BS EN 12594:2024

BSI Standards Publication

Bitumens and bituminous binders — Preparation of test samples

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National foreword

This British Standard is the UK implementation of EN 12594:2024. It supersedes BS EN 12594:2014, which is withdrawn.

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 committee manager.

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Bitumes et liants bitumineux - Préparation des échantillons d'essai

Bitumen und bitumenhaltige Bindemittel - Vorbereitung von Untersuchungsproben

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Contents

European foreword

This document (EN 12594:2024) has been prepared by Technical Committee CEN/TC 336 “Bitumens and 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of an identical text or by endorsement, at the latest by April 2025, and conflicting national standards shall be withdrawn at the latest by April 2025.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 12594:2014.

EN 12594:2024 includes the following significant changes with respect to EN 12594:2014:

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

— definitions of test specimen and 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 Clause 7 has been modified to ensure consistency with test standards and to better precise conditions, in particular heating time and heating temperatures;

— structure of 7.4 has been modified to facilitate its reading;

— recommendations on the storage of emulsions have been added.

Any feedback and questions on this document should be directed to the users’ national standards body. A complete listing of these bodies can be found on the CEN website.

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1 Scope

This document specifies methods for preparing test samples of bitumens 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 12597, *Bitumen and bituminous binders — Terminology*

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 3310-1:2016, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

3 Terms and definitions

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

ISO and IEC maintain terminology 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 taken in accordance with EN 58 and incoming into laboratory for tests

Note 1 to entry: The term “laboratory sample” does not apply to samples from material containing bitumen and bituminous binders, e.g. asphalt mixtures or cores, as they are sampled differently from those described in EN 58.

Note 2 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 produced by treatment or subdivision of a *laboratory sample* (3.1) 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* (3.2)

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* (3.2) and *test specimens* (3.3)

4 Principle

Incoming laboratory samples are prepared for testing depending on their type and their size. This includes preparation of test specimens noting that more detailed information on their preparation may also be given in test standards.

5 Reagents

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

6 Apparatus

The usual laboratory apparatus and glassware, together with the following shall be used.

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

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

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

6.4 Lid or aluminium foil for container (6.3).

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

6.6 Metal sieve, mesh size 0,500 mm, in accordance with ISO 3310-1:2016.

6.7 Test sample container of appropriate material with a lid or other closure.

NOTE Test sample container can be the one originally used to sample the bituminous binder in accordance with EN 58.

6.8 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. It may be that the test sample is not stored in a test sample container (6.7) but directly transferred into moulds or test containers.

6.9 Cooling chamber, if necessary, allowing to condition the samples between 18 °C and 28 °C.

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 (6.4) or other closure of the container (6.3) and place the container with the lid loose in the oven (6.1). The temperature of the oven shall be set at not more than (85 ± 5) °C above the expected softening point measured in accordance with EN 1427; this includes the maximum permissible measurement error described in 6.1.

For modified bitumen, use the procedure provided by the supplier. If no guidance is provided, for polymer modified bitumen complying with EN 14023 [11], 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 6.1. If the times given below are not sufficient to completely melt the polymer modified bitumen sample, then place the sample container into an indirect heating apparatus (6.2) 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.

During the melting and when possible, depending on its viscosity, stir (6.5) the molten sample periodically with care, in order to ensure homogeneity and to prevent local overheating, in such a way not to incorporate air bubbles into the sample. For modified bitumen, stir according to handling suggestion from supplier, when available. Allow any air bubble to escape, if necessary, by placing the sample in the oven (6.1) for not more than 5 min. Pour the liquefied and homogenized sample into the moulds (6.8) or test sample containers (6.7). Prepare all test samples that are required for one property at the same time.

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

— <50 g: maximum 15 min;

— 50 g to 100 g: maximum 30 min;

— >100 g to 500 g: maximum 1 h;

— >500 g to 1 kg: maximum 2 h.

If the sample contains particles e.g. coke or detritus, it can be sieved through a warm sieve (6.6) 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 reheat or reuse the sample for other tests later.

7.2.2 Samples greater than one litre

If division of a 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 using an appropriate tool such as a warm knife and transfer the material into another container (6.3) with a loose lid (6.4). This procedure is not valid for polymer modified bitumen complying with EN 14023 [11] which need to be melted and homogenized following the procedure described below before a sub-sample can be taken.

Place the container (6.3) in the appropriate heating device (6.1 or 6.2).

Melt the material at a maximum temperature not more than (85 ± 5) °C above the expected softening point measured in accordance with EN 1427; this includes the maximum permissible measurement error described in 6.1. For modified bitumen, follow the procedure provided by the supplier. If no other guidance is provided by the supplier for polymer modified bitumen complying with EN 14023 [11], the temperature of the oven (6.1) shall be set at (190 ± 5) °C, irrespective from the softening point; this includes the maximum permissible measurement error described in 6.1. In any case, 200 °C shall not be exceeded.

During the melting and when possible, depending on its viscosity, stir (6.5) the molten sample periodically with care, in order to ensure homogeneity and to prevent local overheating, in such a way not to incorporate air bubbles into the sample. For modified bitumen, 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 (6.3) with aluminium foil or with a loose fitting lid (6.4).

The whole sample shall be heated as follows:

— 1 l to 2 l: maximum 3 h;

— >2 l to 3 l: maximum 3 h and 30 min;

— >3 l to 5 l: maximum 4 h;

— >5 l: overnight with a maximum of 18 h.

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 test sample containers (6.7) or moulds (6.8). Prepare all test samples or test specimens that are required for one property at the same time.

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

If the sample contains particles, e.g. coke or detritus, it can be sieved through a warm sieve (6.6) before collecting the test sample. The particles and sieving shall be mentioned in the test report (see Clause 8) under g) and h).

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 paving grade bitumen, cut-back and fluxed bitumen

To minimize the loss of volatiles during heating, cover the container (6.3) with aluminium foil or a loose fitting lid (6.4).

If the viscosity is too high, the sample shall be heated with care in a ventilated laboratory oven (6.1) or in an indirect heating apparatus (6.2) 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 paving grade bitumen, complying with EN 12591 [2], which are specified by viscosity (V grades):

  — 95 °C for the softer (V1500 and V3900);

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

— 140 °C for fluxed bitumen;

— 60 °C for cut-back bitumen.

Homogenize the entire sample by gently manually stirring. Avoid incorporating air bubbles into the sample by allowing any air bubble to escape.

Pour the homogeneous liquid material into the mould (6.8) or test sample containers (6.7). Prepare all test samples or test specimens that are required for one property at the same time.

After heating, carry out the homogenizing and the pouring procedure within 10 min maximum.

Do not reuse the sample for other tests later.

7.4 Bituminous emulsions

7.4.1 Conditioning of the emulsions prior to preparation for testing

7.4.1.1 The interval between sampling and testing of emulsions shall be as short as possible. In case of dispute, the interval shall be less than 10 days.

7.4.1.2 If the tests are performed within one day after sampling, store the laboratory sample between 18 °C and 28 °C. Depending on the laboratory conditions, this can mean including a cooling chamber (6.9) or an oven (6.1).

7.4.1.3 If it is not possible to perform the tests within one day after sampling, the emulsion sample shall be stored as follows, unless specific recommendation is provided by the bituminous emulsion manufacturer:

a) Store the laboratory samples at (50 ± 10) °C (this includes the maximum permissible measurement error described in 6.1) until the evening before the scheduled date of the tests. In case of prolonged storage of several days (but not exceeding 10 days in case of dispute), the closed container (6.3) 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 (6.5) until the emulsion is uniform. All precautions should be taken to minimize water loss during this operation.

b) Maximum 18 hours before testing, condition the samples between 18 °C and 28 °C.

7.4.1.4 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 (this includes the maximum permissible measurement error described in 6.1) 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 shall be adjusted to the appropriate test temperature for preparing the test sample.

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

7.4.2.1 Stir the liquid sample gently at approximately 50 r/min to 70 r/min, using a glass rod or palette knife (6.5) 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 at the bottom of the container is thoroughly dispersed. Record the ease of re-dispersion of the sediment.

7.4.2.2 Transfer the stirred sample to the test sample container (6.7). Prepare all test samples or test specimens that are required for one property at the same time.

7.4.2.3 Start the residue on sieving determination in accordance with EN 1429 or the settling tendency determination in accordance with EN 12847.

7.4.3 Test samples for all tests except EN 1429 and EN 12847

7.4.3.1 Carry out the procedures described in 7.4.2.1 and 7.4.2.2.

7.4.3.2 Follow the procedure detailed in EN 1429:2013, 8.2, apart from steps that mention weighing the sieve. It may be possible to use a beaker instead of a bottle. For very viscous emulsions that are slow to pass through the sieve, tests may be continued with non-sieved emulsion, which shall be mentioned in the test report.

7.5 Test samples obtained from a specific procedure

Test samples obtained from:

a) extracting the bituminous binder according to EN 12697-1 [6], EN 12697-3 [7], EN 12697-4 [8] or any relevant standard dealing with extraction;

b) hardening or ageing procedure according to EN 12607-1 [3], EN 12607-2 [4], EN 12607-3 [5], EN 14769 [12] or any relevant standard dealing with hardening or ageing;

c) recovery and stabilization from the bituminous emulsion, cut-back or fluxed bitumen, according to EN 13074-1 [9], EN 13074-2 [10], EN 1431 [1] or any relevant standard dealing with recovery and stabilization;

shall be prepared and tested according to the respective and appropriate methods of obtaining and testing.

Where the test material is obtained in parts, the part samples from the same origin (e.g. from an ageing procedure as described in the EN 12607 series) shall be combined into one test sample. If the necessary quantity of binder cannot be received at once, additional runs of procedure(s) shall be conducted and all residues are to be combined.

If the methods of obtaining and testing do not define the conditions for preparing the test samples obtained, proceed as follows:

— the part samples shall be collected for subsequent testing in a container (6.3) and kept liquid or reheated with care and not more than once. For maximum temperatures and durations, refer to 7.2.1 or 7.2.2. In any case, 200 °C shall not be exceeded;

— modified bitumen obtained under 7.5 c) shall be prepared and tested in accordance with the relevant methods of obtaining and testing;

— prepare the test samples and test specimens, if applicable, as described in 7.2 or 7.3;

— carry out the homogenizing procedure and the pouring procedure within 15 min maximum;

— discard the residue sample that has been heated.

8 Test report

The test report shall contain at least the following information:

a) type and complete identification of the sample prepared;

b) reference to this document including its year of publication, i.e. EN 12594:2024;

c) date of sampling and date of arrival in the laboratory;

d) date of sample preparation;

e) storage temperature before preparation of the test sample;

f) temperature and heating procedure for the sample preparation;

g) any non-dispersed sediment;

h) any sieving;

i) any information relative to check perceptible properties;

j) any deviation, by agreement or otherwise, from the procedure specified;

k) any unusual features observed.

Bibliography

[1] EN 1431, *Bitumen and bituminous binders — Determination of residual binder and oil distillate from bitumen emulsions by distillation*

[2] EN 12591, *Bitumen and bituminous binders — Specifications for paving grade bitumens*

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

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

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

[6] EN 12697-1, *Bituminous mixtures — Test methods — Part 1: Soluble binder content*

[7] EN 12697-3, *Bituminous mixtures — Test methods — Part 3: Bitumen recovery: Rotary evaporator*

[8] EN 12697-4, *Bituminous mixtures — Test methods — Part 4: Bitumen recovery: Fractionating column*

[9] EN 13074-1, *Bitumen and bituminous binders — Recovery of binder from bituminous emulsion or cut-back or fluxed bituminous binders — Part 1: Recovery by evaporation*

[10] 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*

[11] EN 14023, *Bitumen and bituminous binders — Specification framework for polymer modified bitumens*

[12] EN 14769, *Bitumen and bituminous binders — Accelerated long-term ageing conditioning by a Pressure Ageing Vessel (PAV)*

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

[14] EN 13808, *Bitumen and bituminous binders — Framework for specifying cationic bituminous emulsions*

[15] EN 15322, *Bitumen and bituminous binders — Framework for specifying cut-back and fluxed bituminous binders*

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