SLOVENSKI STANDARD
SIST EN 14770:2005

01-december-2005

Bitumen in bitumenska veziva – Ugotavljanje kompleksnega strižnega modula in faznega kota – Dinamini 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 - Rhéometre a cisaillement dynamique (DSR)

Ta slovenski standard je istoveten z: **EN 14770:2005**

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 14770:2005 **en**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 14770

October 2005

ICS 75.140; 91.100.50

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 26 August 2005.

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. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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

Management Centre: rue de Stassart, 36 B-1050 Brussels

Contents

Page

Foreword

This European Standard (EN 14770:2005) has been prepared by Technical Committee CEN/TC 336 “Bituminous binders”, the secretariat of which is held by AFNOR.

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 April 2006, and conflicting national standards shall be withdrawn at the latest by April 2006.

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, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and 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 unaged, aged and recovered bituminous binders, cut-back or fluxed bituminous binders stabilised from emulsions.

WARNING — The use of this standard can involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate health and safety practices and 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 test samples.*

3 Terms and definitions

For the purposes of this European Standard, 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 sinusoidal oscillation

3.3
norm of the complex shear 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 the material behaviour. 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 the material behaviour. The tangent of the phase angle δ represents the ratio of its viscous component over its elastic component (e.g. tan δ equals G''/G' or J''/J').

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

3.5
isochron
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.

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 manufacturers 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 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.

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 standard, irrespective of 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 adviseable 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 Gap setting

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

NOTE 1 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. 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 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.

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. Set the temperature of the bottom plate to a maximum of the softening point of the binder plus 20 °C ± 5 °C or at 90 °C ± 5 °C, whichever is the lower, to enable satisfactory bonding of the test specimen to the plates.

NOTE 2 Alternative temperatures may be used for the temperature of the bottom plate provided that adhesion takes place between the binder and the plate, and that the binder is sufficiently fluid to allow the gap to be achieved.

7 Sample preparation

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

7.1 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 by EN 1427. Prepare pure, oxidised or special bitumens in accordance with EN 12594.

NOTE 1 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 °C ± 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.

When the binder reaches temperature after the heating period, stir and mix with a spatula, 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 %.

If using vials, pour aliquots of the sub-sample into vials, filling each to the top, and place a cover on each vial. Pour sufficient vials for testing of the binder plus spares for possible repeat testing. Store the covered vials at ambient temperature prior to use. If using moulds or sheet material, pour into moulds or directly on to sheets. Choose one or more test shapes that will give reliable measurements with the selected test apparatus. The moulds, once cooled to ambient, shall be pared using a suitable trimming tool to the desired height and shall be stored in a refrigerator (approximate temperature 5 °C), not exceeding 30 min prior to testing in the rheometer.

NOTE 2 Heating times should be kept to a minimum.

NOTE 3 The binders, in particular modified binders, should be stirred to ensure homogeneity.

NOTE 4 Any test specimens not tested within 7 days of preparation should be discarded. For specimens prepared from mould paring should only be carried out within 24 h of use.

7.2 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 to either pour onto the rheometer plates or onto a silicone-based material for subsequent transfer into the rheometer. The binder shall not be heated above 100 °C. Test specimens not used immediately shall be covered to prevent loss of volatiles and shall be stored in a refrigerator (approximate temperature 5 °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.

8 Procedure

Heat a vial of binder to its softening point plus 85 °C ± 5 °C for unmodified bitumen, a maximum of 100 °C for cut backs and stabilised binders from emulsions, or to 180 °C - 200 °C for modified binders, stir to ensure homogeneity, and 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.