SLOVENSKI STANDARD
oSIST prEN 12697-1:2019

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Bitumenske zmesi - Preskusne metode - 1. del: Topni delež veziva

Bituminous mixtures - Test methods - Part 1: Soluble binder content

Asphalt - Prüfverfahren - Teil 1: Löslicher Bindemittelgehalt

Mélanges bitumineux - Méthodes d'essai - Partie 1 : Teneur en bitume

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Bituminous mixtures - Test methods - Part 1: Soluble binder content

Mélanges bitumineux - Méthodes d'essai - Partie 1 : Teneur en bitume

Asphalt - Prüfverfahren - Teil 1: Löslicher Bindemittelgehalt

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If this draft becomes a European Standard, 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.

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

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Contents

European foreword

This document (prEN 12697-1:2018) has been prepared by Technical Committee CEN/TC 227 “Road materials”, the secretariat of which is held by BSI.

This document is currently submitted to the enquiry.

This document will supersede EN 12697-1:2012.

The following is a list of significant technical changes since the previous edition:

— the title no longer makes the method exclusively for hot mix asphalt;

— [ge] editorial update according to current standard template;

— [B.2.1.1.1] Acceleration amended to 25000 m/s^2 for consistency with EN 12697-3;

— [C.2.1.5] Capacity of ignitions dishes clarified: **Ignition dish**, of at least 125 mm^3 × 10^3 mm^3 capacity ;

A list of all parts in the EN 12697 series can be found on the CEN website

WARNING — The method described in this European Standard may require the use of dichloromethane (methylene chloride), 1,1,1-trichloroethane, benzene, trichloroethylene, xylene, toluene, perchloroethylene (tetracloroethylene) or other solvents capable of dissolving bitumen. These solvents are hazardous to health and are subject to occupational exposure limits as detailed in relevant legislation and regulations.

Exposure levels are related to both handling procedures and ventilation provision and it is emphasized that adequate training should be given to staff employed in the usage of these substances.

Introduction

This European Standard describes a unified approach to the examination of bituminous mixtures that allows some divergence in the detail of procedures followed by individual laboratories. In Clause 5 of this European Standard, a description is given of the basic operations that together form the test method for the proper determination of the binder content of bituminous mixtures. Guidance on the test method is given in Annex A and Figure A.1, while the use of alternative items of equipment that are equally suitable for carrying out particular parts of the test method are described in Annex B. Although the apparatus specified for the separation of mineral filler from the binder solution obtained after extraction is of a suitably efficient level not to affect the precision of the test described in Clause 8, a method for determining the amount of residual mineral matter in the extract is given in Annex C for use in those particular cases where some doubt may exist.

Methods and equipment other than those described in Annex B and Annex C, including automated equipment, are permissible provided that they have been demonstrated to provide the same results as one of the methods in Annex B or Annex C within the limits of the precision given in this document. Guidance on determination of soluble binder content of mixtures with polymer-modified binders is given in Annex D.

1 Scope

This document describes test methods for the determination of the soluble binder content of samples of bituminous mixtures.

The test methods described are suitable for quality control purposes during the production of plant mix and for checking compliance with a product specification.

For the analysis of mixtures containing modified binders, the guidance of Annex D should be followed.

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 12697-3, *Bituminous mixtures - Test methods for hot mix asphalt - Part 3: Bitumen recovery: Rotary evaporator*

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

EN 12697-14, *Bituminous mixtures - Test methods for hot mix asphalt - Part 14: Water content*

EN 12697-28, *Bituminous mixtures - Test methods for hot mix asphalt - Part 28: Preparation of samples for determining binder content, water content and grading*

EN 933-1, *Tests for geometrical properties of aggregates - Part 1: Determination of particle size distribution - Sieving method*

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

ISO 3310-2, *Test sieves — Technical requirements and testing — Part 2: Test sieves of perforated metal plate*

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 http://www.electropedia.org/
- ISO Online browsing platform: available at http://www.iso.org/obp

3.1
soluble binder content
percentage by mass of extractable binder in an anhydrous sample, determined by extracting the binder from the sample

Note 1 to entry: Extraction may be followed by binder recovery.

3.2
insoluble binder content
percentage by mass of binder that adheres to the aggregate particles after extraction

3.3
precision
closeness of agreement between independent test results obtained under stipulated conditions

Note 1 to entry: Precision depends only on the distribution of random errors and does not relate to the true value or the specified value.

Note 2 to entry: The measure of precision is usually expressed in terms of imprecision and computed as a standard deviation of the test results. Less precision is indicated by a larger standard deviation.

Note 3 to entry: "Independent test results" means results obtained in a manner not influenced by any previous result on the same or similar test sample. Quantitative measures of precision depend critically on the stipulated conditions. Repeatability and reproducibility conditions are particular sets of extreme conditions.

3.4
repeatability
precision under repeatability conditions

3.5
repeatability conditions
conditions in which independent test results are obtained with the same method on identical test items in the same laboratory by the same operator using the same equipment within short intervals of time

3.6
repeatability limit
maximum absolute difference between two test results obtained under repeatability conditions that may be expected with a probability of 95 %

Note 1 to entry: The symbol used for repeatability limit is *r*.

3.7
reproducibility
precision under reproducibility conditions

3.8
reproducibility conditions
conditions in which test results are obtained with the same method on identical test items in different laboratories with different operators using different equipment

3.9
reproducibility limit
maximum absolute difference between two test results obtained under re…bility conditions that may be expected with a probability of 95 %

Note 1 to entry: The symbol used for reproducibility limit is *R*.

3.10
single test result
value obtained by applying the standard test method once, fully, on a single specimen

Note 1 to entry: The single test result may also be the mean of two or more observations or the result of a calculation from a set of observations as specified by the standard test method.

4 Preparatory treatment of laboratory samples of bituminous mixtures

Prepare laboratory samples in accordance with EN 12697-28 to obtain suitable test portions.

5 Determination of binder content

5.1 General principles of test

The test method for determining the binder content of a test portion of bituminous mixture, prepared in accordance with Clause 4, normally comprises the following basic operations:

a) binder extraction by dissolving in a hot or cold solvent;

b) separation of mineral matter from the binder solution;

c) determination of binder quantity by difference or binder recovery;

d) calculation of soluble binder content.

NOTE 1 The sequence of operations and choice of test procedures to be followed are illustrated in Figure A.1.

If it is suspected that water is present in the laboratory sample, the sample should either be dried to constant mass (see Clause 6), or the water content determined by the method described in EN 12697-14, or the sample treated as in EN 12697-28.

NOTE 2 All test procedures and associated equipment relating to each basic operation shown in Figure A.1 are equally acceptable. Other equipment and procedures, including non-extraction methods, may also be used. There are documented data to show that the method and equipment will provide results with an accuracy and a precision no worse than that of one of the procedures explicitly shown in Figure A.1.

5.2 Binder extraction

5.2.1 Solvent

The tests in this European Standard require the use of solvents capable of dissolving bitumen and in some cases involve distilling the solution to recover all or some of the bitumen.

NOTE Currently all hydrocarbon solvents are regarded as “hazardous” and “environmentally unfriendly” to varying degrees.

Until such time as there is an agreed CEN policy with regard to the usage of hydrocarbon solvents, each member state should specify its preferred solvent, taking into account the Montreal Protocol and the views of its own Regulatory Bodies (see also “Warning” in the Foreword).

Trichloroethylene should be stored in sealed bottles or canisters, which are protected against UV radiation.

When trichloroethylene is recovered by distillation for further use, care should be taken to ensure that the solvent still complies with the appropriate requirements. In particular, acidity may develop; a useful precaution is to store the solvent over calcium oxide in coloured glass or suitable metal containers.

5.2.2 Apparatus

The apparatus should be calibrated and traceable.

5.2.2.1 **Balance**, capable of weighing a test portion to an accuracy of 0,05 % of its mass.

5.2.2.2 **Binder extraction apparatus**, conforming to the requirements of the method selected from B.1, as appropriate.

5.2.3 Procedure

5.2.3.1 Prepare laboratory samples in accordance with EN 12697-28 to obtain suitable test portions.

NOTE If determining binder content by difference, see Annex A.

5.2.3.2 Weigh the test portion to the nearest 0,05 % of the mass taken, and place it in the binder extraction apparatus in accordance with the requirements of the method selected from B.1, as appropriate.

5.2.3.3 The binder extraction procedure shall ensure that no soluble binder is left adhering to the aggregate particles after extraction.

NOTE In limited cases, it may be difficult to dissolve every trace of binder adhering to the aggregate (see A.4).

5.3 Separation of mineral matter

5.3.1 Apparatus

5.3.1.1 **Trays,** that can be heated without damage or change in mass and which are used to dry recovered aggregate.

5.3.1.2 **Apparatus for the separation of mineral filler from the binder solution**, conforming to the requirements of the method selected from B.2, as appropriate.

5.3.2 Procedure

5.3.2.1 Collect the binder solution obtained in accordance with 5.2 and proceed in accordance with the method selected from B.2, as appropriate.

5.3.2.2 The procedure used to separate the mineral filler from the binder solution shall ensure that the residue on ignition of the recovered binder does not exceed 0,5 %, if the nominal filler content is less than 6 % of the mass of aggregate, or 1 % if the nominal filler content is 6 % or greater, when determined in accordance with Annex C.

NOTE 1 This check is not necessary for all samples but rather serves to prove the effectiveness of the method.

NOTE 2 The residue depends on the solvent and the equipment used.

5.3.2.3 Transfer, where necessary, the clean recovered aggregate to a tray. Evaporate the solvent from the aggregate and the binder extraction apparatus. Transfer any remaining fine mineral matter from the binder extraction apparatus to the tray with the rest of the recovered aggregate, ensuring that all mineral matter has been removed from the binder extraction apparatus. Weigh and record the mass of the aggregate in the tray.

5.3.2.4 If required, determine the particle size distribution of the recovered aggregate in accordance with EN 933-1, making due allowance for any mineral filler collected by the filter paper, where appropriate.

5.4 Binder quantity

5.4.1 Apparatus

5.4.1.1 **Recovery apparatus**, conforming to the requirements of the method selected from B.3.

The apparatus should be calibrated and traceable.

5.4.2 Procedure

5.4.2.1 Difference method

Where the binder quantity is determined by difference, add the mass of recovered aggregate to the mass of any mineral filler collected by filter paper.

5.4.2.2 Recovery method

Where the binder quantity is determined by recovering the binder of the binder solution, follow the procedures described in B.3.

5.5 Calculation and expression of results

5.5.1 General

The soluble binder content, S, as a percentage of the mass of the original dry test portion, shall be calculated in accordance with 5.5.2, 5.5.3, 5.5.4 or 5.5.5, as appropriate.

NOTE 1 Formulae are given for un-dried test portions. Where test portions have been dried to constant mass, M, becomes the mass of the dried test portion and M_W, is deleted.

NOTE 2 For mixtures with a binder having a significant proportion of insolubles, the total binder content can be calculated by taking account of the insoluble binder content in accordance with A.4.

5.5.2 Binder determined by difference

Calculate the soluble binder content, S, in percentage by mass, by means of the following formula:

$$S = \frac{100 \times [M - (M_1 + M_W)]}{M - M_W} \quad (1)$$

where

S is the soluble binder content, expressed in percent (%);

M is the mass of un-dried test portion, expressed in grams (g);

M_1 is the mass of recovered mineral matter, expressed in grams (g);

M_W is the mass of water in the un-dried test portion, expressed in grams (g).

5.5.3 Binder by total recovery

Calculate the soluble binder content, S, in percentage by mass, by means of the following formula:

$$S = \frac{100 \times M_b}{M - M_W} \quad (2)$$

where

S is the soluble binder content, expressed in percent (%);

M is the mass of un-dried test portion, expressed in grams (g);

M_b is the mass of recovered binder, expressed in grams (g);

M_W is the mass of water in the un-dried test portion, expressed in grams (g).

5.5.4 Binder by recovery from portion (volume calculation)

Calculate the soluble binder content, S, in percentage by mass, by means of the following formula:

$$S = \frac{100 \times z \times V \times d}{(M - M_W) \times (d \times v - z)} \quad (3)$$

where

S is the soluble binder content, expressed in percent (%);

M is the mass of un-dried test portion, expressed in grams (g);

z is the average mass of binder recovered from each aliquot portion of binder solution, expressed in grams (g);

V is the total volume of solvent, expressed in cubic millimetres (mm^3);

v is the volume of each aliquot solution portion, expressed in cubic millimetres (mm^3);

d is the density of the binder at 25 °C, expressed in grams per cubic millimetres (g/mm^3);

M_W is the mass of water in the un-dried test portion, expressed in grams (g).

5.5.5 Binder by recovery from portion (mass calculation)

Calculate the soluble binder content, S, in percentage by mass, by means of the following formula:

$$S = \frac{100 \times M_B}{M - M_W} \quad (4)$$

where

S is the soluble binder content, expressed in percent (%);

M is the mass of un-dried test portion, expressed in grams (g);

M_W is the mass of the water in the un-dried test portion, expressed in grams (g);

M_B is the mass of soluble binder in the test portion, expressed in grams (g).

$$M_B = \frac{M_2 - M_1}{M_3 - M_2} M_P \quad (5)$$

where

M_P is the mass of solvent in the test portion, expressed in grams (g);

M_1, M_2, M_3 are as defined in B.3.2.

6 Drying to constant mass

6.1 General

In all the test procedures in this European Standard it is necessary, at some stage, to ensure that materials or equipment are dried to constant mass. On all such occasions, the method in 6.2 to 6.3 shall be used.

6.2 Apparatus

6.2.1 **Oven or drying cabinet**, of suitable capacity and capable of holding the required temperatures.

6.2.2 **Balance**, with an accuracy of 0,01 % or better.

6.2.3 **Desiccator**, of suitable capacity (optional).

6.3 Procedure

6.3.1 Place the material or equipment in the oven or drying cabinet and dry to constant mass.

NOTE 1 In case of an oven, a temperature of (110 ± 5)°C is usually suitable. Where it is necessary to dry a test portion before analysis a temperature of (80 ± 5) °C may be more suitable to avoid binder drainage, but a longer time will be necessary.

NOTE 2 In case of a drying cabinet lower temperatures are used. The lower the temperature, the longer it will take to dry to constant mass.

NOTE 3 Constant mass is defined as successive weighings after drying at least 1 h apart not differing by more than 0,1 %.

For convenience, it is recommended that the successive weighings to determine constant mass should be carried out whilst the material is hot. It may be advisable to protect the balance from heat.

6.3.2 When constant mass has been achieved, cool in a moisture-free atmosphere and weigh.

NOTE A moisture-free atmosphere can be obtained by cooling in a desiccator.

7 Reporting of results

7.1 Results

Report the soluble binder content, and, where appropriate:

a) the water content to the nearest 0,1 % by mass in accordance with EN 12697-14;

b) and/or the insoluble binder content in accordance with A.4.

7.2 Test report

The report shall contain at least the following information in addition to that in 7.1:

a) name and address of the testing laboratory;

b) unique serial number for the test report;

c) name of the client;