SLOVENSKI STANDARD
oSIST prEN 12594:2013

01-april-2013

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

oSIST prEN 12594:2013 **en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

DRAFT
prEN 12594

January 2013

ICS 75.140; 91.100.50

Will supersede EN 12594:2007

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

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Foreword

This document (prEN 12594:2013) 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 12594:2007.

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 establish appropriate safety and health practices and determine 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 1429:2009, *Bitumen and bituminous binders — Determination of residue on sieving of bitumen 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 bitumen 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)*

ISO 5280, *Xylene for industrial use — Specification*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 58:2009 and the following apply.

3.1
laboratory sample
sample of bituminous binder intended for laboratory tests. 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.

Homogenise 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.

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 Xylene, grade 2 conforming to ISO 5280.

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. hot jacket oven with thermometer, 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 homogenised sample into the moulds or test sample containers.

Carry out the entire procedure (melting, homogenising 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. If a sub sample is needed the whole bulk sample need to be melted and homogenised following the procedure described below before a subsample 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,

— for 2 l to 3 l, 3h 30 maximum,

— for 3 l to 5 l, 4 h maximum and

— 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 homogenised 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 homogenised sample into the moulds or test sample containers.

Carry out the homogenising 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).

Discard the residue sample that has been heated.

7.2 Soft bitumen, fluxed or cut-back binders

If necessary, to minimise the loss of volatiles during heating, cover the container with aluminium foil or a loose fitting lid. Homogenise the entire sample by gently stirring manually.

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

The bulk binder sample shall be placed in an oven maintained at a temperature that should not exceed whichever is the lower of the following temperatures:

— 80 °C above the estimated ring and ball softening point temperature;

— 140 °C for fluxed bituminous binders;

— 80 °C for cut-backs.

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.

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

Discard the residue sample that has been heated except for cut-back binders and binders with a Ring and Ball softening point of 30 °C or less.

7.3 Bituminous emulsions

7.3.1 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.

7.3.2 Test samples for testing for sieve residue (EN 1429) and settling tendency (EN 12847)

7.3.2.1 Store the laboratory sample, obtained according to Clause 4, in the oven, if necessary, between 18 °C to 28 °C, or in the laboratory if the ambient temperature remains within these limits. Record the storage temperature of the laboratory sample.

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 (60 ± 5) °C prior to preparing the test samples. All precautions should be taken to minimise 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.

7.3.2.2 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.

7.3.2.3 Transfer the stirred sample to the test sample container.

7.3.2.4 Start the determination of residue on sieving according to EN 1429 and/or determination of settling tendency according to EN 12847.