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Bitumen and Bituminous Binders — Multiple Stress Creep and Recovery Test (MSCRT)

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National foreword

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The UK participation in its preparation was entrusted to Technical Committee B/510/19, Bitumen and related products.

A list of organizations represented on this committee can be obtained on request to its secretary.

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

Bitumes et liants bitumineux- Essai de fluage-recouvrance sous contraintes répétées (essai MSCR)

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

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Content

European foreword

This document (EN 16659:2015) 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of an identical text or by endorsement, at the latest by June 2016, and conflicting national standards shall be withdrawn at the latest by June 2016.

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

1.3 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.

1.4 This European 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 European 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.

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*

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

ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 12597 and the following apply.

3.1
creep and recovery
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)
recovered strain in a specimen during the recovery portion of a cycle, expressed in percent

3.3
non-recoverable creep compliance (J_{nr})
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 50 °C, 60 °C, 70 °C or 80 °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 10 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. 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 run in stress controlled mode and be able to collect data samples every 0,1 s.

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.

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.

The use of greases or other anti-stick products is not allowed 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.

Carefully prepare the rheometer plates before placing 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

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 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

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 EN 12594.

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 homogenization. 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 according to EN 14023, the temperature shall be between 180 °C and 200 °C irrespective of the softening point; the temperature shall not exceed 200 °C in any case

Binder samples shall not be reheated more than two times.

Reheating times for sub-samples shall conform to following requirements:

— 50g to 100g: max. 30 min

— 100g to 500g: max. 1 h

— 500g to 1 kg: max. 2 h

7.2 Heating procedure for preparation of binders from bituminous emulsion or cut-back or fluxed bituminous binders recovered by evaporation and/or subjected to a stabilizing 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 Residue from emulsion containing a flux free binder

For emulsions not categorized 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 Residue from emulsion containing a mineral fluxed binder

For emulsions categorized 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 Residue from emulsion containing a vegetable fluxed binder

For emulsions categorized 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 bitumens 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 bitumens 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 Specimen 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.

For polymer modified binders, the use of a mixer is recommended.

Pour into moulds or directly on to sheets. The test specimen, 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.

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: 30 min

Maximum delay: 24 h

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 30 min 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 rheometer.

The plates should be heated to a higher temperature if poor bonding between the binder and plates is observed.

8.2 Gap setting

Bring the parallel plates to a gap between 1,025 mm and 1,050 mm and trim any excess binder with a knife, a spatula or a special trimming tool at the test temperature. 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 min.

The plates should be heated to a higher temperature if the sample cannot be deformed to the required gap.

8.3 Testing

8.3.1 Bring the test plates to the selected test temperature with the accuracy of 0,1 °C and allow the specimen to reach thermal equilibrium at least during 15 min.

It should be ensured that the strain value is set to zero at the beginning of the test.

8.3.2 Load the specimen at a constant creep stress of 0,100 kPa for 1,00 s duration creep and follow with a zero stress recovery of 9,00 s duration. Record the stress and strain at least every 0,10 s for the creep cycle and at least every 0,45 s for the recovery cycle on a running accumulated time such that, in addition to other data points, data points at 1,00 s and 10,00 s 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 10 cycles.

— Allowing no rest period following 8.3.3, repeat the 10 creep and recovery cycles of 8.3.2 and 8.3.3 utilizing a load of 3,200 kPa.

— After testing, the specimen shall be discarded because the same specimen cannot be tested twice.

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

9 Calculations

For each of the 20 creep and recovery cycles record the following (see Figure 1):

9.1 Absolute strain value at the beginning of the creep portion of each cycle. This strain shall be denoted as ε_0^N for absolute strain at the start of cycle N (so ε_0^1 is zero).

9.2 The absolute strain 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^N for the cycle N.

9.3 The adjusted strain value at the end of creep portion (that is, after 1,0 s) of each cycle. Use index N, which stands for cycle number since there is a ε_1 for every cycle.

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

9.4 The absolute 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^N for the cycle N.

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

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

10 Expression of Results

10.1 Using the results obtained in Clause 9, determine the average percent recovery, to the nearest 0,1 %, and non-recoverable creep compliance for bitumen and bituminous binders at creep stress levels of 0,100 kPa and 3,200 kPa, as follows:

10.1.1 For each of the 10 cycles at a creep stress of 0,100 kPa calculate the percent recovery, use index N, which stands for cycle number, since there is a $\%R_{0,1kPa}$ for every cycle.

$$\%R_{0,1kPa}^N = 100\cdot\left(\varepsilon_1^N-\varepsilon_{10}^N\right)\Big/\varepsilon_1^N \quad (3)$$

10.1.2 For each of the 10 cycles at a creep stress of 3,200 kPa calculate the percent recovery, use index N, which stands for cycle number, since there is a $\%R_{3,2kPa}$ for every cycle.

$$\%R_{3,2kPa}^N = 100\cdot\left(\varepsilon_1^N-\varepsilon_{10}^N\right)\Big/\varepsilon_1^N \quad (4)$$

10.1.3 Calculate average percent recovery at 0,100 kPa:

$$\%R_{0,1kPa} = \frac{1}{10}\sum_{N=1}^{10}\left(\%R_{0,1kPa}^N\right) \quad (5)$$

10.1.4 Calculate average percent recovery at 3,200 kPa:

$$\%R_{3,2kPa} = \frac{1}{10}\sum_{N=1}^{10}\left(\%R_{3,2kPa}^N\right) \quad (6)$$

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

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

10.1.6 For each of the 10 cycles at a creep stress of 0,100 kPa calculates the non-recoverable creep compliance, $J_{nr0,1kPa}$, expressed in kPa^{-1}.

$$J_{nr\ 0,1kPa}^{\ N} = \varepsilon_{10}^{N} / 0,100 \quad (8)$$

10.1.7 For each of the 10 cycles at a creep stress of 3,200 kPa calculate the non recoverable creep compliance, $J_{nr3,2kPa}$, expressed in kPa^{-1}.

$$J_{nr\ 3,2kPa}^{\ N} = \varepsilon_{10}^{N} / 3,200 \quad (9)$$

10.1.8 Calculate average non-recoverable creep compliance at 0,100 kPa:

$$J_{nr0,1kPa} = \frac{1}{10} \sum_{N=1}^{10} \left(J_{nr\ 0,1kPa}^{\ N} \right) \quad (10)$$

10.1.9 Calculate average non-recoverable creep compliance at 3,200 kPa:

$$J_{nr3,2kPa} = \frac{1}{10} \sum_{N=1}^{10} \left(J_{nr\ 3,2kPa}^{\ N} \right) \quad (11)$$

10.1.10 Calculate percent difference in non-recoverable creep compliance between 0,100 kPa and 3,200 kPa:

$$J_{nr-diff} = 100 \left(J_{nr3,2kPa} - J_{nr0,1kPa} \right) / J_{nr0,1kPa} \quad (12)$$

11 Precision

11.1 General

Precision was determined in two round-robin exercises: the first one in 2012 including 4 binders and 26 participating laboratories and the second in 2014 including 7 binders and 23 laboratories. All measurements were repeated three times. This produced sufficient data to estimate the repeatability and reproducibility of the test method according to ISO 5725-2.

The results of both round robin tests are summarized in Table 1 for plain bitumen and in Table 2 for Polymer Modified Bitumen not containing EVA, all at a test temperature of 60 °C and 70 °C.

11.2 Repeatability, r

The difference between two tests results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the values of r given for each binder in Tables 1 and 2 only in one case in 20.

11.3 Reproducibility, R

The difference between two tests results, obtained by the different operators working in different Laboratories on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the values of R given for each binder in Tables 1 and 2 only in one case in 20.

Table 1 — Precision results for tests at 60 °C and 70°C for paving grade bitumen

	% Recovery (in %)	**Jnr** (in % of Jnr)
r	1 %	6 %
R	5 %	33 %

Table 2 — Precision results for tests at 60 °C and 70°C for polymer modified bitumen

	% Recovery (in %)	**Jnr** (in % of Jnr)
r	2 %	9 %
R	12 %	43 %

12 Report

Report the following information:

a) sample identification (including an identification of the aging e.g. RTFOT, PAV, RCAT, if applicable);

b) date of test;

c) test temperature;

d) average percent recovery at 0,100 kPa, $R_{0,1\ kPa}$;

e) average percent recovery at 3,200 kPa, $R_{3,2\ kPa}$;

f) percent difference between average recovery at 0,100 kPa and 3,200 kPa, R_{diff};

g) non-recoverable creep compliance at 0,100 kPa, $J_{nr0,1kPa}$ to three significant figures expressed as kPa^{-1};

h) non-recoverable creep compliance at 3,200 kPa, $J_{nr3,2kPa}$ to three significant figures expressed as kPa^{-1};

i) percent difference between non-recoverable creep compliance at 0,100 kPa and 3,200 kPa, $J_{nr\text{-}diff}$.

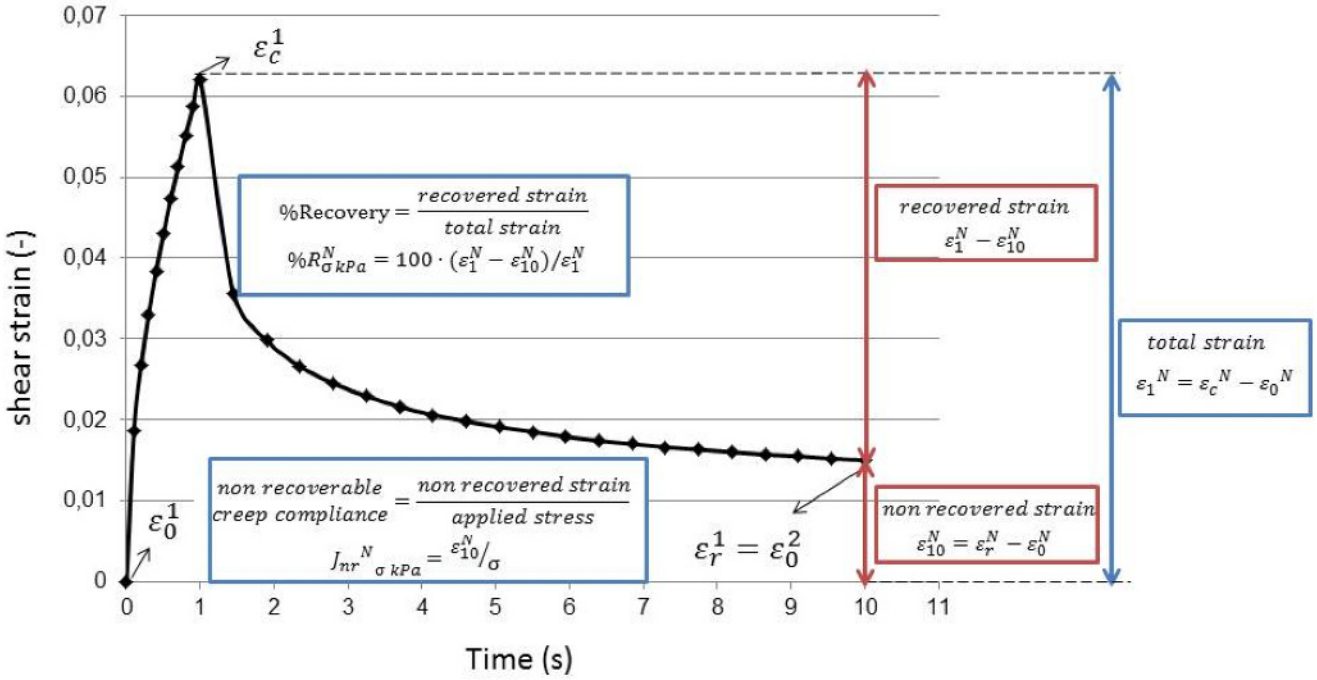

Figure 1 — Typical creep-recovery cycle

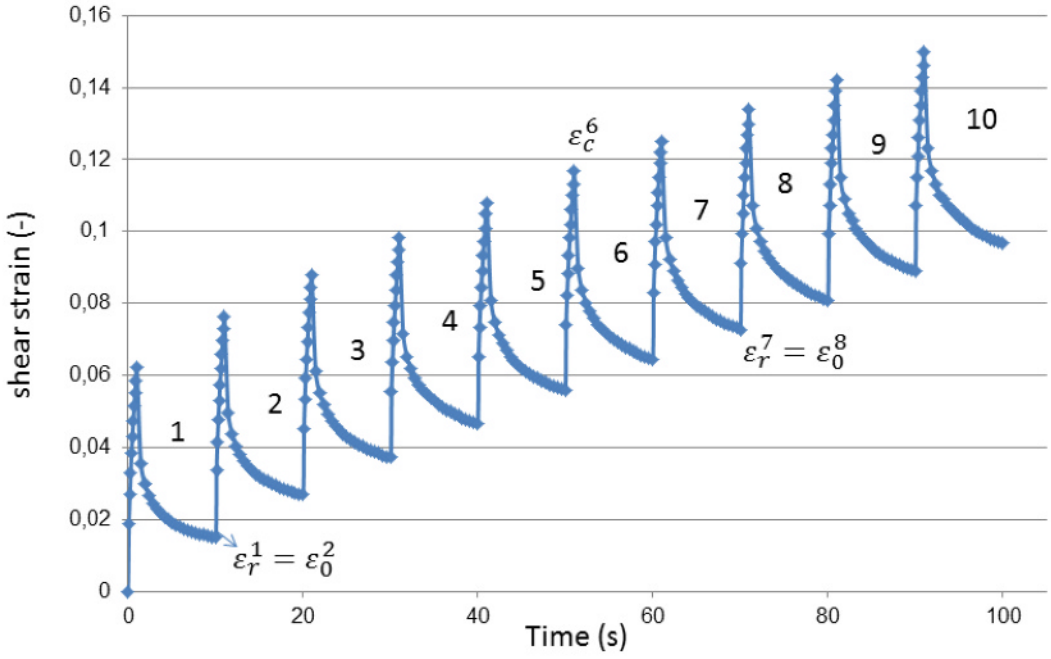

Figure 2 — Typical creep-recovery curve after 10 consecutive cycles

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