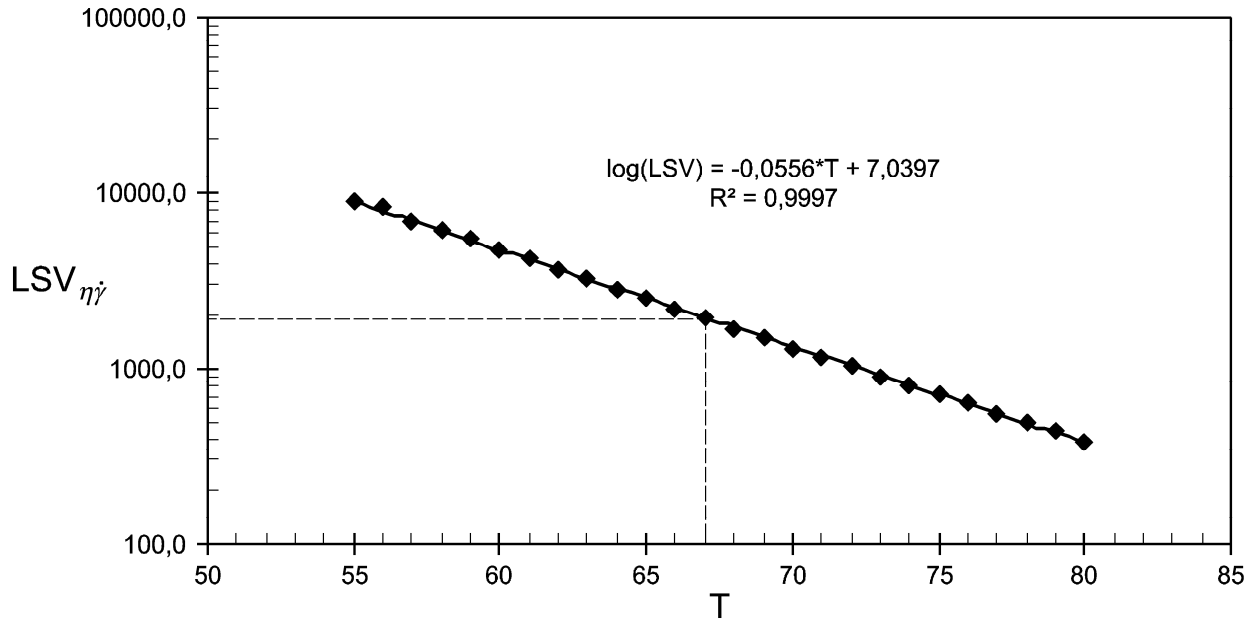
Figure A.3 shows the graph of $\log(\eta_{\dot{\gamma}})$ versus temperature, fitted by linear regression (see Equation 7):

$$\log \eta_{\dot{\gamma}} = -a * T + b = -0{,}0556.T + 7{,}0397 \quad \text{(A.8)}$$

EVT1, corresponding to 2,0 kPa,s, is calculated as follows (see Equation 8):

$$EVT1 = \frac{\left(\log(2.10^{3}) - 7{,}0397\right)}{-0{,}0556} = 67{,}2°C \quad \text{(A.9)}$$

NOTE EVT1 is determined as the average result of at least two temperature sweeps. For brevity, only one temperature sweep is shown in this example.



Key

X Temperature, °C

Y low shear viscosity, Pa.s

Figure A.3 — Temperature sweep for determination of EVT1 (for $\eta_{\dot{\gamma}}$ =2,0 kPa.s)

A.3.3 Test Part 2: determination of ΔT and EVT2

Test conditions: test temperature = 70 °C, γ = 0,1

NOTE 1 This example was taken from a Round Robin test where the test temperature was prescribed at 70 °C. According to the present standard, the test temperature should have been 67,2 °C. However, this will not have a large effect on ΔT and the principal aim of this example is to demonstrate the calculations.

A frequency sweep is performed from 1,59 Hz down to 0,008 Hz. The data are given in Table A.4.

Table A.4 — Frequency sweep at EVT1= 70 °C and γ = 0,1

f	| G* |	η
Hz	Pa	Pa.s
1,596	4 453,9	444,2
0,424	1 634,2	613,8
0,113	5 86,22	828,8
0,030	209,42	1 113,7
0,008	74,359	1 488,6

NOTE 2 The viscosity at 0,01 Hz is lower than 2 000 Pa,s, since this frequency sweep was performed at a test temperature higher than EVT1, which was 67,2 °C.

Figure A.4 shows the graph of $\eta_{\dot{\gamma}}$ versus log (f), fitted by linear regression (see Equation 11):

$$\eta_{\dot{\gamma}} = -c * \log(f) + d = -449{,}69 * \log(f) + 471{,}32 \quad \text{(A.10)}$$

Using this equation, $\eta_{\dot{\gamma}2}$ and $\eta_{\dot{\gamma}1}$ are calculated

$\eta_{\dot{\gamma}2}$ = $\eta_{\dot{\gamma}}$ at 0,0001 Hz= 2270 Pa.s (A.11)

$\eta_{\dot{\gamma}1}$ = $\eta_{\dot{\gamma}}$ at 0,01 Hz= 1371 Pa.s (A.12)

ΔT is calculated using Equation (12):

$$\Delta T = \frac{\log \frac{1371}{2270}}{-0{,}0556} \quad \text{(A.13)}$$

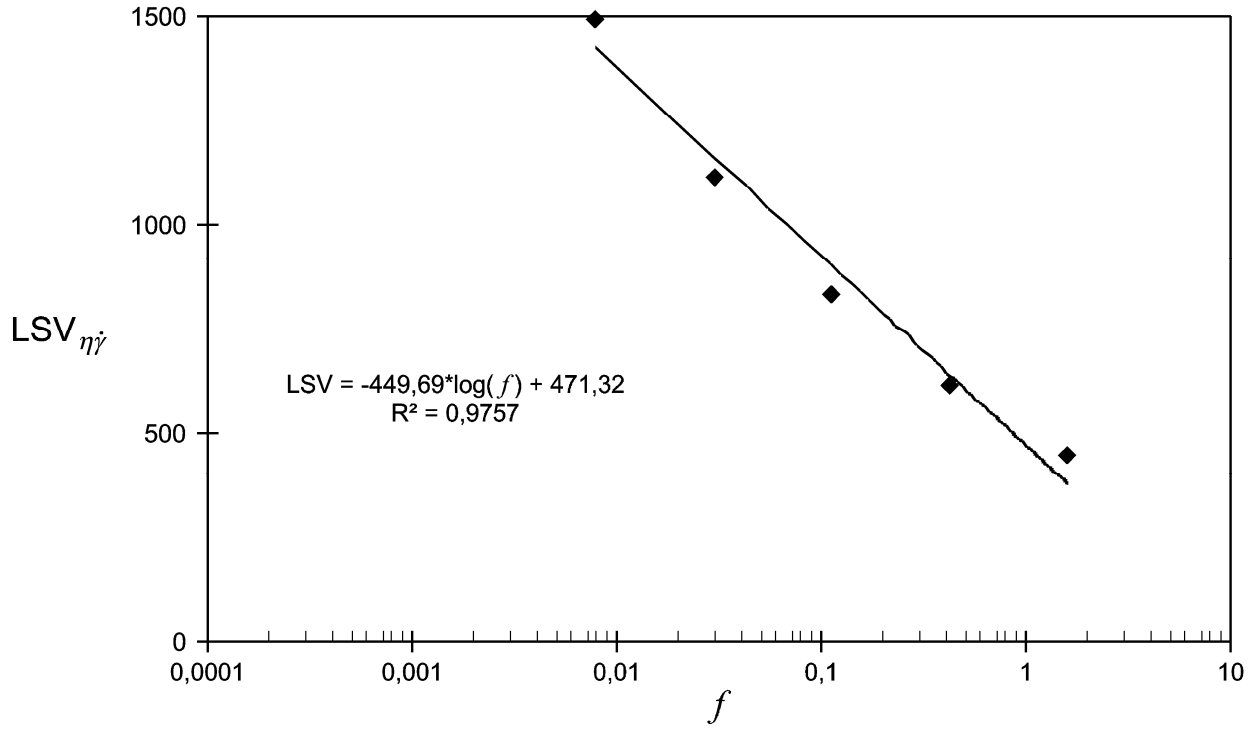
where

-0,0556 is the slope of the log($\eta_{\dot{\gamma}}$) versus temperature curve (see Figure A.3).

For this example, ΔT= 3,9 °C

Finally, EVT2 is calculated according to Equation 10, and expressed with one decimal digit:

$$EVT2 = EVT1 + \Delta T = 71{,}1\ ^{\circ}C \quad \text{(A.14)}$$



Key

X Radial frequency, (rad/sec), Hz

Y low viscosity, Pa.s

Figure A.4 — Frequency sweep for extrapolation to f=0,0 001 Hz (≈ZSV)

NOTE 3 ΔT is determined as the average result of at least two frequency sweeps. For brevity, only one frequency sweep is shown in this example.

Annex B
(informative)

Temperature verification procedure

Thermal gradients within the rheometer and the difficulty of calibrating the DSR temperature instrument while it is mounted in the rheometer require a temperature verification of the DSR temperature transducer. For that purpose, temperature measurements obtained from a dummy specimen with a calibrated thermal detector (to an accuracy of ± 0,02 °C) and the DSR temperature transducer can be compared. A dummy specimen of bituminous binder or silicone wafer may be used.

NOTE Alternatively use may be made of specially designed thermometers that can be inserted between the plates to verify the temperature.

Prepare the dummy specimen or use the silicon wafer following standard procedures. Use the dummy specimen only for temperature verification measurements (DSR-measurements are not valid if a temperature detector is inserted into the asphalt binder). Adjust the temperature in the chamber to the minimum temperature that will be used for testing and allow the chamber to come to thermal equilibrium. Read the DSR-temperature and the temperature of the dummy specimen. Increase the temperature in increments of not more than 6 °C and repeat the measurements to cover the range of test temperatures. The difference between the temperature probe and the temperature indicated by the DSR-transducer varies with temperature and depends on testing geometry.

Apply an appropriate temperature correction to the temperature measurement indicated by the DSR transducer if both readings do not agree within ± 0,1 °C.

Bibliography

[1] EN 58, *Bitumen and bituminous binders – Sampling bituminous binders*

[2] EN 1425, *Bitumen and bituminous binders – Characterization of perceptible properties*

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