

Bituminous mixtures - Test methods - Part 4: Bitumen recovery: Fractionating column

## EESTI STANDARDI EESSÕNA

## NATIONAL FOREWORD

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English Version

**Bituminous mixtures - Test methods - Part 4: Bitumen recovery:  
Fractionating column**Mélanges bitumineux - Méthodes d'essai - Partie 4 :  
Récupération des bitumes à la colonne à distillerAsphalt - Prüfverfahren - Teil 4: Rückgewinnung des  
Bindemittels: Fraktionierkolonne

This European Standard was approved by CEN on 19 March 2015.

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## Foreword

This document (EN 12697-4:2015) 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of an identical text or by endorsement, at the latest by December 2015 and conflicting national standards shall be withdrawn at the latest by December 2015.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 12697-4:2005.

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

- the series title no longer makes the method exclusively for hot mix asphalt;
- definitions for precision have been removed;
- Formula (1) in 5.2.1 was updated;
- 5.3.3 has been extended;
- Figures 2 and 3 have been corrected;
- the Figure for CO<sub>2</sub> supply has been added.

This European Standard is one of a series of standards as follows:

- EN 12697-1, *Bituminous mixtures — Test methods for hot mix asphalt — Part 1: Soluble binder content*
- EN 12697-2, *Bituminous mixtures — Test method — 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 — Part 4: Bitumen recovery: Fractionating column* [the present document]
- 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*
- 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*
- EN 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 method 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 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>*
- EN 12697-49, *Bituminous mixtures — Test methods for hot mix asphalt — Part 49: Determination of friction after polishing*
- prCEN/TS 12697-50, *Bituminous mixtures — Test methods — Part 50: Resistance to Scuffing<sup>1)</sup>*
- FprCEN/TS 12697-51, *Bituminous mixtures — Test methods — Part 51: Surface shear strength test*

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1) Currently at Enquiry stage.

- prEN 12697-52, *Bituminous mixtures — Test methods — Part 52: Conditioning to address oxidative ageing*<sup>2)</sup>
- prEN 12697-53, *Bituminous mixtures — Test methods — Part 53: Cohesion increase by spreadability-meter method*<sup>3)</sup>

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.

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2) In preparation.

3) Currently at drafting stage.

## Introduction

**WARNING** — The method described in this European Standard may require the use of dichloromethane (methylene chloride), 1,1,1 trichloroethane, benzene, trichloroethylene, xylene, toluene or other solvent 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.

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## 1 Scope

This European Standard specifies a test method for the recovery of soluble bitumen from bituminous mixtures from pavements in a form suitable for further testing. The procedure is suitable for the recovery of paving grade bitumen and is also suitable for mixtures containing volatile matter such as cut-back bitumen but the results may be less precise. This European Standard is the reference method for mixtures containing volatile matter, but the rotary evaporator procedure (see EN 12697-3) for mixtures with paving grade bitumen.

NOTE There is limited experience of recovery when polymer-modified bitumen is used.

## 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 12594, *Bitumen and bituminous binders — Preparation of test samples*

EN 12697-1:2012, *Bituminous mixtures — Test methods for hot mix asphalt — Part 1: Soluble binder content*

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

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

## 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 after extraction

## 4 Principle

The bitumen is separated from the sample by dissolving in dichloromethane (or other suitable solvent). After removal of undissolved solids, the bitumen solution is concentrated by atmospheric distillation in a fractionating column. The last traces of solvent are removed from the concentrate by distillation at a temperature of 100 °C above the expected softening point or 175 °C, whichever is the higher, with the pressure reduced from atmospheric pressure 100 kPa to 20 kPa and with the aid of a stream of carbon dioxide gas. When cutback bitumens containing very volatile fluxes, e.g. white spirit, are being recovered the carbon dioxide gas is omitted.