SLOVENSKI STANDARD
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Bitumen in bitumenska veziva - Ponavljajoči obremenilni in razbremenilni preskus lezenja (MSCRT)

Bituminen and Bituminous Binders - Multiple Stress Creep and Recovery Test (MSCRT)

Bitumen und bitumenhaltige Bindemittel - MSCR-Prüfung (Multiple Stress Creep and Recovery Test)

Bitumes et liants bitumineux- Essai de fluage-recouvrance sous contraintes répétées

Ta slovenski standard je istoveten z: **prEN 16659**

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 COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

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Foreword

This document (prEN 16659:2013) 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.

1 Scope

1.1 This test method covers the determination of percent recovery and non-recoverable creep compliance of bitumen and bituminous binders by means of Multiple Stress Creep and Recovery (MSCR) testing. The MSCR test is conducted using the Dynamic Shear Rheometer (DSR) in creep mode at a specified temperature.

1.2 This standard is appropriate for unaged material, material aged in accordance with EN 12607-1 (RTFOT), material aged in accordance with EN 14769 (PAV), material aged in accordance with both EN 12607-1 and EN 14769.

Other ageing methods, for example EN 15323 (RCAT) can also be used to produce material for this standard.

1.3 The percent recovery at multiple stress levels is intended to determine the presence of elastic response and stress dependence of bituminous binders.

1.4 The non-recoverable creep compliance at multiple stress levels is intended as an indicator for the sensitivity to permanent deformation and stress dependence of bituminous binders.

1.5 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and 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.

2.1 *ASTM Standards:*

ASTM D7405-10a, Standard Test Method for Multiple Stress Creep and Recovery (MSCR) of Asphalt Binder Using a Dynamic Shear Rheometer

2.2 *AASHTO Standards*

AASHTO TP 70-10, Standard Method of Test for Multiple Stress Creep Recovery (MSCR) Test of Asphalt Binder Using a Dynamic Shear Rheometer (DSR)

M320 Specifications for performance graded asphalt binder

2.3 EN Standards

EN 58, Bitumen and bituminous binders – Sampling bituminous binders.

EN 12594, Bitumen and bituminous binders – Preparation of test samples.

EN 12597, Bitumen and bituminous binders – Terminology.

EN12607-1, Bitumen and bituminous binders - Determination of the resistance to hardening under the influence of heat and air. Part 1 – RTFOT

EN 14769, Bitumen and bituminous binders - Accelerated long-term ageing conditioning by a Pressure Ageing Vessel (PAV).

EN 15323, Bitumen and bituminous binders - Accelerated long-term ageing/conditioning by the rotating cylinder method (RCAT)

EN 14770, Bitumen and bituminous binders - Determination of complex shear modulus and phase angle - Dynamic Shear Rheometer (DSR).

EN ISO 4259, Petroleum products. Determination and application of precision data in relation to methods of test.(ISO 4259)

3 Terms and definitions

For definitions of general terms used in this standard, refer to EN 12597.

3.1 creep and recovery
a standard rheological test protocol whereby a specimen is subjected to a constant load for a fixed time period, then allowed to recover, at zero load, for a fixed time period.

3.2 percent recovery (%R)
the recovered strain in a specimen during the recovery portion of a cycle, expressed in percent.

3.3 non-recoverable creep compliance (J_{nr})
the residual strain in a specimen after a creep and recovery cycle divided by the stress applied.

4 Principle

This test method is used to determine the presence of elastic response in bitumen and bituminous binders under shear creep and recovery at two stress levels at a specified temperature. The presence of this elastic response is determined by measuring the percent recovery and non-recoverable compliance of the binder. Non-recoverable creep compliance has been shown to be an indicator of the resistance of bitumen and bituminous binders to permanent deformation under repeated load.

The test shall be conducted at 40, 50, 60 or 70°C as appropriate. Other test temperatures may be used for comparative purposes. Sample preparation and apparatus are in accordance with EN 14770 using the 25 mm parallel plate geometry with a 1 mm gap setting.

The sample is loaded at constant stress for 1 s, then allowed to recover for 9 s. Ten creep and recovery cycles are run at 0,100 kPa creep stress followed by ten more cycles at 3,200 kPa creep stress (see Figure 1).

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 run in stress control mode and be able to collect data samples every 0.1 seconds.

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 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.

5.2 Moulds or sheet materials.

For the preparation of the test specimens, the moulds or sheet material shall be of silicone or similar material, which does not adhere to the test specimen.

NOTE: The use of greases 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 using 25 mm parallel plates and a 1 mm gap.

NOTE : The rheometer and temperature control system shall 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 are 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.

6.2 Zero gap setting

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

7 Sample preparation

CAUTION — This 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.

Samples shall be taken in accordance with EN 58 and prepared in accordance with EN12594

7.1 Heating procedure for preparation of the binder

The softening point of the binder may be determined by EN 1427 or, 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.

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 hour

500 g to 1 kg: max. 2 hour

7.2 Heating procedure for preparation of binders from bituminous emulsion of cut-back or fluxed bituminous binders recovered by evaporation and/or subject to a stabilising procedure.

In order to protect the recovered and/or stabilized binder from excessive damage (due to volatile loss or thermal effects), heating of the recovered and/or stabilized binder shall be strictly controlled in accordance with EN 12594 and this method. In all cases, heating times shall be kept to a minimum. Binder samples shall not be reheated more than two times

7.2.1 Emulsion containing a free flux binder

For emulsions not categorised as a fluxed emulsion, the binder shall be heated to a temperature between expected softening point + 80 °C and expected softening point + 100°C.

7.2.2 Emulsion containing a mineral fluxed binder

For emulsions categorised as a fluxed emulsion, the binder shall be heated to a temperature between expected softening point + 60 °C and expected softening point + 80°C.

7.2.3 Emulsion containing a vegetable fluxed binder

For emulsions categorised as a fluxed emulsion, the binder shall be heated to a temperature between expected softening point + 80 °C and expected softening point + 100°C.

7.2.4 Cut-back or fluxed bitumen with mineral oil

For cut-backs or fluxed bitumen where the flux is a mineral oil, the binder shall be heated to a temperature between expected softening point + 60 °C and expected softening point + 80°C.

7.2.5 Cut-back or fluxed bitumen with vegetable oil

For cut-backs or fluxed bitumen where the flux is a vegetable oil, the binder shall be heated to a temperature between expected softening point + 80 °C and expected softening point + 100°C.

7.3 Sample manufacturing and storage conditions

Moulds or sheet materials can be used for all types of binders.
When the binder reaches the workability temperature, stir and mix to ensure homogeneity.

NOTE 1: For polymer modified binders, the use of a high shear mixer is recommended.

Pour into moulds or directly on to sheets. The moulds, once cooled to ambient, shall be pared using a suitable trimming tool to the desired height and shall be stored at ambient temperature. Samples likely to contain volatiles shall be covered.

NOTE 2: Paring should be avoided as much as possible by controlling the mass of binder to be poured into the moulds.

Following minimum and maximum storage times before the de-moulding and testing procedure shall be observed:

Minimum delay:

2 hours for pure bitumen

12 hours for PmB's

Maximum delay:

3 days whatever the binder

8 Procedure

8.1 Sample placing onto the rheometer

The samples may be placed in the refrigerator (approximate temperature of 5°C) for a maximum time of 30min prior to demoulding. Demoulding and loading onto the rheometer shall occur just after removal from the refrigerator.

Plates of the rheometer shall be pre-heated at the test temperature. If the upper plate has no heating this can be done by contact with the lower plate and/or by using a water bath.

Place the sample onto the bottom plate.

8.2 Gap setting

Bring the parallel plates to a gap from 1,025 mm to 1,050 mm and trim any excess binder with a knife, a spatula or a special trimming tool. After trimming, bring the parallel plates to the 1 mm 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, re-prepare the rheometer plates, and prepare a fresh test specimen. All the process shall not take more than 10 minutes.

8.3 Testing

8.3.1 Bring the test plates to the selected test temperature and allow the specimen to reach thermal equilibrium at least during 15 minutes.

8.3.2 Load the specimen at a constant creep stress of 0,100 kPa for 1,00 second duration creep and follow with a zero stress recovery of 9,00 seconds duration. The required full torque for each creep cycle shall be achieved within 0,003 seconds from the start of the creep cycle as certified by the equipment manufacturer. Record the stress and strain at least every 0,10 seconds for the creep cycle and at least every 0,45 seconds for the recovery cycle on a running accumulated time such that, in addition to other data points, data points at 1,00 second and 10,00 seconds for each cycle's local time are recorded if possible (see Figure 2).

If the DSR does not record the strain at 1,00 (± 0,05) second and 10,00 (± 0,05) seconds, the software of the DSR should be updated by the manufacturer, if possible. If not, the test is not acceptable.

NOTE: If the creep and recovery curves will be used for modelling, more frequent data points may be required.

8.3.3 Allowing no rest period between cycles, repeat the creep and recovery cycle in 8.3.2 nine times for a total of ten cycles.

8.3.4 Allowing no rest period following 8.3.3, repeat the ten creep and recovery cycles of 8.3.2 and 8.3.3 utilizing a load of 3,200 kPa.

NOTE: The total time required to complete the two-step creep and recovery at the two stress levels is 200 seconds.

8.4 Calculations

For each of the twenty creep and recovery cycles record the following (see figure 1):

8.4.1 Initial strain absolute value at the beginning of the creep portion of each cycle. This strain shall be denoted as ε0. The first strain value of the test, (that is, at time 0s) is always 0.

8.4.2 The strain absolute value at the end of the creep portion (that is, after 1,0 s) of each cycle. This strain shall be denoted as ε_c.

8.4.3 The adjusted strain value at the end of creep portion (that is, after 1,0 s) of each cycle:

$$\varepsilon_1=\varepsilon_c-\varepsilon_0 \quad (1)$$

8.4.4 The strain value at the end of the recovery portion (that is, after 10,0 s) of each cycle. This strain shall be denoted as ε_r.

8.4.5 The adjusted strain value at the end of recovery portion (that is, after 10,0 s) of each cycle:

$$\varepsilon_{10}=\varepsilon_r-\varepsilon_0 \quad (2)$$

9 Expression of results

9.1 Using the results obtained in 8.4, determine the average percent recovery and non recoverable creep compliance for bitumen and bituminous binders at creep stress levels of 0,100 kPa and 3,200 kPa as follows:

9.1.1 For each of the ten cycles at a creep stress of 0,100 kPa calculate the percent recovery, R_N (0,1kPa, *N*), for *N* = 1 to 10:

$$R_N\,(0{,}1\text{kPa, N}) = \left(\frac{\varepsilon_1-\varepsilon_{10}}{\varepsilon_1}\right)\times 100 \quad (3)$$

9.1.2 For each of the ten cycles at a creep stress of 3,200 kPa calculate the percent recovery, R_N (3,2kPa, *N*), for *N* = 1 to 10:

$$R_N(3{,}2\text{kPa, N}) = \left(\frac{\varepsilon_1-\varepsilon_{10}}{\varepsilon_1}\right)\times 100 \quad (4)$$

9.1.3 Calculate average percent recovery at 0,100 kPa:

$$R_{0,1\text{kPa}} = \Sigma\,(R_N\,(0{,}1\text{kPa, N}))/10 \text{ for N=1 to 10} \quad (5)$$

9.1.4 Calculate average percent recovery at 3,200 kPa:

$$R_{3,2\text{kPa}} = \Sigma\,(R_N\,(3{,}2\text{kPa, N}))\,/\,10 \text{ for } N = 1 \text{ to } 10 \quad (6)$$

9.1.5 Calculate percent difference in recovery between 0,100 kPa and 3,200 kPa:

$$R_{diff} = ((R_{0,1\text{kPa}}-R_{3,2\text{kPa}})\cdot 100)\,/\,(R_{0,1\text{kPa}}) \quad (7)$$