



SLOVENSKI STANDARD

SIST EN 12594:2014

01-december-2014

Nadomešča:

SIST EN 12594:2007

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

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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

EN 12594

NORME EUROPÉENNE

EUROPÄISCHE NORM

November 2014

ICS 75.140; 91.100.50

Supersedes EN 12594:2007

English Version

Bitumen and bituminous binders - Preparation of test samples

Bitumes et liants bitumineux - Préparation des échantillons
d'essaiBitumen und bitumenhaltige Bindemittel - Vorbereitung von
Untersuchungsproben

This European Standard was approved by CEN on 16 August 2014.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

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COMITÉ EUROPÉEN DE NORMALISATION
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Foreword

This document (EN 12594:2014) 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of an identical text or by endorsement, at the latest by May 2015 and conflicting national standards shall be withdrawn at the latest by May 2015.

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

This document supersedes EN 12594:2007.

In comparison with EN 12594:2007, the following significant changes have been made:

- changed/added wording of the Warning in the Scope;
- normative reference to ISO 5280 Xylene for industrial use – specification has been deleted;
- subclause 5.3: reference to xylene deleted;
- references to hot jacket oven (subclause 6.2) and hot air bath (subclause 7.2) have been deleted;
- changed wording of first paragraph of subclause 7.1.2;
- subclause 7.1.2: time unit added for sample size 2 l to 3 l containers;
- subclause 7.2: maximum temperature for cutbacks has been changed to 60 °C;
- subclause 7.4 has been rephrased.

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, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

EN 12594:2014 (E)

1 Scope

This European Standard specifies a method for preparing samples of bituminous binders in order to test their properties.

WARNING — Use of this European Standard can involve hazardous materials, operations and equipment. This European Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this European Standard to identify the hazards and assess the risks involved in performing this test method and to implement sufficient control measures to protect individual operators (and the environment). This includes appropriate safety and health practices and determination of 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 58:2012, *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, *Bitumen and bituminous binders — Determination of residue on sieving of bituminous emulsions, and determination of storage stability by sieving*

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

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

EN 12697-1, *Bituminous mixtures — Test methods for hot mix asphalt — Part 1: Soluble binder content*

EN 12697-2, *Bituminous mixtures — Test method for hot mix asphalt — Part 2: Determination of particle size distribution*

EN 12697-4, *Bituminous mixtures — Test methods for hot mix asphalt — Part 4: Bitumen recovery: Fractionating column*

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

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*

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

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

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

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 58:2012 and the following apply.

3.1

laboratory sample

sample of bituminous binder intended for laboratory tests

Note 1 to entry: It 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

4 Principle

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

Perceptible properties of the test material shall be checked in accordance with EN 1425, prior to performing this European Standard.

Homogenize the laboratory sample before the test samples are taken.

For certain tests (7.3.2), transfer the laboratory sample directly to the test sample containers, and for other tests (7.3.3), sieve the laboratory sample prior to testing.

Store the laboratory sample from which the test samples are taken under controlled conditions. Stir gently to render it uniform and warm the sample, if necessary.

Prepare all test samples that are required for one property at the same time.

5 Reagents and materials

Use only reagents of standard analytical grade and water conforming to grade 3 of EN ISO 3696:1995.

5.1 Solution S_a

Aqueous solution with a nominal 0,01 mol/l of sodium hydroxide (NaOH) containing a nominal 1 % mass fraction sodium oleate. This solution may be used for preparing anionic emulsion samples. The actual soap solution may be used if available on site, in case of routine tests or for simplicity.

NOTE S_a stands for "anionic solution" as this solution is used for preparing anionic emulsion samples.

EN 12594:2014 (E)**5.2 Solution S_c**

Aqueous solution of nominal 0,01 mol/l of hydrochloric acid (HCl) containing a nominal 1 % mass fraction cetyltrimethylammonium bromide. This solution may be used for preparing cationic emulsion samples. The actual aqueous phase may be used if available on site, in case of routine tests or for simplicity.

NOTE S_c stands for "cationic solution" as this solution is used for preparing cationic emulsion samples.

5.3 Efficient rinsing agent, suitable solvent rinsing agents shall be used.

5.4 Ethanol, 99 % volume fraction minimum or 95 % volume fraction, denatured.

6 Apparatus

Usual laboratory apparatus and glassware, together with the following:

6.1 Ventilated oven or ventilated laboratory oven with a temperature accuracy of ± 5 °C, checked at midpoint and working space at suitable intervals.

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

6.3 Melting pan of appropriate material.

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

6.5 Metal sieve, mesh size 0,500 mm, made from brass or stainless steel if used with bitumen emulsions.

6.6 Aluminium foil or lid for melting pan.

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

7 Procedure for sample preparation**7.1 Solid or semi-solid samples****7.1.1 Samples up to one litre**

Ease the lid or other closure of the sample container and place the container with the lid loose in the oven for a maximum of 120 min at not more than 100 °C above the expected softening point as defined in EN 1427.

For modified bitumen, use the procedure provided by the supplier. If no guidance is provided for polymer modified bitumen according to EN 14023, the temperature of the oven shall be within 180 °C to 200 °C, irrespective from the softening point. If 120 min are not sufficient to melt the polymer modified bitumen sample completely, place the sample container into a heating jacket and continue the heating of the sample under continuous stirring. In any case, 200 °C shall not be exceeded.

Remove the container from the oven and stir (6.4) the melted 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.

Carry out the entire procedure (melting, homogenizing and moulding) within 135 min.

If the sample contains coke/detritus, it is allowed to sieve the sample through a warm sieve (6.5) before collecting the test sample. The coke/detritus and sieving shall be mentioned in the test report under g) and h) (see Clause 8).

Discard the residue sample that has been heated.

7.1.2 Samples greater than one litre

If division of a sub-sample is necessary, ensure that the sub-sample is representative of the bulk sample. If necessary, take a sufficient amount of material (100 g minimum) from the container by using an appropriate tool (e.g. a warmed but not glowing knife) and transfer the material into the melting pan. 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 melting pan in the appropriate heating device (6.1 or 6.2).

Melt the material by stirring (6.4) at a maximum temperature not more than 100 °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 within 180 °C to 200 °C, irrespective from the softening point. In any case, 200 °C shall not be exceeded.

Lower temperatures required at later stages shall be achieved by cooling.

The whole sample shall be melted as follows:

- for 1 l to 2 l, 3 h maximum, [standards.iteh.ai](https://standards.iteh.ai/catalog/standards/sist/ef0eac09-ea75-4d8d-bbac-78e6e0c6a954/sist-en-12594-2014)
- for 2 l to 3 l, 3 h 30 min maximum, [SIST EN 12594:2014](https://standards.iteh.ai/catalog/standards/sist/ef0eac09-ea75-4d8d-bbac-78e6e0c6a954/sist-en-12594-2014)
- for 3 l to 5 l, 4 h maximum and [78e6e0c6a954/sist-en-12594-2014](https://standards.iteh.ai/catalog/standards/sist/ef0eac09-ea75-4d8d-bbac-78e6e0c6a954/sist-en-12594-2014)
- for more than 5 l, overnight.

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

Stir the melting sample with care in order to prevent local overheating and avoid incorporating air bubbles into the sample. For a modified binder, stir according to the handling guidance provided by the supplier, if available. If the handling guidance provided by the supplier is unavailable, the modified bitumen shall be homogenized for up to 10 min, avoiding incorporating air bubbles into the sample. If necessary, cover the melting pan with aluminium foil or with a loose fitting lid.

Pour the liquefied and homogenized sample into the moulds or test sample containers.

Carry out the homogenizing procedure and the pouring procedure within 15 min maximum.

If the sample contains coke/detritus, it is allowed to sieve the sample through a warm sieve (6.5) before collecting the test sample. The coke/detritus 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.