SLOVENSKI STANDARD
kSIST FprEN 14770:2011

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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 using a 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 - Rhéomètre à cisaillement dynamique (DSR)

Ta slovenski standard je istoveten z: **FprEN 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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FINAL DRAFT
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English Version

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

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

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If this draft becomes a European Standard, CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

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

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Foreword

This document (FprEN 14770:2011) 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 Unique Acceptance Procedure.

This document will supersede EN 14770:2005.

This European standard is based on IP PM CM-02 [1] and XPT 66-065 [2].

In this document, Annex A and Annex B are informative.

1 Scope

This European standard specifies a number of methods using a dynamic shear rheometer (DSR) capable of 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 norm of the complex shear modulus, **IG*I**, and its phase angle, δ, at a given temperature and frequency can be calculated, as well as the components G', G", J' and J" of the complex shear modulus and of the complex compliance.

This method is applicable to unaged, aged and recovered bituminous binders, cut-backs and bituminous binders stabilised from emulsions.

WARNING — The 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 to determine the applicability of regulatory limitations prior to use.

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 1427, *Bitumen and bituminous binders — Determination of softening point — Ring and Ball method*

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

3 Terms and definitions

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

3.1
norm of the complex shear modulus
IG*I
ratio of peak stress to the peak strain in harmonic sinusoidal oscillation

3.2
phase angle
δ
phase difference between stress and strain in harmonic oscillation

3.3
norm of the complex compliance
IJ*I
ratio of the peak strain to the peak stress in harmonic sinusoidal oscillation

NOTE The real parts of the complex shear modulus IG*I and the complex shear compliance IJ*I are respectively G´ and J´ and are associated with the elastic part of material behaviour which represent energy stored during a shear cycle. They are complex shear modulus or complex shear compliance multiplied with cosine of phase angle expressed in degrees.

The imaginary parts of the complex shear modulus and the complex shear compliance are respectively G´´ and J´´ and are associated with the viscous part of material behaviour which represents energy dissipated during a shear cycle. They are complex shear modulus or complex shear compliance multiplied with sine of phase angle expressed in degrees.

3.4
isotherm
equation or curve on a graph representing the behaviour of a material at a constant temperature

3.5
isochrone
equation or curve on a graph representing the behaviour of a material at a constant frequency

3.6
region of linear viscoelastic behaviour
region in which complex dynamic (shear) modulus is independent of (shear) stress or strain

4 Principle

A known oscillatory shear stress is applied to the temperature controlled test geometry, in which is held the bituminous test specimen. The binder's strain response to the 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, test is operated 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. The temperature control system shall encompass both plates, to avoid temperature gradients across the plates. Where 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 1 kPa to 10 MPa (± 2 %) and the phase angle (δ), in the range 0° to 90° (± 0,1°).

NOTE 1 For rheometers using an air bearing, and to avoid damage, the air supply to the bearing should be switched on before the instrument is switched on. When not in use, the spindle should be secured.

NOTE 2 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 a temperature it is intended to test. Rheometers using radio frequency (RF) heating and/or liquid gas cooling should be used in accordance with the manufacturer's instructions.

NOTE 3 Where the bottom plate is nominally the same diameter as the top plate, then 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, then the manufacturer, or a qualified technician, should re-align the plate geometry.

NOTE 4 For information, diameters from 8 mm to 25 mm and gap settings from 0,5 mm to 2,0 mm have been found to be suitable for bituminous binders. In terms of operational ranges, 25 mm plates are generally suitable for stiffnesses in the range 1 kPa to 100 kPa and 8 mm plates suitable for stiffnesses (|G*|) in the range 100 kPa to 10 MPa. Plates of other diameters can also be used, providing compliance effects of the instrument are not affecting the results (see 6.1, Note 1) and the testing is done in the linear region, see Clause 8.

NOTE 5 The fact that the temperature control range is 5 °C to 85 °C should not be taken to imply that accurate results will necessarily be obtained for all binders over this range (see 5.1, Note 4 and 6.1, Note 1). Also temperatures outside this range can be used provided results are not affected by machine compliance.

5.2 Moulds, sheet materials or vials, 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. Vials or containers, where used, shall be of an appropriate material and size for the purpose.

NOTE The use of grease or other anti-stick products should be avoided because they can affect the adherence of the sample 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 Set up

Set up the rheometer in the sequence given in the manufacturer's instructions, including the procedure for the selecting and setting the correct geometry and gap. Select the appropriate oscillation package, if applicable, from the software menu. It is essential that the operational limits of stiffness for the selected geometry are determined.

NOTE 1 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, 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 for this European Standard, irrespective of geometry chosen, values of |G*| in excess of 108 Pa are likely to be suspect. Software corrections to the stiffness may be acceptable provided appropriate validation is supplied to the operator.

NOTE 2 The rheometer and temperature control system should be calibrated at regular intervals in accordance with the quality assurance procedure of the laboratory. A suitable method is that 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. 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. A procedure for determining equilibration time is described in Annex B.

6.2 Zero gap setting

Set the zero gap between the plates prior to loading the test specimen, with both plates at nominally the same temperature.

Carefully prepare the rheometer plates for receipt of 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.

NOTE Gap settings within the range 0,5 mm to 2,0 mm have been found to be suitable for bituminous binders over the temperature range of 5 °C to 85 °C for parallel plate geometries. Values of 1 mm for 25 mm plates and 2 mm for 8 mm plates are recommended. The gap set will change with temperature and appropriate steps will need to be taken to account for these changes. If the DSR has an automatic gap compensation feature, the gap may be set at any temperature within the range to be covered. If the DSR has no gap compensation feature, the gap should be set at a number of different mid-point temperatures not exceeding 15 °C intervals within the range to be tested. A suitable means of correcting for gap changes for temperatures different from the gap setting temperature should be reported. One way is to set the gap at each test temperature; another is to apply a software correction.