SLOVENSKI STANDARD
oSIST prEN 14770:2022

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Bitumen in bitumenska veziva - Ugotavljanje kompleksnega strižnega modula in faznega kota - Dinamični strižni reometer (DSR)

Bitumen and bituminous binders - Determination of complex shear modulus and phase angle - Dynamic Shear Rheometer (DSR)

Bitumen und bitumenhaltige Bindemittel - Bestimmung des komplexen Schermoduls und des Phasenwinkels - Dynamisches Scherrheometer (DSR)

Bitumes et liants bitumineux - Détermination du module complexe en cisaillement et de l'angle de phase à l'aide d'un rhéomètre à cisaillement dynamique (DSR)

Ta slovenski standard je istoveten z: **prEN 14770**

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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EUROPEAN STANDARD

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English Version

Bitumen and bituminous binders - Determination of complex shear modulus and phase angle - Dynamic Shear Rheometer (DSR)

Bitumes et liants bitumineux - Détermination du module complexe en cisaillement et de l'angle de phase à l'aide d'un rhéomètre à cisaillement dynamique (DSR)

Bitumen und bitumenhaltige Bindemittel - Bestimmung des komplexen Schermoduls und des Phasenwinkels - Dynamisches Scherrheometer (DSR)

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 336.

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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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

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European foreword

This document (prEN 14770:2022) has been prepared by Technical Committee CEN/TC 336 “Bituminous binders”, the secretariat of which is held by AFNOR.

This document is currently submitted to the CEN Enquiry.

This document will supersede EN 14770:2012.

In comparison with the previous edition, the following technical modifications have been made:

a) reference to outdated standards IP PM CM-02 and XPT 66-065 removed;

b) integration of “complex compliance” removed;

c) use of the terms “shear strain” and “shear stress” unified;

d) use of the term “bituminous binder” unified;

e) reference to EN 1427 moved from Clause 2 to Bibliography; references to EN 12607-1, EN 14023 and EN 14769 added to Bibliography;

f) definitions “shear strain controlled mode“ and “shear stress controlled mode” added;

g) use of the term “range of linear viscoelastic behaviour” unified;

h) use of the term “complex shear modulus” together with the corresponding symbol |G*| unified; description of the complex shear modulus slightly revised;

i) 6.1, 7.1 and 8.1 added with reference to Annex E;

j) information about different plate diameters relocated from 5.1 to new 6.2; information about different plate diameters in 6.2 updated and plate diameter of 4 mm added;

k) deviation for rheometer specification removed in 5.1;

l) suitable dimensions for silicone moulds added in 5.2;

m) vials for preparation of test specimen removed in 5.2. 7.3, 7.4 and 8.2;

n) use of the term “specimen” unified;

o) 6.4 “Zero gap setting” revised and clarified;

p) sub-samples smaller 50 g introduced in 7.2;

q) paring of specimen at room temperature removed in 7.4;

r) storage conditions and storage duration of specimens revised in 7.4;

s) 8.2 “Specimen placing onto the rheometer” and 8.3 “Gap setting” revised;

t) gap compensation added in 8.4;

u) calculation of TX and δ_{TX} added in Clause 9 and new Annex D;

v) Clause 10 revised and complemented with new precision data, instead of coefficient of variation repeatability *r* and reproducibility *R* are now used;

w) terms c) and d) added in Clause 11;

x) revision of Annex C “Determination of the linear viscoelastic (LVE) range”;

y) Annex E “Flow Chart” added.

1 Scope

This document specifies a general method of using a dynamic shear rheometer (DSR) for measuring the rheological properties of bituminous binders. The procedure involves determining the complex shear modulus and phase angle of binders over a range of test frequencies and test temperatures when tested in oscillatory shear.

From the test, the complex shear modulus, $|G^*|$, and its phase angle, δ, at a given temperature and frequency are calculated, as well as the components G' and G'' of the complex shear modulus.

This method is applicable to un-aged, aged and recovered bituminous binders.

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 determine the applicability of regulatory limitations prior to use.

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 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.

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
shear strain controlled mode
rheometer control mode where a demand angular displacement is applied to the specimen and the corresponding torque is measured

Note 1 to entry: Using the shear strain factor of the measuring geometry, a specimen shear strain can be calculated from the applied angular displacement. Using the shear stress factor of the measuring geometry, a specimen shear stress can be calculated from the measured torque. Additional corrections can be applied to calculate true specimen shear strain and true specimen shear stress.

3.2
shear stress controlled mode
rheometer control mode where a demand torque is applied to the specimen and the corresponding angular displacement is measured

Note 1 to entry: Using the shear stress factor of the measuring geometry, a specimen shear stress can be calculated from the applied torque. Using the shear strain factor of the measuring geometry, a specimen shear strain can be calculated from the measured angular displacement. Additional corrections can be applied to calculate true specimen shear stress and true specimen shear strain.

3.3
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 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.

3.4
phase angle
δ
phase difference between shear stress and shear strain in harmonic oscillation

3.5
isotherm
equation or curve on a graph representing the behaviour of a material at a constant temperature

3.6
isochrone
equation or curve on a graph representing the behaviour of a material at a constant frequency

3.7
range of linear viscoelastic behaviour
range in which complex shear modulus is independent of shear stress or shear strain

4 Principle

A known oscillatory shear stress is applied to the temperature controlled test geometry, in which the bituminous test specimen is held. The binder's shear strain response to the shear stress is measured. Alternatively, a known oscillatory shear strain is applied to the test specimen and the resulting shear stress is measured.

Except for specific purposes, the test is performed in the region of linear viscoelastic behaviour.

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. Depending on the expected complex shear modulus range different plate diameters (for example 25 mm, 8 mm or 4 mm) are used (see 6.2). 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 capable to determine $|G^*|$, at least in the range of 1 kPa to 10 MPa and the phase angle (δ), in the range 0° to 90°.

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 is nominally 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 The fact that the temperature control range is 5 °C to 85 °C does not imply that accurate results will necessarily be obtained for all binders over this range (see 6.2 and 6.3, Note 1). Furthermore, temperatures outside this range can also be used, provided the results are not affected by material or instrument limitations (see 6.2).

5.2 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, a mould with a cavity of approximately 18 mm in diameter and 2 mm deep may be used. For a testing geometry with a diameter of 8 mm and a gap setting of 2 mm, a mould with a cavity of approximately 8 mm in diameter and 2,5 mm deep may be used. For a testing geometry with a diameter of 4 mm with different gap settings, a mould with a cavity of approximately 4 mm in diameter and 3 mm deep may be used. In any case, the operator shall assure adequate filling of the gap according to 8.3.

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.

5.3 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 rheometers

6.1 General

An informative flow chart for preparation of rheometers is given in Annex E, Figure E.1.

6.2 Selection of Geometry

For different ranges of complex shear modulus plates of different diameters and gap settings shall be used to respect the instruments limitations.

For determining complex shear modulus of bituminous binders in the range 1 kPa to 100 kPa, the geometry with a diameter of 25 mm and a gap setting of 1,0 mm is suitable for most instruments. For determining complex shear modulus of bituminous binders in the range 100 kPa to 10 MPa, the geometry with a diameter of 8 mm a gap setting from 2,0 mm is suitable for most instruments. Overlapping of test results from both geometries is recommended (see 8.5).

For determining complex shear modulus of bituminous binders below 1 kPa, a geometry with a diameter bigger than 25 mm is recommended. Alternatively, the geometry with a diameter of 25 mm may be used provided that test results in the expected range of the complex shear modulus are verified with a calibrated fluid.

Plates of other diameters and other gap settings with different ranges of complex shear modulus may also be used, ensuring compliance effects of the instrument do not affect the results (see 6.3, Note 1), the minimum torque specification of the rheometer is respected and the testing is done in the linear viscoelastic range (see Clause 8).

NOTE Recent research results demonstrate the suitability of a plate diameter of 4 mm for testing complex shear modulus in a range 10 MPa to 1 GPa. Depending on the specimen installation procedure, a gap setting between 1,0 mm and 3,0 mm is generally suitable.

6.3 Set up

Set up the rheometer in the sequence given in the manufacturer's instructions, including the procedure for selecting and setting the correct geometry and gap.

NOTE 1 The selection of system geometry can affect the accuracy of results. The manufacturer can have determined the operational limits and this information can 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, and plotting $|G^*|$ against either frequency or phase angle (δ). Where the divergence between the plots for each geometry exceeds 15 %, this is an indication that compliance effects are affecting one or more of the geometries. The chosen geometry(ies) which shows the more rapid fall in $|G^*|$, or the lower phase angle, indicates that its accuracy limit has been reached. Also, for most rheometers generally used in this document, irrespective of the geometry chosen, values of $|G^*|$ in excess of 10^9 Pa are likely to be suspect. Software corrections to the stiffness can be acceptable provided appropriate validation is supplied by the manufacturer.

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.

NOTE 2 The temperature in the test specimen can differ from the temperature read by the device if insufficient equilibration time is used. A procedure for determining equilibration time is described in Annex B.

6.4 Zero Gap Setting

For initialization, the gap between the plates shall 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 can be set at the mid-point of the temperature range to be tested.

7 Specimen preparation

CAUTION — This document involves handling of apparatus and binders at very high temperatures. Always wear protective gloves and eyeglasses when handling hot binder, and avoid contact with any exposed skin.

7.1 General

An informative flow chart for specimen preparation is given in Annex E, Figure E.1.

7.2 Heating procedure for binders prepared above 100 °C

This procedure is applicable for all binders except cut-backs and stabilised binders from emulsions. 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 in EN 12594 as the maximum time prior to withdrawal of (a) sub-sample(s). For very large bulk samples, it is convenient to redistribute the binder in smaller bulk samples after heating and careful homogenisation. 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.

Binder samples shall not be reheated more than two times.

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 Heating procedure for binders prepared at temperatures less than 100 °C

This procedure is intended for cut-back binders and stabilised binders from emulsions. Warm the binder sufficiently and for the minimum time required until it becomes sufficiently fluid either to prepare smaller bulk samples or to directly prepare moulded test specimens. The binder shall not be heated above 100 °C.

NOTE Normally, warming the binder to its softening point is sufficient. For heavily modified stabilised binders from emulsions, a temperature closer to 100 °C can be more appropriate. For too viscous samples, a spatula can be used to remove small quantities at a time from the bulk to place onto the rheometer plate.

Binder samples shall not be reheated more than two times.

7.4 Specimen manufacturing and storage conditions

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

When the binder reaches 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 of convenient size for handling safely and of sufficient volume, to prepare the required number of test specimens plus approximately 50 %.

Pour into moulds or directly on to sheets. Care shall be taken to avoid air bubbles in the specimen. Choose one or more test shapes that will give reliable measurements with the selected test apparatus. 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 5 °C. All specimens shall be covered.

A minimum storage duration before the de-moulding and testing procedure of 30 min shall be maintained for all bituminous binders. For modified binders that do 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.

8 Procedure

8.1 General

An informative flow chart for the test procedure is given in Annex E, Figure E.2.