SLOVENSKI STANDARD
oSIST prEN 12594:2023

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Bitumen in bitumenska veziva - Priprava preskusnih vzorcev

Bitumen and bituminous binders - Preparation of test samples

Bitumen und bitumenhaltige Bindemittel - Vorbereitung von Untersuchungsproben

Bitumes et liants bitumineux - Préparation des échantillons d'essai

Ta slovenski standard je istoveten z: **prEN 12594**

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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ICS

Will supersede EN 12594:2014

English Version

Bitumen and bituminous binders - Preparation of test samples

Bitumes et liants bitumineux - Préparation des échantillons d'essai

Bitumen und bitumenhaltige Bindemittel - Vorbereitung von Untersuchungsproben

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 336.

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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 12594:2023) 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 12594:2014.

In comparison with the previous edition, the main technical changes are:

— definitions of laboratory sample and test sample have been reviewed;

— definitions of test specimen and of melting have been added;

— reference to most reageants and materials have been deleted as already covered in other standards;

— description of apparatus has been completed;

— procedure in paragraph 7 has been modified to ensure consistency with test standards and to better precise conditions in particular heating time and heating temperatures;

— recommendation on storage of emulsions have been added.

1 Scope

This document specifies a method for preparing samples of bitumen and bituminous binders in order to test their properties.

WARNING — The use of this document can involve hazardous materials, operations and equipment. 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 safety and health practices and to determine the applicability of regulatory limitations prior to use.

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 58, *Bitumen and bituminous binders - Sampling bituminous binders*

EN 1425, *Bitumen and bituminous binders - Characterization of perceptible properties*

EN 1427, *Bitumen and bituminous binders - Determination of the softening point - Ring and Ball method*

EN 1429:2013, *Bitumen and bituminous binders - Determination of residue on sieving of bituminous emulsions, and determination of storage stability by sieving*

EN 12847, *Bitumen and bituminous binders - Determination of settling tendency of bituminous emulsions*

EN ISO 3696:1995, *Water for analytical laboratory use - Specification and test methods (ISO 3696:1987)*

ISO 565:1990, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

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:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org

3.1
laboratory sample
sample of bitumen or bituminous binder incoming into laboratory for tests

Note 1 to entry: Sampling is conducted according to EN 58.

Note 2 to entry: Incoming material containing bitumen or bituminous binders, e.g. asphalt mixtures, are taken according to other procedures than the ones described in EN 58; therefore, they are here not used with the term laboratory sample.

Note 3 to entry: The laboratory sample may be a spot sample, a composite sample, or a part thereof (a divided sample).

3.2
test sample
sample of bituminous binder produced by treatment or subdivision of a laboratory sample for individual testing

Note 1 to entry: the same test sample can be treated to prepare various tests in parallel; some test methods need preparation of test specimen, while for others a portion of the test sample will be used directly.

3.3
test specimen
specimen for specific tests prepared from a test sample

Note 1 to entry: In principle, details on the test specimen preparation are described in the respective test standards, where applicable, and are not within the scope of EN 12594. However, EN 12594 is covering common areas like temperatures or duration of preparation.

3.4
melting
heating the sample to lower its viscosity and ensure homogeneity while preparing test samples and test specimen

4 Principle

Incoming laboratory samples are prepared for testing depending on their types and their sizes, in as-is state.

5 Reagents and materials

Only reagents of recognized standard analytical grade and water conforming to grade 3 of EN ISO 3696:1995 shall be used.

6 Apparatus

6.1 General

Usual laboratory apparatus and glassware, together with the following.

6.2 Ventilated laboratory oven with a maximum permissible measurement error of ± 5 °C, checked at midpoint and working space at suitable intervals.

6.3 Indirect heating apparatus, e.g. heating jacket, oil bath with thermometer or equivalent

6.4 Container of appropriate materials (i.e heat resistant and not interacting with the sample) to heat the sample.

6.5 Lid or aluminium foil for container (6.4).

6.6 Any appropriate stirrer, e.g. manual (such as spatula), mechanical equipped with a propeller or magnetic.

6.7 Metal sieve, mesh size 0,500 mm, as described in ISO 565:1990.

6.8 Test sample container of appropriate material with a lid or other closure, or glass conical flask with a ground glass stopper.

NOTE Test sample containers can be the one originally used to sample the bituminous binder according to EN 58.

6.9 Test containers or moulds for test specimen preparation, as described in the respective test standards, into which the test sample is transferred prior to testing.

NOTE It may be that the test sample is not stored in a test sample container but directly transferred into moulds or test containers.

7 Procedure for preparation of test samples

7.1 General

In order to produce the laboratory sample, the material shall have been sampled in accordance with EN 58.

Perceptible properties of the laboratory sample shall be checked in accordance with EN 1425, prior to sample preparation according to this document.

7.2 Solid or semi-solid samples

7.2.1 Samples up to one litre

Ease the lid or other closure of the container (6.4) and place the container with the lid loose in the oven (6.2). The temperature of the oven shall be set at not more than (85 ± 5) °C above the expected softening point as defined in EN 1427; this includes the maximum permissible measurement error described in 6.2.

For modified bitumen, use the procedure provided by the supplier. If no guidance is provided, for polymer modified bitumen complying with EN 14023, the temperature of the oven shall be set at (190 ± 5) °C, irrespective from the softening point; this includes the maximum permissible measurement error described in 6.2. If the times given below are not sufficient to melt the polymer modified bitumen sample completely, then place the sample container into an indirect heating apparatus (6.3) and continue the heating of the sample under continuous stirring until the sample is homogeneous and pourable. In any case, 200 °C shall not be exceeded.

Remove the container from the oven and stir (6.6) the heated sample with care in order to avoid incorporating air bubbles into the sample. For modified binders, stir according to handling suggestion from supplier, when available. Allow any air bubbles to escape, if necessary, by placing the sample in the oven for not more than 5 min. Pour the liquefied and homogenized sample into the moulds or test sample containers. Prepare all test samples that are required for one property at the same time.

The entire procedure (heating, homogenizing and moulding) shall conform to following requirements:

— < 50 g: max. 15 min;

— 50 g to 100 g: max 30 min;

— 100 g to 500 g: max 1 h;

— 500 g to 1 kg: max 2 h.

If the sample contains particles eg coke or detritus, it can be sieved through a warm sieve (6.7) before collecting the test sample. The particles and sieving shall be mentioned in the test report under g) and h) (see Clause 8).

Do not reuse the sample for other tests later.

7.2.2 Samples greater than one litre

If division of a sub-sample is necessary, ensure that the sub-sample is representative of the laboratory sample. If necessary, take a sufficient amount of material (100 g minimum) from the container and transfer the material into another container (6.4) with a loose lid. This procedure is not valid for polymer modified bitumen according to EN 14023 which needs to be melted and homogenized following the procedure described below before a sub-sample can be taken.

Place the container in the appropriate heating device (6.2 or 6.3).

Melt the material at a maximum temperature not more than (85 ± 5) °C above the expected softening point as defined in EN 1427. For modified bitumen, follow the procedure provided by the supplier. If no other guidance is provided by the supplier for polymer modified bitumen according to EN 14023, the temperature of the oven shall be set at (190 ± 5) °C, irrespective from the softening point; this includes the maximum permissible measurement error described in 6.2. In any case, 200 °C shall not be exceeded.

During melting and when possible, depending on its viscosity, stir the molten sample periodically with care in order to ensure homogeneity, to prevent local overheating and avoid incorporating air bubbles into the sample. For modified binders, stir according to the handling guidance provided by the supplier, if available. If the handling guidance is unavailable, modified bitumen shall be homogenized for up to 5 min, avoiding incorporating air bubbles into the sample. Cover the container with aluminium foil or with a loose fitting lid.

The whole sample shall be heated as follows:

— 1 l to 2 l: max 3 h;

— 2 l to 3 l: max 3 h and 30 min;

— 3 l to 5 l: max 4 h;

— more than 5 l: overnight.

For samples larger than 5 l, the melting temperature of the material should be 50 °C above the expected softening point. As melting overnight is performed at a lower temperature, the temperature will be increased approximately 2 h before starting the sampling.

Pour the liquefied and homogenized sample into the moulds or test sample containers. Prepare all test samples that are required for one property at the same time.

Carry out the homogenizing procedure and the pouring procedure within 10 min maximum.

If the sample contains particles eg coke or detritus, it can be sieved through a warm sieve (6.7) before collecting the test sample. The particles and sieving shall be mentioned in the test report under g) and h) (see Clause 8).

For quality control purposes, samples should only be heated once. For other purposes, in case of a large laboratory sample, it can be useful to divide it into smaller fractions of test samples to be used at later stage. In that case, test samples should not be reheated more than once.

7.3 Soft bitumen, fluxed or cut-back binders

To minimize the loss of volatiles during heating, cover the container with aluminium foil or a loose fitting lid. Homogenize the entire sample by gently stirring manually.

If the viscosity is too high, the sample shall be heated with care in a ventilated oven, oil bath or equivalent for the minimum time required until it becomes sufficiently fluid to pour.

The laboratory sample shall be placed in an oven maintained at a temperature that should not exceed the following temperatures:

— for soft bitumen, according to EN 12591, which are specified by viscosity (V grades):

  — 95°C for the softer (V1500 and V3000);

  — 115°C for the more viscous (V6000 to V12000);

— 140 °C for fluxed bituminous binders;

— 60 °C for cut-back binders.

Avoid incorporating air bubbles into the sample by allowing any air bubbles to escape.

Pour the homogeneous liquid material into the mould or test sample containers. Prepare all test samples that are required for one property at the same time.

After heating, carry out the homogenizing and the pouring procedure within 15 min maximum.

Do not reuse the sample for other tests later.

7.4 Bituminous emulsions

7.4.1 General

The interval between sampling and testing of emulsions shall be as short as possible. For referee analysis, the interval shall be less than 10 days.

If it is not possible to perform the tests within one day after sampling, the following mode should be adopted for the storage of emulsion unless specific recommendation is provided by the bituminous emulsion manufacturer:

a) storage of the samples at (50 ± 10) °C until the evening before the scheduled date of the tests;

b) the evening before the test day, condition the samples between +18 °C and +28 °C.

In case of prolonged storage of several days (but not exceeding 10 days for referee analysis), the closed container of emulsion should be regularly and gently shaken to minimize the settling effects for some emulsions. For emulsions with a well-known tendency to settle, the liquid sample should be stirred gently at approximately 50 r/min to 70 r/min, using a glass rod or palette knife until the emulsion is uniform. All precautions should be taken to minimize water loss during this operation.

7.4.2 Test samples for testing for sieve residue according to EN 1429 and settling tendency according to EN 12847

7.4.2.1 Store the laboratory sample, obtained according to 7.1, in the oven, if necessary, between 18 °C and 28 °C, or in the laboratory if the ambient temperature remains within these limits.

For emulsions that are unstable at ambient temperature and which are normally stored and applied at elevated temperatures, it is possible to store the laboratory sample at (50 ± 10) °C prior to preparing the test samples. All precautions should be taken to minimize water loss and/or skin formation during this operation. If this procedure is adopted, the laboratory sample should be adjusted to the appropriate test temperature for preparing the test sample.

7.4.2.2 Stir the liquid sample gently at approximately 50 r/min to 70 r/min, using a glass rod or palette knife until the emulsion is uniform. Avoid the entrainment of air whilst stirring and should any entrapment occur, allow air bubbles to escape. Ensure that any sediment on the bottom of the container is thoroughly dispersed. Record the ease of re-dispersion of the sediment.