DRAFT FOR DEVELOPMENT

DD CEN/TS 15325:2008

Bitumen and bituminous binders — Determination of Zero-Shear Viscosity (ZSV) using a Shear Stress Rheometer in creep mode

ICS 91.100.50

British Standards

National foreword

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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.

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English Version

Bitumen and bituminous binders - Determination of Zero-Shear Viscosity (ZSV) using a Shear Stress Rheometer in creep mode

Bitumes et liants bitumineux - Détermination de la viscosité à taux de cisaillement nul (ZSV) utilisant un rhéomètre à contrainte de cisaillement en mode de fluage

Bitumen und bitumenhaltige Bindemittel - Bestimmung der Null-Scherviskosität (ZSV) mit Hilfe eines Schubspannungs-Rheometers im Kriechmodus

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

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Foreword

This document (CEN/TS 15325:2008) 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 European standard describes the determination of Zero Shear Viscosity (ZSV), η_o, for bitumens and bituminous binders, preferably using test temperature domains in which 100 Pa.s < η_o < 50 000 Pa.s. The preferred test temperature is 60 °C but other temperatures for example, 45 °C or 50 °C could be used.

Under these conditions, ZSV (also referred to as the first Newtonian viscosity or absolute viscosity) is a suitable indicator to evaluate the partial contribution of the bituminous binder (including Polymer Modified Binders) to the rutting resistance of asphalt pavement layers.

This European standard describes the determination of ZSV using a Shear Stress Rheometer (SSR) in creep mode.

This method is applicable to unaged, aged and recovered bituminous binders including Polymer Modified Binders (PMBs).

WARNING — Use of this European standard can involve hazardous materials, operations and equipment. This European standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this European standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Since this European standard 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 European standards are indispensable for the application of this European standard. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced European standard (including any amendments) applies.

EN 12594, *Bitumen and bituminous binders — Preparation of test samples*

3 Terms and definitions

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

3.1
creep test
rheological test in which constant stress is applied to a sample and the resulting deformation is then measured as a function of loading time

3.2
steady-state flow
state at which the rate of deformation reaches a constant value

3.3
steady-state viscosity (SSV)
ratio of the applied stress to the deformation rate under steady-state flow, in Pascal.seconds (Pa.s)

3.4
zero-shear viscosity (ZSV) or Newtonian dynamic viscosity (η_0)
constant value to which the SSV tends at low shear stress or shear rate values, measured in Pascal.seconds (Pa.s)

3.5
compliance (J)
ratio of a component of strain to a component of stress, in Pascal $^{-1}$ (Pa^{-1})

4 Apparatus

4.1 Small tools

- Spatula or any tool that can be used to trim the sample.
- Moulds and vials, for preparing the test specimens. The moulds, where used, shall be silicone or similar material which does not adhere to the test specimen. Vials, where used, shall be glass with a nominal capacity of 10 ml.

4.2 Oven, ventilated laboratory model, capable of being controlled between 50 °C and 200 °C with an accuracy of ± 5 °C.

4.3 Controlled-stress rheometer with temperature control

- rheometer that applies a constant stress to the specimen. It provides torsional or direct shear stress, depending on the specimen shape.

NOTE 1 Usually a microcomputer is connected to the rheometer for data acquisition and processing.

- temperature controller capable of maintaining a temperature in and around the specimen within ± 0,1 °C throughout the test period. The test temperature range depends on the controller and needs to include the operating temperature, generally 60 °C.

The rheometer and temperature control system shall be calibrated at regular intervals in accordance with the quality assurance procedure of the laboratory.

NOTE 2 A suitable method is that the rheometer and temperature control system is 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. Also note that external devices read the accurate temperature value only if they are calibrated correctly. A temperature verification procedure is described in Annex A.

NOTE 3 The temperature in the test sample may differ from the temperature read by the device if insufficient equilibration time is used.

4.4 Appropriate testing geometries

The parallel plate geometries are appropriate for all bituminous binders, with preferably a 25 mm diameter and with a 1 mm gap.

NOTE 1 Plates of different diameters and gaps between 0,5 mm and 2 mm can also be used, provided compliance effects of the instrument do not affect the results and the testing is carried out within the specified range of torque and angular deformation and within the linear region.

NOTE 2 Use of parallel plate geometry improves the precision of test method.

In any case, for Polymer Modified Bitumens, the smallest dimension of the sample geometry, d, must satisfy the following:

d > three times the size of the largest polymeric inclusions (usually, d $\geq$ 1 mm).

NOTE The selection of system geometry may affect the accuracy of results. The manufacturer may have determined the operational limits and this information may be available but, if not, it can be determined by running a test specimen over a range of test temperatures using all the test geometries likely to be used in practice.

4.5 Zero-gap setting

Carefully prepare the rheometer plates to receive the test specimen 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.

Set the geometry temperature to the required test temperature and wait for thermal equilibrium (10 min to 30 min).

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

5 Specimen preparation

5.1 Test specimen preparation

Prepare the binder in accordance with EN 12594.

Two methods can be used:

— specimen preparation in a mould followed by attachment to a test plate (preferred method);

— loading directly into the plate gap.

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 (proceed to sub-clause 5.2).

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

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

Attach the specimens to the clean, dry test plate.

5.2 Setting the gap and trimming the sample

After the specimen has been placed on one of the test plates as described above, bring the test specimen to the selected gap setting plus 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.

6 Test execution

6.1 General

The experiment is conducted in two steps.

Step 1 is an optional stress sweep performed in conditions close to steady-state to gain some knowledge on the binder. In case of unknown binders, this step will shed some light on the non-Newtonian character of the binder and allow the proper stress selection for Step 2. In case of bitumen quality control (with a well-known binder), this step may be skipped.

In Step 2, the Zero Shear Viscosity value is determined at a given temperature and at a single (low) stress value. In no case, should Step 1 replace Step 2 in the determination of the ZSV because it is possible that not enough time has been spent in Step 1 to reach a steady state flow.

6.2 Step 1: stress sweep

The following sequence is applied to the same sample, without interruption between the consecutive creep tests:

Table 1 — Sequence applied with the same sample

Stress, Pa σ	**Time, min** t	**Accumulated time,** min. t'	**Viscosity, Pa.s** η
10	100	100	Computed during last 20 min
20	50	150	Computed during last 10 min
50	20	170	Computed during last 4 min
100	10	180	Computed during last 2 min
200	5	185	Computed during last 1 min
500	2	187	Computed during last 24 s
1000	1	188	Computed during last 12 s

NOTE The choice of these seven creep times has been selected to minimise the duration of the stress sweep. It is based on experience but does not guarantee a steady state flow. In other words, it is not certain that the measured properties are steady state viscosities (SSV). The obtained flow curve may thus only be an approximation of the real one.

The computed viscosity values can be plotted versus stress as shown for example in Figure 1 to Figure 3.

Figure 1 shows the flow curve of conventional 10/20 bitumen. The viscosity is almost independent of the applied shear stress. The binder presents a Newtonian character.

Figure 2 shows the flow curve of slightly modified bitumen (< 4 % of elastomer). The binder presents a higher non-Newtonian character when the viscosity plateaus at stresses below 50 Pa.

Figure 3 shows the flow curve of heavily modified bitumen (~ 7 % of polymer). The binder presents a non-Newtonian character and even the existence of the Newtonian plateau is not clear.

6.3 Step 2: creep test at a given stress level

The critical point here is to select the right stress level for the creep test. This is generally below 5 000 Pa for conventional, oxidised and special bitumens, and between 10 Pa and 50 Pa for non-fluxed polymer-modified bitumens. To remain within the linear viscoelastic range of the specimen, the stress must be kept sufficiently low.

Especially for very viscous binders, the stress selection depends on the shear rate experienced by the sample. The shear rate, noted dγ/dt, is defined as follows:

$$d\gamma / dt = \sigma / \eta \qquad (1)$$

If the strain rate is too low, the equipment limits can be reached and measurements become inaccurate. Check whether the operational limits of your rheometer have not been exceeded.

If the strain rate is too high, the conditions of “zero shear” are not achieved and the integrity of the sample can be threatened.

The data shown on Figure 1, Figure 2 and Figure 3 have also been presented (Figure 4) in a plot of viscosity versus shear rate. It can be seen that when the viscosity becomes large, the shear rate drops to very low values (of the order of 10^{-5} s^{-1} in the case of the highly modified bitumen).

In the case of a non modified binder, the flow curve is likely to be Newtonian as in Figure 1. Here, the ZSV may be measured at any stress between 10 Pa and 100 Pa. The advised (reference) stress level is 50 Pa.

In the case of a polymer modified binder, the selection of the right shear stress may be delicate:

- stress value should be chosen within the Newtonian plateau;
- shear rate should preferably be above 10^{-4} s^{-1} (see Figure 4).

For heavily modified binders, it may be impossible to obey the combination of these two constraints. In the particular example of Figure 3, the Newtonian plateau is not clearly visible even at stresses as low as 10 Pa where the shear rate gets close to 10^{-5} s^{-1}. Instead of decreasing the stress level more and more (with the risk of increasing scatter), the reference stress of 50 Pa may be selected but it must be mentioned that the measurement was not performed in the Newtonian plateau.

An alternative is to increase the temperature to reduce the non-Newtonian character of the binder. It must be noted that the reference temperature, T for the ZSV method is 60 °C and that it is necessary to compare different binders at the same temperature.

The creep times have been chosen at 1 h and 4 h for respectively non-modified and polymer modified binders. These creep times are generally sufficiently long to guarantee a steady state flow. If after the recommended creep time, the instantaneous viscosity still increases by more than 5 % in 15 min, the creep time may be extended to a maximum of 8 h.

Table 2 — Recommended values of the test conditions

Type of binder	Stress σ, Pa	Time t, h	Temperature T, °C
Non modified	50	1	60
Polymer modified	10 to 50	4	60

Perform the creep test with the selected parameters (σ, t, T).

Compute the average viscosity over the last 15 min. This is obtained by dividing the change in time (Δt) by the change in compliance (ΔJ) over the last 15 min (900 s) of the test. This can be expressed in mathematical terms as follows:

$$\eta_i = \Delta t / \Delta J = 900 / \left(J_{end} - J_{15\,\min - before - end}\right) \quad (2)$$

Repeat the measurement (each time with a new sample).

Perform a third measurement (with a new sample) if the difference between the first two results exceeds 5 % of their average value.

7 Expression of results

The ZSV value is the average of the two (or three) individual values determined in Step 2. Express the ZSV value to three significant figures.

8 Precision

Precision was determined in a Round Robin exercise including nine laboratories. Five binders were tested: bitumens A and B are conventional grades. PMB 1 and 2 are lightly modified binders. PMB 3 is a highly modified binder (~ 7% of polymer). The creep tests were repeated two or three times as requested in Section 6.3. The standard deviations for repeatability and reproducibility as well as the coefficient of variation (defined as the standard deviation divided by the average value of the population) of the ZSV values were measured. In the statistics in Table 2 below, all data were taken into account, as the Grubbs test did not detect outliers.

Table 3 — Repeatability and reproducibility data of the ZSV by creep method

Binders	**Bitumen** A	**Bitumen** B	**PMB 1**	**PMB 2**	**PMB 3**
General mean (Pa.s)	190	10 481	3 355	11 908	904 788
Repeatability, standard deviation (Pa.s)	10	1 222	203	912	331 448
Repeatability limits, (Pa.s)	27,7	3 387	563	2 528	918 727
Reproducibility, standard deviation (Pa.s)	29	1 823	413	2 057	826 965
Reproducibility limits, (Pa.s)	80	5 053	1 145	5 702	2 292 230

PMB 3 should be excluded from the discussion concerning repeatability and reproducibility as its general mean ZSV value is clearly outside the preferred ZSV range (100 Pa.s to 50 000 Pa.s).

For the four other grades (conventional and lightly modified binders), the repeatability coefficients of variations are typically of the order of 8 % and the reproducibility coefficients of variations are about twice as much (15,5 % in average). Similar values are obtained for the SSV (i.e. the apparent viscosities obtained during Step 1).

NOTE For ZSV determination of a PMB, the ZSV is very sensitive to the higher molecular weight fraction and to the polymer concentration. For this reason, it is important for the polymer concentration of the specimen to be equal to the polymer concentration of the bitumen sample whose ZSV must be determined. Moreover, in parallel plate geometry configurations, the sample sheared between the plates experiences a non-uniform shear strain. For a PMB, a wrong viscosity value will be obtained if the polymer is not uniformly distributed in the specimen. In other words, the polymer concentration and distribution in the specimen severely affect the ZSV value. Thus the poor repeatability and reproducibility of PMB3 reflect large variations in specimen state. Therefore, it is stressed that the utmost care should be taken for sample preparation.

9 Test report

The test report shall include at least the following information:

a) type and identification of the sample being tested;

b) reference to the present testing procedure;

c) apparatus type (trademark and model);

d) test geometry used (including gap);

e) test temperature;

f) applied stress;

g) number of measurements (minimum two, at the same stress level and at a given temperature);

h) creep time;

i) individual ZSV values, noted η_i, expressed in Pa.s;

j) ZSV, noted η_0 and expressed in Pa.s, average of at least two determinations;

k) eventually, the flow curve (see Step 1 in clause 6.2);

l) any deviation from the procedure described;

m) test date;

n) operator's name.

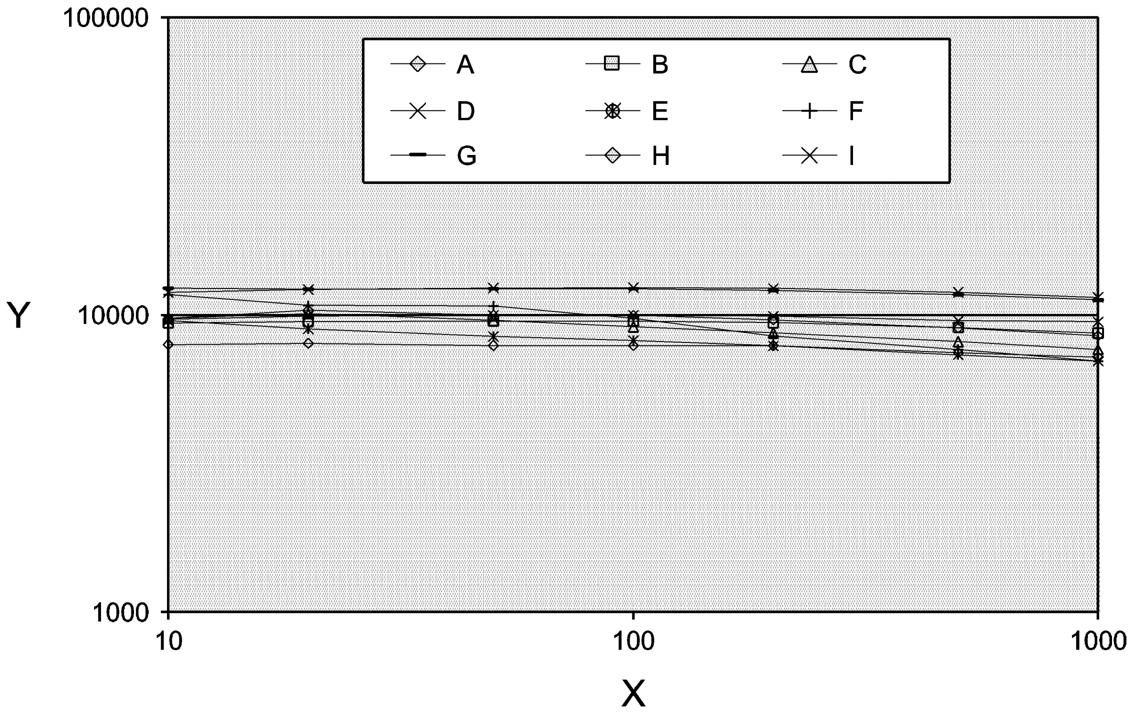
This information may be summarised in two tables. Examples are given below:

Table 4 — Example of information to be given concerning flow curve (optional)

Stress, σ **Pa**	**Time, t** min	**Viscosity, η** Pa.s	**Shear rate, $d\gamma/dt$** s^{-1}
10	100		
20	50		
50	20		
100	10		
200	5		
500	2		
1 000	1		

Table 5 — Example of information to be given concerning creep flow (optional)

Temperature, T °C	Stress, σ Pa	Number of measurements	Individual results, η_i Pa.s	Creep time, t h	ZSV, η_0 Pa.s
60,0	50,0				



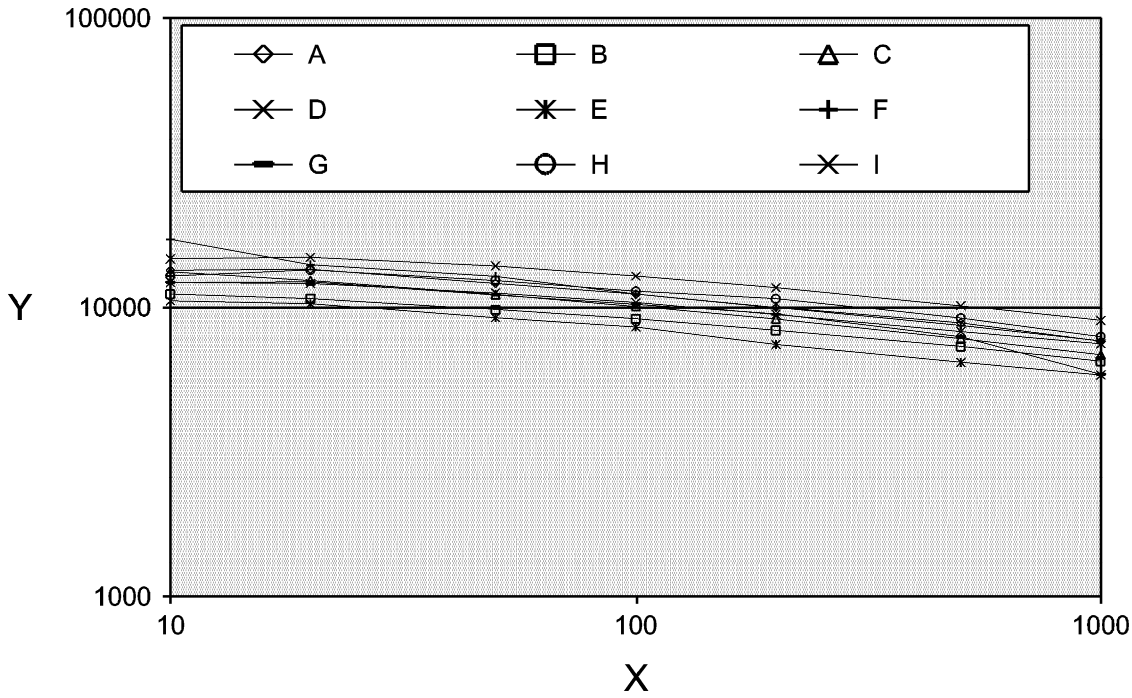
Key

X Stress, Pa

Y Viscosity, Pa.s

A Labo 1
B Labo 2
C Labo 3
D Labo 4
E Labo 5
F Labo 7
G Labo 9
H Labo 10
I Labo 13

Figure 1 — Flow chart of a Newtonian binder (10/20 at 60 °C)



Key

X Stress, Pa

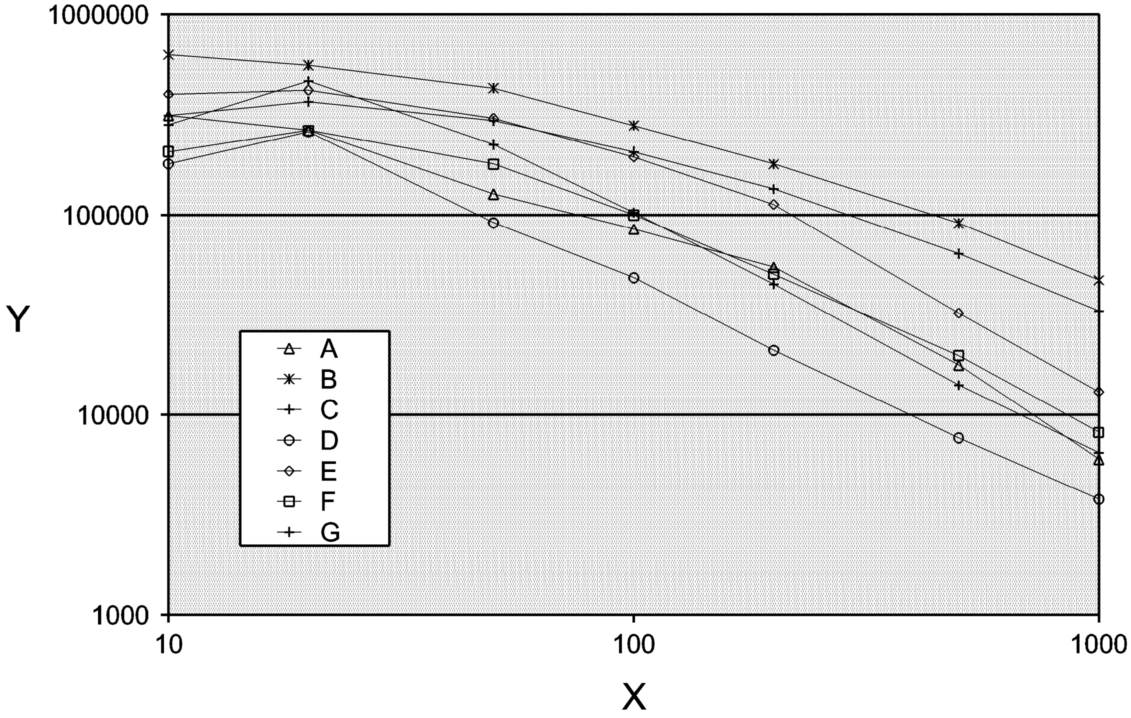
Y Viscosity, Pa.s

A Labo 1 **D** Labo 4 **G** Labo 9

B Labo 2 **E** Labo 5 **H** Labo 10

C Labo 3 **F** Labo 7 **I** Labo 13

Figure 2 — Flow chart of a binder showing a more non-Newtonian plateau (slightly polymer modified bitumen at 60 °C)



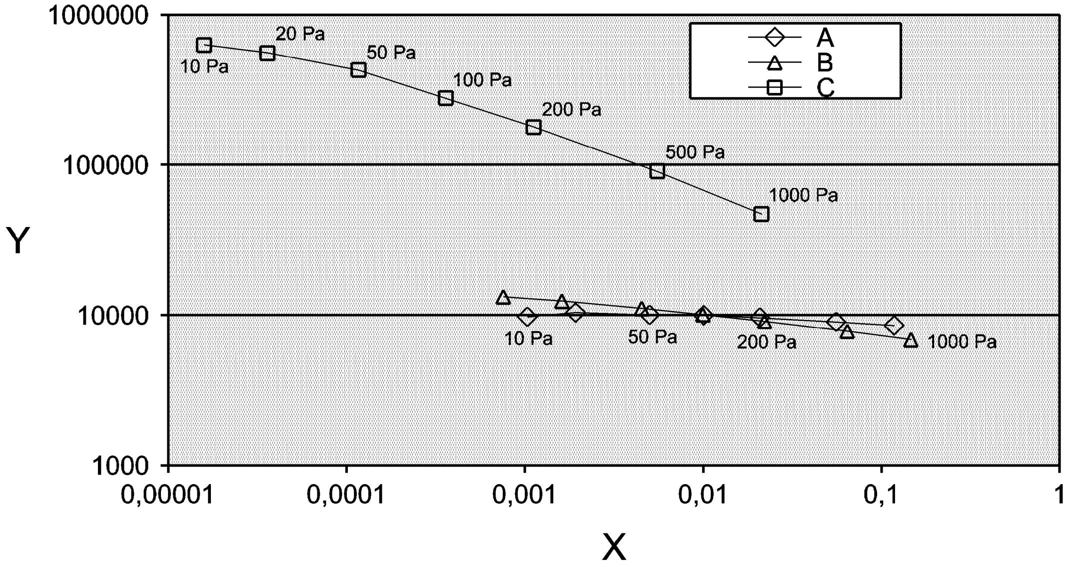
Key

X Stress, Pa
Y Viscosity, Pa.s

A Labo 1
B Labo 5
C Labo 7
D Labo 7 bis

E Labo 9
F Labo 10
G Labo 13

Figure 3 — Flow chart of a non-Newtonian binder (highly polymer modified bitumen at 60 °C)



Key

X Shear rate, s-1

Y Viscosity, Pa.s

A 10/20

B Lightly modified

C Heavily modified

Figure 4 — Flow curves presented in terms of viscosity versus shear rate

Annex A
(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, requires 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 specially designed thermometers may be used 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 binder*s

[2] EN 1427, *Bitumen and bituminous binders — Determination of the softening point — Ring and Ball method*

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