SLOVENSKI STANDARD
SIST EN 14770:2012

01-september-2012

Nadomešča:
SIST EN 14770:2005

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 - Rhéomètre à cisaillement dynamique (DSR)

Ta slovenski standard je istoveten z: **EN 14770:2012**

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

SIST EN 14770:2012 **en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 14770

May 2012

ICS 75.140; 91.100.50

Supersedes EN 14770:2005

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 - Rhéomètre à cisaillement dynamique (DSR)

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

This European Standard was approved by CEN on 7 April 2012.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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Foreword

This document (EN 14770:2012) has been prepared by Technical Committee CEN/TC 336 “Bituminous binders”, the secretariat of which is held by AFNOR/BNPé.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by November 2012, and conflicting national standards shall be withdrawn at the latest by November 2012.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 14770:2005.

Compared with EN 14770:2005, the following changes have been made:

a) Note 2 added to 3.3;

b) Principle application clarified in Clause 4;

c) Note 2 improved in 5.1;

d) Rewording of 6.2 and previous Note 2 deleted;

e) 7.1 has been re-structured and requirements for reheating times added;

f) requirement for reheating added in 7.2;

g) sub-clause 7.3 added;

h) Clause 8 revised and Note 4 added in 8.3;

i) Annex C (informative) added.

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

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

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 un-aged, 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 documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 1 to entry: 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 represents 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 the bituminous test specimen is held. 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, 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. 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 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 the temperature intended for testing is. Rheometers using radio frequency (RF) heating and/or liquid gas cooling or other heating/cooling systems 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, 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 4 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 do not affect 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). Furthermore, temperatures outside this range can also be used, provided the 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 the geometry chosen, values of |G*| in excess of 10^8 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. 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 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.

7 Sample preparation

CAUTION — This European Standard 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 Heating procedure for binders prepared above 100 °C

This procedure is for all binders except cut-backs and stabilised binders from emulsions. If the softening point of the binder is unknown, determine using EN 1427. Prepare pure, oxidised or special bitumens in accordance with EN 12594.

NOTE 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 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 to 100 g: max 30 min;

— 100 g to 500 g: max 1 h;

— 500 g to 1 kg: max 2 h.

7.2 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 vials or moulded test samples. 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 closed to 100 °C may be more appropriate. For too viscous samples, a spatula may 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.

7.3 Sample manufacturing and storage conditions

7.3.1 Using moulds or sheet materials

Moulds or sheet materials can 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 %.