SLOVENSKI STANDARD
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Bituminous mixtures - Test methods for hot mix asphalt - Part 1: Soluble binder content

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Asphalt - Prüfverfahren für Heißasphalt - Teil 1: Löslicher Bindemittelgehalt

Mélanges bitumineux - Essais pour enrobés à chaud - Teneur en bitume

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Bituminous mixtures - Test methods for hot mix asphalt - Part 1: Soluble binder content

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

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Contents

Foreword

This European Standard has been prepared by Technical Committee CEN/TC 227 "Road materials", the secretariat of which is held by DIN.

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of an identical text or by endorsement, at the latest by April 2001, and conflicting national standards shall be withdrawn at the latest by April 2001.

This European Standard is one of a series of standards as listet below:

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

prEN 12697-2, *Bituminous mixtures - Test methods for hot mix asphalt - Part 2: Particle size distribution*

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

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

prEN 12697-5, *Bituminous mixtures - Test methods for hot mix asphalt - Part 5: Determination of the maximum density*

prEN 12697-6, *Bituminous mixtures - Test methods for hot mix asphalt - Part 6: Determination of bulk density of bituminous specimen by hydro-static method*

prEN 12697-7, *Bituminous mixtures - Test methods for hot mix asphalt - Part 7: Determination of bulk density of bituminous specimens by gamma rays*

prEN 12697-8, *Bituminous mixtures - Test methods for hot mix asphalt - Part 8: Determination of the air voids content of bituminous materials*

prEN 12697-9, *Bituminous mixtures - Test methods for hot mix asphalt - Part 9: Determination of the reference density, gyrator compactor*

prEN 12697-10, *Bituminous mixtures - Test methods for hot mix asphalt - Part 10: Compactibility*

prEN 12697-11, *Bituminous mixtures - Test methods for hot mix asphalt - Part 11: Determination of the affinity between aggregates and binders*

prEN 12697-12, *Bituminous mixtures - Test methods for hot mix asphalt - Part 12: Determination of the water sensitivity of specimen*

EN 12697-13, *Bituminous mixtures - Test methods for hot mix asphalt - Part 13: Temperature measurement*

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

prEN 12697-15, *Bituminous mixtures - Test methods for hot mix asphalt - Part 15: Determination of the segregation sensitivity of bituminous mixtures*

prEN 12697-16, *Bituminous mixtures - Test methods for hot mix asphalt – Part 16: Abrasion by studded tyres*

prEN 12697-17, *Bituminous mixtures - Test methods for hot mix asphalt – Part 17: Particle loss of specimen*

prEN 12697-18, *Bituminous mixtures - Test methods for hot mix asphalt - Part 18: Binder drainage from porous asphalt*

prEN 12697-19, *Bituminous mixtures - Test methods for hot mix asphalt – Part 19: Permeability of specimen*

prEN 12697-20, *Bituminous mixtures - Test methods for hot mix asphalt - Part 20: Indentation using cube or marshall specimen*

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prEN 12697-22, *Bituminous mixtures - Test methods for hot mix asphalt - Part 22: Wheel tracking test*

prEN 12697-23, *Bituminous mixtures - Test methods for hot mix asphalt - Part 23: Indirect tensile test*

prEN 12697-24, *Bituminous mixtures - Test methods for hot mix asphalt - Part 24: Resistance to fatique*

prEN 12697-25, *Bituminous mixtures - Test methods for hot mix asphalt – Part 25: Dynamic creep test*

prEN 12697-26, *Bituminous mixtures - Test methods for hot mix asphalt - Part 26: Stiffness*

EN 12697-27, *Bituminous mixtures - Test methods for hot mix asphalt - Part 27: Sampling*

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

prEN 12697-29, *Bituminous mixtures - Test methods for hot mix asphalt - Part 29: Determination of the dimensions of bituminous specimen*

prEN 12697-30, *Bituminous mixtures - Test methods for hot mix asphalt - Part 30: Preparation of specimen by impact compactor*

prEN 12697-31, *Bituminous mixtures - Test methods for hot mix asphalt - Part 31: Specimen preparation, gyratory compactor*

prEN 12697-32, *Bituminous mixtures - Test methods for hot mix asphalt - Part 32: Laboratory compaction of bituminous mixtures by a vibratory compactor*

prEN 12697-33, *Bituminous mixtures - Test methods for hot mix asphalt – Part 33: Specimen preparation, slab compactor*

prEN 12697-34, *Bituminous mixtures - Test methods for hot mix asphalt - Part 34: Marshall test*

prEN 12697-35, *Bituminous mixtures - Test methods for hot mix asphalt – Part 35: Laboratory mixing*

prEN 12697-36, *Bituminous mixtures - Test methods for hot mix asphalt - Part 36: Method for the determination of the thickness of a bituminous pavement*

prEN 12697-37, *Bituminous mixtures - Test methods for hot mix asphalt - Part 37: Hot sand test for the adhesivity of binder on precoated chippings for HRA*

prEN 12697-38, *Common equipment and calibration*

The applicability of this European Standard is described in the product standards for bituminous mixtures.

No existing European Standard is superseded.

WARNING The method described in this European Standard may require the use of dichloromethane (methylene chloride). This solvent is hazardous to health and is subject to occupational exposure limits as described in relevant legislation and regulations.

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

Annex A is informative. The annexes B and C are normative.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

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 whilst 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 it has been demonstrated that they provide the same results as one of the methods in Annex B or Annex C within the limits of the precision given in the Standard.

1 Scope

This European Standard 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.

Test methods for the analysis of mixtures containing modified binders are outside the scope of this European Standard.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

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

EN 12697-3:2000, *Bituminous mixtures – Test methods for hot mix asphalt – Part 3: Bitumen recovery: Rotary evaporator.*

EN 12697-4:2000, *Bituminous mixtures – Test methods for hot mix asphalt –Part 4: Bitumen recovery: Fractionating column.*

EN 12697-14:2000, *Bituminous mixtures – Test methods for hot mix asphalt – Part 14: Water content.*

prEN 12697-27:2000, *Bituminous mixtures – Test methods for hot mix asphalt – Part 27: Sampling*

prEN 12697-28:2000, *Bituminous mixtures – Test methods for hot mix asphalt – Part 28: Preparation of samples for determining binder content, water content and grading.*

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.*

ISO 5725, *Accurancy (trueness and precision) of measurement methods and results.*

3 Terms and definitions

For the purposes of this European Standard, the following terms and definitions apply.

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

NOTE 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 Precision depends only on the distribution of random errors and does not relate to the true value or the specified value.

NOTE 2 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 "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
value less than or equal to which the absolute difference between two test results obtained under repeatability conditions may be expected to be with a probability of 95 %

NOTE 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
value less than or equal to which the absolute difference between two test results obtained under reproducibility conditions may be expected to be with a probability of 95 %

NOTE The symbol used for reproducibility limit is *R*.

3.10
single test result
value obtained by applying the standard test method fully, once to a single specimen may 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 prEN 12697-28:2000 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.

NOTE 2 If it is suspected that water is present in the laboratory sample, it should be dried to constant mass (see clause 6) or the water content may be determined by the method described in EN 12697-14:2000 or the sample treated as in prEN 12697-28:2000.

NOTE 3 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, can also be used. There are documented data to show that the method and equipment will provide results with 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 1 Currently all hydrocarbon solvents are regarded as "hazardous" and "environmentally unfriendly" to varying degrees.

NOTE 2 Until such time as there is an agreed CEN policy with regard to their usage, 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).

NOTE 3 Trichloroethylene should be stored in sealed bottles or canisters, which are protected against UV radiation.

NOTE 4 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 and a useful precaution is to store the solvent over calcium oxide in coloured glass or suitable metal containers.

5.2.2 Apparatus

NOTE 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 clause B.1 as appropriate.

5.2.3 Procedure

5.2.3.1
Prepare laboratory samples in accordance with prEN 12697-28:2000 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 clause 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 clause 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 in which 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 clause 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 clause 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 This check is not necessary on all samples but only to prove the effectiveness of the method.

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 is 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

Recovery apparatus, conforming to the requirements of the method selected from clause B.3 of EN 12697-3:2000 or EN 12697-4:2000, as appropriate.

NOTE 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 from the total or a portion of the binder solution, follow the procedures described in clause B.3 of EN 12697-3:2000 or EN 12697-4:2000, as appropriate.

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 undried 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 clause A.4.

5.5.2 Binder determined by difference

Calculate the soluble binder content, S, by means of the following equation:

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

where

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

M is the mass of undried 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 undried 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 equation:

$$S = \frac{100 \times M_b}{M - M_w} \qquad (2)$$

where

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

M is the mass of undried 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 undried 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 equation:

$$S = \frac{100 \times z \times V}{V\,(M - M_w)\,(1 - z / d_v)} \qquad (3)$$

where

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

M is the mass of undried 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 millilitres (ml);

v is the volume of each aliquot solution portion, expressed in millilitres (ml);

d_v is the relative density, 25/25 ^{0}C of the binder, expressed in grams per millilitres (g/ml);

M_w is the mass of water in the undried 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 equation:

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

where

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

M is the mass of undried test portion, expressed in grams (g);

M_w is the mass of the water in the undried test portion, expressed in grams (g);

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

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

where

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

M, 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, of suitable capacity and capable of holding the required temperatures.

6.2.2
Balance, of suitable accuracy.

6.2.3
Desiccator, of suitable capacity.

6.3 Procedure

6.3.1
Place the material or equipment in the oven and dry to constant mass at a temperature of (110 ± 5) ^{0}C.

NOTE 1 Constant mass is deemed to be achieved when the difference between successive weighings at half-hourly intervals does not exceed 0,05 % of the last weighed mass.

NOTE 2 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 desiccator and weigh.

NOTE Where it is necessary to dry a test portion before analysis a temperature of (80 ± 5) ^{0}C may be more suitable to avoid binder drainage but a longer time will be necessary.

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 12697-14:2000;

b) and/or the insoluble binder content in accordance with clause 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;

d) description and an identification of the sample, and the date of receipt;

e) identification of the test method by reference to the Annex B apparatus used.

f) any deviations, additions to or exclusions from the test method;

g) whether or not the sample was accompanied by a sampling certificate;

h) signature of officer accepting the technical responsibility for the test report;

i) date of issue.

8 Precision data

NOTE 1 Criteria for judging the acceptability of the binder content of bituminous mixtures determined by this European Standard are limited. The data that are tabulated herein were obtained by three separate precision experiments on materials from two different geographical regions of the European Union. An indication of the precision of this method of test can be obtained from the data presented in 8.1, 8.2 and 8.3.

NOTE 2 Differences in the results obtained by two laboratories on test samples taken from the same bulk sample may be due to errors in the sample reduction and/or the procedures adopted by the analysis. Any systematic differences in the comparison of a group of test results should be investigated to detect the cause or causes of the bias.

NOTE 3 This bias will generally be due to non-adherence to the detail of the method of analysis if the same test procedures are being followed or, in isolated cases, small differences may be due to the particular test procedure selected.

NOTE 4 If differences arise when the same test procedures are being followed it will normally be possible, with co-operation between the laboratories, to eliminate the cause. If small differences occur due to the test procedures being different, it is recommended that the two laboratories concerned at the earliest opportunity should examine the interpretation of the test results.

8.1 Precision - Experiment 1

NOTE The limits for the differences between the analysis results of two test samples obtained from the same bulk sample are as given in 8.1.4.