SLOVENSKI STANDARD
oSIST prEN 14769:2022

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Bitumen in bitumenska veziva - Pospešeno staranje v tlačni posodi (PAV)

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

Bitumen und bitumenhaltige Bindemittel - Beschleunigte Langzeit-Alterung mit einem Druckalterungsbehälter (PAV)

Bitumes et liants bitumineux - Vieillissement long-terme accéléré réalisé dans un récipient de vieillissement sous pression (PAV)

Ta slovenski standard je istoveten z: **prEN 14769**

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 STANDARD

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

Bitumes et liants bitumineux - Vieillissement long-terme accéléré réalisé dans un récipient de vieillissement sous pression (PAV)

Bitumen und bitumenhaltige Bindemittel - Beschleunigte Langzeit-Alterung mit einem Druckalterungsbehälter (PAV)

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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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

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European foreword

This document (prEN 14769:2022) 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.

This document will supersede EN 14769:2012.

In comparison with the previous edition, the following technical modifications have been made:

a) Terms and definition: update of 3.2, 3.3 and addition of 3.4;

b) Apparatus: 5.1.3 and 5.1.1 merged; 5.3 updated; 5.9 and 5.10 added;

c) Procedure: updated in order to ease understanding;

d) Report updated;

e) Figure 1: key updated;

f) Figure 2: updated.

1 Scope

This document specifies an accelerated ageing/conditioning procedure for bituminous binders. The procedure involves ageing trays of binder at elevated temperatures under pressurized conditions in a pressure ageing vessel (PAV).

NOTE For binders to be used in hot asphalt applications, the pre-conditioning of the sample can be performed using one of the methods in the EN 12607 series. For binders to be used in bituminous emulsion and cut-back or fluxed applications, the stabilizing of the sample is such that there are no volatiles remaining.

WARNING — The use of this document can involve hazardous materials, operations and equipment, in particular, the use of a high pressure ageing vessel. 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 the likelihood of volatile components being present in a binder, this procedure is not used.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 12594, *Bitumen and bituminous binders - Preparation of test samples*

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

EN 12607-2:2014, *Bitumen and bituminous binders - Determination of the resistance to hardening under influence of heat and air - Part 2: TFOT method*

EN 12607-3, *Bitumen and bituminous binders - Determination of the resistance to hardening under influence of heat and air - Part 3: RFT method*

EN 13074-2, *Bitumen and bituminous binders - Recovery of binder from bituminous emulsion or cut-back or fluxed bituminous binders - Part 2: Stabilization after recovery by evaporation*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at https://www.electropedia.org/
- ISO Online browsing platform: available at https://www.iso.org/obp

3.1
short-term ageing
ageing that the binder experiences during the production, laying and compaction of asphalt mixtures

3.2
long-term ageing
ageing that the binder experiences during its service life in asphalt pavements

3.3
stabilization
conditioning of the binders from bituminous emulsions, cut-back or fluxed bitumens to produce residual binders for further determination of their characteristics

Note 1 to entry: The conditioning procedure is described in EN 13074-2.

3.4
short-term ageing conditioning
conditioning that the binder experiences during the method according to EN 12607-1, EN 12607-2 or EN 12607-3

3.5
accelerated long-term ageing conditioning
simulated long-term ageing that the binder experiences during the ageing procedure described in this test standard; this procedure is called "accelerated" because the ageing of the binder during long-term service life is speeded up for laboratory purposes by use of high pressure and high temperature

Note 1 to entry: In the case of hot mix asphalt binders, the long-term ageing conditioning is carried out on binders that have already been conditioned through short-term ageing conditioning procedures (see 3.4).

Note 2 to entry: In the case of bituminous emulsion, cut-back and fluxed bitumens, the long-term ageing conditioning is carried out on binders after a stabilisation procedure (see 3.3).

4 Principle

A static film of binder is heated to a specified temperature under a specified air pressure for a given period of time. This is done in order to simulate the changes occurring to the binder within the pavement during service life.

The effects of this ageing procedure are evaluated on the residual binder after the test.

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

5 Apparatus

Usual laboratory apparatus and glassware, together with the following:

5.1 PAV equipment, designed to operate at (2,1 ± 0,1) MPa between 80 °C and 115 °C. Either 5.1.1 or 5.1.2 is applicable (see Figure 1).

5.1.1 Pressure vessel, which shall be made from stainless steel and shall have internal dimensions adequate to contain a pan holder capable of holding a number of containers (according to requirements and dimensions given in EN 12607-2:2014, 4.3).

The bottom of the pressure vessel shall be such that the containers are held in a horizontal position with the binder evenly distributed across the diameter of the container in equal binder film thickness. A diagram showing a possible configuration of the vessel pan holder and containers and specifying dimensional requirements is shown in Figure 2.

Other pressure vessels of different internal dimensions can be used provided that the operating conditions can be satisfied. In such cases, the containers used may differ from the standard dimensions given in EN 12607-2.

The pressure vessel may be a separate unit to be placed in a forced draft oven or an integral part with a temperature control system.

The PAV equipment shall have an integrated, built-in temperature control system that is capable of:

a) bringing the loaded pressure vessel to the desired conditioning temperature ± 1,0 °C, as recorded by the temperature measuring device inside the vessel, within two hours;

b) maintaining the conditioning temperature at ± 1,0 °C.

SAFETY PRECAUTIONS — The pressure vessel operates at high temperatures and high pressures. All safety guidelines issued by equipment manufacturers shall be adhered to.

5.1.2 Forced draft oven, to be used with 5.1.1 and which is capable of:

a) bringing the loaded pressure vessel to the desired conditioning temperature ± 1,0 °C, as recorded by a suitable temperature measuring device inside the vessel, within two hours after loading the pressure vessel into the oven;

b) maintaining the conditioning temperature at ± 1,0 °C.

The oven shall have sufficiently large interior dimensions to allow forced air to freely circulate within the oven and around the pressure vessel. The oven shall contain a stand or a shelf, which supports the loaded pressure vessel in a level position above the lower surface of the oven.

5.2 Pressure controlling devices, which include the following:

5.2.1 Pressure release valve, which prevents pressure in the vessel from exceeding 2,5 MPa during the ageing procedure.

5.2.2 Pressure regulator, capable of controlling the pressure within the vessel to ± 0,1 MPa and with a capacity sufficient to reduce the pressure from the source of compressed air so that the pressure within the vessel is maintained at the operating pressure of (2,1 ± 0,1) MPa.

5.2.3 Slow release bleed valve, which allows the pressure in the vessel at the completion of the test to be reduced from 2,1 MPa (the operating pressure) to atmospheric pressure within 8 min to 15 min.

5.2.4 Pressure gauge, capable of measuring the pressure within the vessel to within 0,1 MPa during the test, and which shall be calibrated to an accuracy of ± 0,1 MPa at appropriate intervals.

5.3 Temperature measuring device, accurate to 0,1 °C for measuring the temperature inside the pressure vessel.

NOTE A resistance thermal detector (RTD) has been found to be suitable.

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

NOTE The current method of monitoring temperature consists of a computerized log of time and temperature. It is assumed that the temperature recorded is the temperature at every area within the ageing vessel.

5.5 Metal containers (or pans), with diameters of (140 ± 1) mm, the standard which is defined in EN 12607-2:2014, 4.3.

Similar containers with other diameters may be used if their diameters are determined with the same tolerance and the amount of binder is adjusted as given in Formula (1). Weigh (50,0 ± 0,5) g in the container (nominal diameter 140 mm). If a container with another diameter is used weigh the amount given in Formula (1):

$$M = 50{,}0\ \text{g} \times (d_2 \times d_2)/(d_1 \times d_1) \pm 0{,}5\ \text{g} \quad (1)$$

where

d_1 is the normal container diameter (140 ± 1) mm

d_2 is the actual container diameter in millimetres with a tolerance of ± 1 mm

M is the mass of binder at ambient temperature for the actual container

5.6 Balance, which shall be capable of weighing to an accuracy of ± 0,1 g.

5.7 Vacuum oven (optional), which shall be capable of maintaining a temperature up to 180 °C with an accuracy of ± 5 °C.

The vacuum system shall be capable of maintaining pressures below (15,0 ± 2,5) kPa absolute. Alternatively, the pressure vessel itself may be used instead of the vacuum oven if it is capable of maintaining a temperature of 180 °C and a reduced pressure of (15,0 ± 2,5) kPa.

5.8 Commercial bottled compressed air, which is suitable for the purpose of providing a sufficient supply of pressurized air to carry out the test. Only bottled air is suitable.

5.9 Ventilated oven to pre heat pan holder, if needed, with a temperature accuracy of ± 5 °C, checked at midpoint of the working space at suitable intervals.

5.10 Pan holder, capable of holding all metal containers/pans in a horizontal position within the pressure vessel.

NOTE 1 Most pan holder assemblies hold up to 10 containers.

NOTE 2 Depending on the individual PAV equipment used, e.g. of heat content/capacity of the pan holder, it can be necessary to pre heat the pan holder before placing into the vessel, e.g. to reach an even binder film thickness, or to avoid drop in temperature before start of the conditioning.

6 Procedure

6.1 General

SAFETY PRECAUTIONS — Use laboratory safety procedures when handling the hot bituminous binders and preparing the specimens and when removing the residue from the pressure vessel. Take particular care when lifting the lid of the pressure vessel.

The binder is firstly pre-conditioned as necessary, prior to the long-term ageing conditioning procedure. The procedure used shall be stated in the report.

The chemistry of bituminous binder ageing is complex and the mechanism changes with increasing temperature. For this reason, the conditioning temperature should be kept as low as possible. High temperatures may make this ageing procedure unrepresentative of field ageing. The 110 °C condition should be limited to conditioning of binders for use in high ambient temperature (desert) areas.

Binders containing polymers could exhibit separation and/or degradation in a way that does not occur during natural ageing.

NOTE 1 Typical conditioning temperatures are 85 °C, 90 °C, 100 °C and 110 °C. Typical ageing times found to be suitable are 20 h ± 10 min for the 90 °C, 100 °C and 110 °C temperature conditions, and 65 h ± 30 min for the 85 °C temperature condition.

NOTE 2 Several temperatures are currently used for the accelerated long-term ageing conditioning procedure. No correlation of a particular temperature with performance has been established at this time.

6.2 Preparation of binder

If it is necessary to prepare more than one set of samples in the short-term ageing/conditioning procedure (using either EN 12607 series) or a stabilisation procedure (using EN 13074-2) in order to obtain sufficient quantity for this procedure, sub-samples shall be combined and homogenised before the test. The short-term ageing or stabilisation procedure shall be stated in the report.

The hot residue from short-term ageing/conditioning or stabilisation procedure may, after combination of all residual binder as described in the respective part of the EN 12607 series or EN 13074-2 respectively, be

a) poured immediately into the PAV container for immediate PAV ageing, or

b) poured immediately into the PAV container and allowed to cool for PAV ageing later; the container with the binder should then be covered and stored at ambient temperature. When it is to be used, the sample should be re-heated in accordance with EN 12594, or

c) stored in a separate sampling container, which should then be covered and stored at ambient temperature. When it is to be used, the sample should be re-heated in accordance with EN 12594 for the minimum possible time and stirred for homogenisation prior to pouring into the pans.

NOTE Subsection c) is also applicable if pre-treated binders are received from other laboratories.

If change of mass after PAV ageing should be addressed, e.g. for research purposes, the procedures described in EN 12607 series should be applied.

6.3 Preparation of pressure vessel

Pre-heat the pressure vessel or forced draft oven to the ageing temperature.

NOTE 1 Pre-heating the vessel up to 15 °C above the conditioning temperature can be used to reduce the drop in PAV temperature during the loading process and minimise the time required for stabilisation of the set temperature after loading. However, pressurising the vessel causes an increase in temperature and the vessel might cool slowly, making it difficult to achieve the specified temperature at the start of the test.

Place the pan holder inside the heated pressure vessel and set the ageing temperature to the specified temperature.

NOTE 2 Alternatively, pre-heating of the pan holder can be done in a ventilated oven (see 5.9), in order to avoid opening the pressure vessel twice and thus temperature loss.

6.4 Filling of containers

Pre-heat the containers to the PAV conditioning temperature. The heating of the containers should be sufficient to enable the flow of the binder.

Place the pre-heated container on a balance and pour (50,0 ± 0,5) g of the pre-treated binder (see 6.1) into the container. Ensure that the binder is distributed over the entire base of the container by e.g. tilting or carefully heating the filled container (not above the pouring temperature of the binder and for the