SLOVENSKI STANDARD
SIST EN 15323:2007

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Bitumen in bitumenska veziva - Pospešeno staranje z rotacijsko cilindrično metodo (RCAT)

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

Bitumen und bitumenhaltige Bindemittel - Beschleunigte Langzeit-Alterung mit dem Verfahren mit rotierendem Zylinder (RCAT)

Bitumes et liants bitumineux - Vieillissement/conditionnement long-terme accéléré par la méthode du cylindre tournant (RCAT)

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Foreword

This document (EN 15323:2007) 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of an identical text or by endorsement, at the latest by October 2007, and conflicting national standards shall be withdrawn at the latest by October 2007.

This European Standard is based upon documents [1], [2] and [3], referenced in Bibliography.

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, 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 and United Kingdom.

1 Scope

This European Standard specifies an accelerated ageing/conditioning procedure for bitumen, bituminous binders and bituminous mastics. The procedure involves rotating cylinder ageing (RCA), i.e binder ageing at moderate temperatures in a large cylinder rotating in an oven under oxygen flow conditions. Prior to long-term ageing with this method, samples are prepared in the condition they would be applied to the road.

This method is also applicable to modified binders and bituminous mastics.

NOTE For binders used in hot asphalt applications pre-conditioning of the sample would typically be by one of the methods given in EN 12607-1 or EN 12607-2 or directly in the RCAT cylinder to an equivalent ageing level. For binders used in bituminous emulsion and cut-back application, stabilising the sample would typically be by methods given in EN 14895. For bituminous emulsion, stabilising the sample can also be realised directly in the RCAT cylinder under a nitrogen flow (quantity-temperature-time have still to be experienced).

WARNING — Use of this document can involve hazardous materials, operations and equipment, in particular, the use of a flow of oxygen as ageing atmosphere. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this document to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.
If there is a likelihood that more than 1 % mass fraction in a binder of volatile components is present, this procedure must not be used.

2 Normative references

The following referenced standards are indispensable for the application of this European Standard. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

EN 12607-1, *Bitumen and bituminous binders – Determination of the resistance to hardening under the influence of heat and air – Part 1: RTFOT method*

EN 12607-2, *Bitumen and bituminous binders – Determination of the resistance to hardening under the influence of heat and air – Part 2: TFOT method*

EN 14895, *Bitumen and bituminous binders – Stabilisation of binder from bituminous emulsions or from cut-back and fluxed bituminous binders*

3 Terms and definitions

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

3.1
rotating cylinder ageing test
RCAT
ageing procedure performed using a rotating cylinder

3.2
short-term ageing/conditioning
conditioning a binder undergoes during the method described in EN 12607-1 and EN 12607-2 or directly in the rotating cylinder to an equivalent level of ageing

NOTE 1 Short-term ageing/conditioning simulates the hardening which a bituminous binder undergoes during the mixing in an asphalt mixing plant

NOTE 2 See Annex A

3.3
stabilisation
conditioning of the binders from bituminous emulsions, cut-back or fluxed bitumen to produce residual binders to further determine their characteristics

NOTE E.g. the procedure described in EN 14895

3.4
long-term ageing
ageing that the binder undergoes during its service life

3.5
accelerated long-term ageing conditioning
simulated long-term ageing that the binder undergoes during the accelerated rotating cylinder ageing procedure.

NOTE 1 In the case of hot-mix asphalt binders the long-term ageing is carried out on binders that have already been conditioned through short-term ageing/conditioning.

NOTE 2 In the case of bituminous emulsions or cut-back or fluxed bitumen the long-term ageing is carried out on binders that have already been conditioned through the stabilisation procedure

3.6
mastic
homogenous mixture of filler and bituminous binder

NOTE 1 The mixture can be prepared directly in the rotating cylinder

NOTE 2 See Annex B

4 Principle

A rotating film of binder is heated to a specified temperature under a specified rotation speed and under a specified oxygen flow for a given period of time. This is to simulate the changes that occur to the binder during service (pavement or other bituminous applications, e.g. roofing).The standard procedure is RCAT90 as described in Subclause 6.4. For other purposes, e.g. research, other conditions may be used.

The effects of this ageing procedure are evaluated on the residual binder after the test. However, as samples can be taken at intermediate exposure times, the ageing process can be monitored on the basis of a kinetic approach.

NOTE Ageing of binders during service is affected by ambient temperature and air pressure as well as by mixture-associated variables such as volumetric mixture proportions, mixture permeability, aggregate properties and other factors. This procedure is intended to provide an evaluation of the relative ageing behaviour of binders under specified conditions, but cannot account entirely for mixture variables or provide relative resistance to ageing at in-service conditions.

5 Apparatus

Usual laboratory apparatus and glassware, together with the following:

5.1 Test system comprised of a cylinder with a grooved inner roller, an oxygen supply system with flow control devices, a forced–draft oven equipped with an appropriate outlet for the evacuation of volatile

components and placed under an appropriate hood, temperature control/measuring devices, a temperature measuring apparatus and preferably, a temperature recording device (Figure 1).

5.2 Testing cylinder of stainless steel construction (Figure 2), with a volume of 3,7 l. The cylinder shall be closed at one end and be fitted with a removable screw-on closure at the other. An effective seal is provided by a heat- and solvent-resistant flat rubber gasket. The removable closure has a central orifice 42 mm to 43 mm in diameter through which it is possible to take small test samples at predetermined intervals ("exposure times"). During testing this opening is fitted with a PTFE plug (type 1) as illustrated in Figure 3. A long stainless steel tube is inserted through the opening in this plug to provide an oxygen atmosphere (Figure 4).

5.3 Grooved solid stainless steel roller 34 mm in diameter (Figure 2) making a gravity-induced rotating movement about its axis inside the rotating cylinder. This roller is fitted at both ends with a rim 3,5 mm thick and 40 mm in diameter. Using this roller, the binder in the cylinder is constantly pressed and distributed against the inner wall of the cylinder. As a result, the bitumen surface exposed to oxygen is constantly renewed and remixed with the bulk of the mass of binder.

5.4 Drive mechanism rotating the testing cylinder about its axis on two round drive bars in a ventilated oven. The mechanism is such that the cylinder makes one revolution per minute (1 ± 0,05) r/min.

5.5 Forced-draft oven to be used with 5.2, 5.3 and 5.4, capable of bringing the oven temperature to the desired ageing temperature ± 0,5 °C, as recorded by a suitable thermometer, within 1 h and maintaining the temperature of the binder inside the cylinder at the ageing temperature ± 0,5 °C. The oven is thermostatically controllable by a control and measuring device with a reading to 0,1 °C. The oven shall have interior dimensions 480 mm x 380 mm x 500 mm (width x height x depth) ± 20 mm and a protected cylindrical fan that is able to efficiently homogenize the temperature of the air. The temperature in the oven shall be set very accurately (between 70 °C and 95 °C, depending on the test). It must remain constant to within ± 0,5 °C during the procedure.

SAFETY PRECAUTIONS — The ageing procedure operates at high temperatures and with a slow flow of oxygen. All safety guidelines issued by the equipment manufacturer must be followed.

5.6 Pressure reducer fitted with a pressure gauge adjustable between 0 MPa and 0,5 MPa (5 bar) with 0,01 MPa subdivisions. If possible, the pressure reducer should be fitted with a sensitive excess flow valve. Otherwise, a general control needle valve shall be used to limit oxygen flow to approximately 10 l/h in case of a break in the oxygen line.

5.7 Oxygen temperature safety break valve which prevents oxygen flow when the temperature in the oven exceeds 105 °C during the ageing procedure when operating with oxygen.

5.8 Oxygen flow meter that can be adjusted over a measuring range of 0 l/h to 10 l/h with 0,5 l/h subdivisions. The rate of this flow shall be (4,5 ± 0,5) l/h.

5.9 Spiral-shaped stainless steel tube (2 m to 3 m long, 6 mm to 6,35 mm in diameter, approximately 1 mm in wall thickness) to preheat the oxygen as it passes through and before it is released into the cylinder.

5.10 Temperature probe accurate to 0,1 °C, for measuring the temperature inside the cylinder when calibrating the device (Annex C). The thermometer shall be calibrated to an accuracy of ± 0,1 °C at appropriate intervals. This thermometer (or a second one) may also be used to monitor the temperature of the oven.

NOTE A Resistance Thermal Detector (RTD) has been found to be suitable.

5.11 Temperature recording device with a data acquisition system, capable of recording the temperature throughout the test to 0,1 °C.

5.12 Balance capable of weighing 10,0 kg to an accuracy of ± 0,1 g.

5.13 Multi purpose oven(s) capable of maintaining a temperature up to 200 °C with an accuracy of ± 5 °C.

5.14 Sampling spoon (Figure 5) to take samples of the aged binder at predetermined exposure times, with a view to monitoring the ageing process.

NOTE Exposure times are generally 17 h, 65 h and 140 h when ageing is performed at 90 °C (to comply with normal working hours, long-term ageing conditioning is best started between 3 h and 5 h in the afternoon according to the longest procedure involved).

5.15 Commercial bottled oxygen. A sufficient supply of pressurised oxygen shall be available to carry out the procedure; commercially available bottled oxygen is suitable for this. Purity: ≥ 99,9 volume %.

6 Procedure

SAFETY PRECAUTIONS — Use laboratory safety procedures in handling the hot asphalt binder when preparing the specimens and removing the residue from the cylinder.

6.1 Ageing of mastics

When RCAT ageing is performed on a mastic, the mastic is prepared directly in the cylinder as described in Annex B. After mixing, a sample of approximately 25 g is taken for characterization. After that, apply the procedure detailed in Subclauses 6.2 (RCAT163 conditioning) or 6.4 (where the word “binder” is replaced by “mastic”).

6.2 Preconditioning

The binder is first preconditioned as necessary to simulate the condition in which it would be applied to the road. For binders to be used in hot asphalt applications the preconditioning of the sample would typically be by one of the methods in EN 12607-1 or EN 12607-2. Short-term ageing/conditioning can also be performed directly in the RCAT according to the procedure RCAT163 described in Annex A. If possible, this method should be used, as the two ageing procedures are then performed in the same device. The procedure used shall be stated in the report. For reference purposes, RTFOT procedure according to EN 12607-1 is used.

As it is necessary to prepare more than one set of samples in the short-term ageing/conditioning or stabilisation procedure (either EN 12607-1, EN 12607-2 or EN 14895), it is permissible to combine and homogenise sub-samples to obtain a sufficient quantity (550 g to 600 g) for the RCAT procedure. All of the residue shall be combined and any supplementary heating to homogenise the residue shall be kept to a minimum. Therefore, short-term ageing/conditioning shall be performed by preference directly in the RCAT device (Annex A) and the long-term ageing may then be applied immediately after cooling to less than 5 °C above the test temperature.

If EN 12607-1 or EN 12607-2 is used, the hot residue may be poured immediately into the RCAT cylinder or allowed to cool for RCAT testing later. If allowed to cool, the container with the binder shall be covered and stored at ambient temperature. When it is to be used, the sample shall be re-heated as in Subclause 6.3. In any event, heating shall be carried out for the minimum possible time and the sample is stirred prior to pouring into the cylinder.

6.3 Preparing the binder

The day before the test, heat the ageing oven as well as the cylinder and the roller to the chosen test temperature: typically 90 °C (RCAT90). If another long-term ageing test temperature is chosen, all figures referring to 90 °C shall be modified accordingly.

If the cylinder and the roller can be preheated in a separate oven, heating of the ageing device may be switched on just before preparing the binder as described hereafter.

Preparation of the laboratory sample to be aged shall be performed starting with a divided sample or with a recovered preconditioned sample. The sample with a mass of approximately 650 g to a maximum of 900 g shall be in a metallic container.

On the starting day of the ageing procedure prepare the binder sample according to EN 12594.

NOTE 1 When the longest procedure is involved (i.e. preparing a mastic + short-term ageing by RCAT163) it is advised to attempt to reduce the preparation time of the laboratory sample to remain within normal working hours. As a guideline, the use of a forced-draft oven is recommended with a preparation time of less than 100 min.

HPMB and (very) high viscous binders may produce difficulties for long-term ageing using the RCAT. As a guideline, the test temperature shall be at least 10 °C higher than the initial softening point R&B of the sample to be tested. The highest allowed test temperature is 100 °C for RCAT long-term ageing using oxygen.

When preparation of the test sample involves oven temperatures higher than 170 °C, preheating the cylinder and roller shall be reduced.

NOTE 2 As a guideline, a decrease of 2 °C (with respect to 90 °C) in the preheating temperature of the cylinder and the roller may be suggested for each 10 °C above 170 °C (taking into account that the mass of the cylinder and the roller is approximately 8 500 g).

Heating time may be adapted where appropriate as suggested by experience gained with the bituminous binder to be investigated and with the oven used. Preheating shall be kept as short as possible while still allowing homogenisation and pouring.

After preheating, stir the binder thoroughly to homogenize it and pour 525 g to 550 g into the preheated cylinder placed on a balance (protect plate from heat with an appropriate item, e.g. a thick cork plate or support). By preference pouring should be done via the opening in the removable end. Then insert the preheated roller into the cylinder while making sure that the threaded screw hole for its extraction faces the outside.

At the same time, while the sample is still hot take the necessary portions from the remaining binder in the container to perform the identification tests at exposure time t = 0 h.

All preparation, handling and conditioning (i.e. heating time, preheating of the cylinder and the roller, and cooling period if necessary) shall be conducted in such a manner to begin the procedure described in 6.4 with a sample which has a temperature within 5 °C of the test temperature.

6.4 Performing the long-term ageing procedure

The standard procedure RCAT90 is performed as follows. Lay the cylinder on the drive mechanism in the ageing oven, place the PTFE plug type 1 (Figure 3) and fasten the oxygen feed tube with a wing nut so it is centred in the opening of the cylinder. Allow the cylinder to lie for (60 ± 5) min without rotation or inflow of oxygen, to enable the binder to reach the test temperature. Adjust the pressure gauge of the gas bottle that supplies the oxygen to 0,1 MPa (1 bar).

Start the cylinder's rotation (1 r/min) and open the oxygen feed (time t = 0). Adjust the rate of flow to (4,5 ± 0,5) l/h using the flow meter of the ageing oven. The oxygen is preheated as it passes through the spiral-shaped steel tube.

At predetermined exposure times (typically 17 h and 65 h), remove the feed tube and PTFE plug and take a portion of approximately 25 g to 30 g of the sample from the cylinder using the sampling spoon, with a view to monitoring the ageing process. Replace all items and continue the procedure.

NOTE Sampling is easier when the sampling spoon is preheated 10 °C to 20 °C above test temperature.

At this stage the total mass of removed samples shall not exceed 120 g. Maintain the temperature and oxygen flow for 140 h (± 15 min).

If the temperature indicated by the temperature-recording device is above or below the set ageing temperature by more than 0,5 °C for more than 5 h during the ageing procedure (except during sampling periods), the procedure shall be terminated and the samples discarded.

6.5 Recovering the aged binder

At ageing time completion (140 h ± 15 min), stop heating and oxygen flow. Remove the cylinder from the oven. Unscrew the top disc, remove the inner roller and place it on an inverted metal lid to contain any spillage of the binder. To empty the open cylinder, turn it upside down and place it with the opening down on a 0,5 l metal box against the inner wall of an oven preheated to (160 ± 5) °C (Figure 4). Allow to stand for:

- 30 min to 35 min for penetration grade bitumen and for residue from emulsion or cut-back;
- 40 min to 45 min for binders containing polymers or for rubber-bitumen.

If it is felt necessary a moderate scraping of the binder from the cylinder or from the inner roller is permissible.

Take the necessary samples after stirring to characterize the binder subjected to 140 h of ageing.

If the RCAT residue testing does not take place immediately after the ageing procedure, distribute the recovered binder while still hot in several sample containers (according to quantity necessary for the intended test). The sample containers shall be allowed to cool and be sealed and stored at ambient temperature.

NOTE The development in time of various characteristics of the binder, e.g. penetration, softening point Ring-and-Ball, can be monitored. When measuring penetration, a “mini-penetration” facility (metal can: diameter 36 mm and height 20 mm or 25 mm) may be used.

7 Precision

This document describes a conditioning procedure that has no result in itself. To obtain information on repeatability and reproducibility of the procedure, Round Robin Tests can be conducted measuring some usual characteristics such as penetration, softening point, viscosity and others.

A Round Robin Test involving five laboratories and three binders (pure bitumen 50/70, SBS [Styrene-Butadiene-Styrene] PMB and EVA [Ethylene-Vinyl-Acetate] PMB) has been conducted with the RCAT procedure. Monitored characteristics were penetration and softening point R&B for exposure times of 0 h, 17 h, 65 h and 140 h at 90 °C. This limited Round Robin Test was based on single tests per laboratory and so no indication of “repeatability” can be given, although it would be expected to be better than the reproducibility levels that are discussed. These reproducibility figures are therefore, lower bound values and if by accident the repeatability is high, reproducibility figures will be significantly higher than those quoted. This limited Round Robin Test showed that the standard deviation of reproducibility (s_R) is almost unaffected by the ageing procedure, except in case of penetration for the EVA PMB. The mean value of s_R for the four levels (0 and the three ageing times) has been taken into account for the evaluation of reproducibility (R) for penetration and softening point R&B.

As an outcome of this preliminary Round Robin Test, the difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the