



SLOVENSKI STANDARD
kSIST FprEN 12697-4:2014

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**Bitumenske zmesi - Preskusne metode - 4. del: Ponovna pridobitev bitumna:
kolonska frakcionirana destilacija**

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

Asphalt - Prüfverfahren - Teil 4: Rückgewinnung des Bindemittels: Fraktionierkolonne

Mélanges bitumineux - Méthodes d'essai - Partie 4 : Récupération des bitumes à la
colonne à distiller

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

Mélanges bitumineux - Méthodes d'essai - Partie 4 :
Récupération des bitumes à la colonne à distiller

Asphalt - Prüfverfahren - Teil 4: Rückgewinnung des
Bindemittels: Fraktionierkolonne

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Foreword

This document (FprEN 12697-4:2014) has been prepared by Technical Committee CEN/TC 227 "Road materials", the secretariat of which is held by DIN.

This document is currently submitted to the Unique Acceptance Procedure.

This document will supersede 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;
- Subclause 5.3.3 has been extended;
- Figures 2 and 3 have been corrected;
- the figure for CO₂ supply has been added.

This European Standard is one of a series of standards 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 — 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 aggregates and bitumen*;
- EN 12697-12, *Bituminous mixtures — Test methods for hot mix asphalt — Part 12: Determination of the water sensitivity of bituminous specimens*;

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- 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 specimen 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 (in preparation)*;
- 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 for hot mix asphalt — Part 50: Resistance to Scuffing (in preparation)*;
- prCEN/TS 12697-51, *Bituminous mixtures — Test methods for hot mix asphalt — Part 51: Interlayer Bonding (Compressed shear bond test - CSBT, Cyclic compressed shear bond test - CCSBT). (in preparation)*;
- prEN 12697-52, *Bituminous mixtures — Test methods — Part 53, Cohesion increase by spreadability-meter method (in preparation)*.

Introduction

WARNING — The method described in this European Standard may require the use of dichloromethane (methylene chloride), 1,1,1 trichlorethane, benzene, trichlorethylene, 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.

iTeh STANDARD PREVIEW
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SIST EN 12697-4:2015

<https://standards.iteh.ai/catalog/standards/sist/0adb540f-ba49-4a4a-a8e2-46ac7fc44604/sist-en-12697-4-2015>

1 Scope

This European Standard (EN 12697-4:2014) describes 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.

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5 Apparatus

5.1 Apparatus for the extraction of the soluble bitumen

Suitable container with stopper, in which the sample and solvent can be agitated together, or other apparatus for the extraction of soluble bitumen defined in EN 12697-1.

NOTE The use of the hot extraction methods in EN 12697-1 may harden the binder and hence affect the results from subsequent tests. However, this hardening is usually regarded as approximately balancing the softening resulting from any remaining solvent.

5.2 Apparatus for the clarification of the bitumen solution

For separation of solids from the bitumen solution, a sample-tube centrifuge, a continuous centrifuge or a filtration system may be used.

Centrifuges are suitable for separation of solids from any bitumen solutions and are the recommended apparatus for use with this method. The filtration apparatus may not be suitable for the separation of solids from all types of bituminous solutions but it has been included in this method because of the general availability of this equipment in asphalt testing laboratories. If difficulties are experienced using a pressure filter the centrifuge technique should be used.

5.2.1 Sample tube centrifuge, capable of developing an acceleration of at least 15 000 m/s² in accordance with the following formula:

$$a = 1,097 n^2 \times r - 10^{-5} \quad (1)$$

where

a is the acceleration, expressed in metres per second squared (m/s²);

n is the number of revolutions, expressed in revolutions per minute (r/min);

r is the radius to the bottom of the tubes (internal) when rotating, expressed in millimetres (mm).

The centrifuge tubes shall be fitted with effective closures.

The speed of rotation shall be verified regularly in accordance with EN 12697-38 to ensure that the centrifuge maintains its performance at all times. The centrifuge shall be maintained in accordance with EN 12697-3.

NOTE A typical centrifuge of this type, suitable for this method, carries four or six tubes of 200 ml or 500 ml capacity rotating at 3 000 r/min at a radius (as defined above) of 250 mm.

5.2.2 Continuous laboratory centrifuge, that takes a continuous feed of material, giving a continuous discharge of solution and capable of achieving an acceleration of 25 000 m/s².

5.2.3 A pressure filter, of an appropriate size.

5.2.4 An air pump, for supplying oil-free air at about 200 kPa.

5.2.5 A supply of filter papers with a minimum retention size of 11 µm, to fit the pressure filter.

NOTE A pressure filter taking a paper of 270 mm diameter is suitable.