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Standards

Irish Standard  
I.S. EN 12697-1:2012

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

## I.S. EN 12697-1:2012

*Incorporating amendments/corrigenda/National Annexes issued since publication:*

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**NSAI**  
1 Swift Square,  
Northwood, Santry  
Dublin 9

T +353 1 807 3800  
F +353 1 807 3838  
E standards@nsai.ie  
W NSAI.ie

**Sales:**  
T +353 1 857 6730  
F +353 1 857 6729  
W standards.ie

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English Version

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

Mélanges bitumineux - Méthode d'essai pour mélange  
hydrocarboné à chaud - Partie 1: Teneur en liant soluble

Asphalt - Prüfverfahren für Heißasphalt - Teil 1: Löslicher  
Bindemittelgehalt

This European Standard was approved by CEN on 28 April 2012.

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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**Management Centre: Avenue Marnix 17, B-1000 Brussels**

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## Foreword

This document (EN 12697-1:2012) has been prepared by Technical Committee CEN/TC TC "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of an identical text or by endorsement, at the latest by December 2012, and conflicting national standards shall be withdrawn at the latest by December 2012.

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 12697-1:2005.

Compared with EN 12697-1:2005, the following changes have been made:

- a) Removal of warning that the precision may be comprised with polymer-modified binders even when following Annex D;
- b) For the separation of mineral, a note is added that the residue depends on the solvent and the equipment used;
- c) The desiccator is made optional with the method of achieving a moisture-free atmosphere not fixed;
- d) The definition of constant mass is changed;
- e) Alternative procedures for determination of binder are extended;
- f) Volume units are corrected from  $\text{mm}^3$  to  $10^3 \text{ mm}^3$  as appropriate;
- g) In Annex B, note is added to ensure the binder is well dissolved;
- h) In Annex B, density of perchloroethylene is changed from  $(1,6 \pm 0,05) \text{ g/cm}^3$  at  $24 \text{ }^\circ\text{C}$  to  $(1,6 \pm 0,05) \text{ Mg/m}^3$  at  $20 \text{ }^\circ\text{C}$ ;
- i) In Annex B, weighing of multiple sieves that are fitted to the feed funnel is allowed;
- j) In Annex B, repeating the procedure with a second cup in the centrifuge is undertaken with half the flow rate;
- k) In Annex B, note is added that the second run is unnecessary for some centrifuges with larger capacities;
- l) In Annex C,  $M_2$  is clarified as being the mass of fine mineral matter;
- m) In Annex C, the procedures for collecting the recovered binder are clarified;
- n) In Annex D, a note is added that a good solubility of the polymer-modified binder does not always guarantee a good extraction of the PmB in the bituminous mixture;
- o) In Annex D, the discussion on different solvents is refined;
- p) In Annex D, the flow rate is adjusted rather than just checked to avoid overflowing;

- q) In Annex D, the exceptions on the apparatus and procedure for the centrifuge extractor method are removed and notes added;
- r) In Annex D, note is added to continuous flow centrifuge;
- s) In Annex D, precision statement is changed.

This European Standard is one of a series of standards for Bituminous mixtures as listed below:

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

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

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

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

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

EN 12697-6, *Bituminous mixtures — Test methods for hot mix asphalt — Part 6: Determination of bulk density of bituminous specimens*

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

EN 12697-8, *Bituminous mixtures — Test methods for hot mix asphalt — Part 8: Determination of void characteristics of bituminous specimens*

EN 12697-10, *Bituminous mixtures — Test methods for hot mix asphalt — Part 10: Compactability*

EN 12697-11, *Bituminous mixtures — Test methods for hot mix asphalt — Part 11: Determination of the affinity between aggregate and bitumen*

EN 12697-12, *Bituminous mixtures — Test methods for hot mix asphalt — Part 12: Determination of the water sensitivity of bituminous specimens*

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*

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

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

EN 12697-17, *Bituminous mixtures — Test methods for hot mix asphalt — Part 17: Particle loss of porous asphalt specimen*

EN 12697-18, *Bituminous mixtures — Test methods for hot mix asphalt — Part 18: Binder drainage*

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

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EN 12697-20, *Bituminous mixtures — Test methods for hot mix asphalt — Part 20: Indentation using cube or cylindrical specimens(CY)*

EN 12697-21, *Bituminous mixtures — Test methods for hot mix asphalt — Part 21: Indentation using plate specimens*

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

EN 12697-23, *Bituminous mixtures — Test methods for hot mix asphalt — Part 23: Determination of the indirect tensile strength of bituminous specimens*

EN 12697-24, *Bituminous mixtures — Test methods for hot mix asphalt — Part 24: Resistance to fatigue*

prEN 12697-25, *Bituminous mixtures — Test methods for hot mix asphalt — Part 25: Cyclic compression test*

EN 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*

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

EN 12697-30, *Bituminous mixtures — Test methods for hot mix asphalt — Part 30: Specimen preparation by impact compactor*

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

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

EN 12697-33, *Bituminous mixtures — Test methods for hot mix asphalt — Part 33: Specimen prepared by roller compactor*

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

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

EN 12697-36, *Bituminous mixtures — Test methods for hot mix asphalt — Part 36: Determination of the thickness of a bituminous pavement*

EN 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*

EN 12697-38, *Bituminous mixtures — Test methods for hot mix asphalt — Part 38: Common equipment and calibration*

EN 12697-39, *Bituminous mixtures — Test methods for hot mix asphalt — Part 39: Binder content by ignition*

EN 12697-40, *Bituminous mixtures — Test methods for hot mix asphalt — Part 40: In-situ drainability*

EN 12697-41, *Bituminous mixtures — Test methods for hot mix asphalt — Part 41: Resistance to de-icing fluids*



EN 12697-42, *Bituminous mixtures — Test methods for hot mix asphalt — Part 42: Amount of coarse foreign matter in reclaimed asphalt*

EN 12697-43, *Bituminous mixtures — Test methods for hot mix asphalt — Part 43: Resistance to fuel*

EN 12697-44, *Bituminous mixtures — Test methods for hot mix asphalt — Part 44: Crack propagation by semi-circular bending test*

EN 12697-45, *Bituminous mixtures — Test methods for hot mix asphalt — Part 45: Saturation ageing tensile stiffness (SATS) conditioning test*

EN 12697-46, *Bituminous mixtures — Test methods for hot mix asphalt — Part 46: Low temperature cracking and properties by uniaxial tension tests*

EN 12697-47, *Bituminous mixtures — Test methods for hot mix asphalt — Part 47: Determination of the ash content of natural asphalts*

prEN 12697-48, *Bituminous mixtures — Test methods for hot mix asphalt — Part 48: Interlayer bonding<sup>1)</sup>*

prEN 12697-49, *Bituminous mixtures — Test methods for hot mix asphalt — Part 49: Determination of friction after polishing*

prEN 12697-50, *Bituminous mixtures — Test methods for hot mix asphalt — Part 50: Scuffing resistance of surface course<sup>1)</sup>*

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

**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 (tetrachloroethylene) 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 emphasised that adequate training should be given to staff employed in the usage of these substances.

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, 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.

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1) In preparation.

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

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

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

EN 12697-28:2000, *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.

### 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.

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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.

NOTE 2 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 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, 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 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 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).

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

### 5.2.2 Apparatus

NOTE 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).

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