SLOVENSKI STANDARD
SIST EN 12697-3:2013+A1:2019

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Nadomešča:
SIST EN 12697-3:2013

Bitumenske zmesi - Preskusne metode - 3. del: Ugotavljanje deleža veziva: rotacijski uparjalnik

Bituminous mixtures - Test methods - Part 3: Bitumen recovery: Rotary evaporator

Asphalt - Prüfverfahren - Teil 3: Rückgewinnung des Bindemittels: Rotationsverdampfer

Mélanges Bitumineux - Méthodes d'essai - Partie 3: Extraction des bitumes à l'évaporateur rotatif

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English Version

Bituminous mixtures - Test methods - Part 3: Bitumen recovery: Rotary evaporator

Mélanges Bitumineux - Méthodes d'essai - Partie 3: Extraction des bitumes à l'évaporateur rotatif

Asphalt - Prüfverfahren - Teil 3: Rückgewinnung des Bindemittels: Rotationsverdampfer

This European Standard was approved by CEN on 28 March 2013 and includes Amendment 1 approved by CEN on 9 November 2018.

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

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Foreword

This document (EN 12697-3:2013+A1:2018) has been prepared by Technical Committee CEN/TC 227 "Road materials", the secretariat of which is held by BSI.

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of an identical text or by endorsement, at the latest by June 2019, and conflicting national standards shall be withdrawn at the latest by June 2019.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

This document includes Amendment 1 approved by CEN on 9 November 2018.

The start and finish of text introduced or altered by amendment is indicated in the text by tags ⟦A1⟩ ⟨A1⟧.

This document supersedes ⟦A1⟩ EN 12697-3:2013 ⟨A1⟧.

⟦A1⟩ The significant changes made in EN 12697-3:2013+A1:2018 compared to EN 12697-3:2013 are:

— [Title] The series title no longer makes the method exclusively for hot mix asphalt;

— [Foreword] The list of standards in the Foreword was deleted and the list of significant changes was adapted;

— [5.1] NOTE: Paragraph deleted, stating that the hardening is usually approximately equivalent to the softening resulting from any solvent residue;

— [5.3.6] The temperature range 100 °C to 200 °C for thermometer is replaced by range of temperatures specified in Table 1 according to the solvent used;

— [7.1.2] Clarification that the 0,063 mm sieve is used for removal of any insoluble material;

— [7.3] The whole sub-clause has been replaced, with the following significant changes:

  — the order of the procedure in the previous version (7.3.8 to 7.3.25) has been altered for clarity and is now described in 7.3.8 to 7.3.18;

  — NOTES introduced in 7.3.9 and 7.3.11 with advise to not exceed 120 °C for T_2 for bitumen's, designated and specified by kinematic viscosity at 60 °C regardless of solvent used;

— [Clause 9] Reference to (7.1.15) amended to (7.3.13) followed by the changes in 7.3.

— editorial updates. ⟨A1⟧

⟦A1⟩ A list of all parts in the EN 12697 series can be found on the CEN website. ⟨A1⟧

WARNING — The method described in this European Standard may require the use of dichloromethane (methylene chloride), ⟦A1⟩ 1,1,1-Trichlorethane ⟨A1⟧, benzene, trichlorethylene, xylene, toluene, tetrachloroethylene 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 important that adequate training be given to staff employed in the usage of these substances.

According to the CEN-CENELEC Internal Regulations, the national standards organisations 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, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

1 Scope

This document describes a test method for the recovery of soluble bitumen from bituminous mixtures used in road, airfield or similar pavements in a form suitable for further testing. The test can be undertaken on either loose or compacted asphalt materials. The procedure is suitable for the recovery of paving grade bitumens, for which materials this European Standard is the reference method. The fractionating column procedure (see EN 12697-4) is the reference method for mixtures containing volatile matter such as cut-back bitumen.

For recovery of polymer modified bitumens, the rotary evaporator procedure is recommended.

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 12697-38, *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 terms and definitions given in EN 12697-1:2012 and the following apply.

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

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

Note 2 to entry: The soluble binder content is expressed in percent by mass.

3.2
insoluble binder content
proportion of binder that adheres to the aggregate after extraction

Note 1 to entry: The insoluble binder content is expressed in percent by mass.

4 Principle

The bitumen is separated from the sample by dissolving in dichloromethane (or other suitable solvent). After removal of undissolved solids from the bitumen solution, the bitumen is recovered from it by vacuum distillation using a rotary evaporator. The bitumen is in solution for less than 24 h.

5 Apparatus

5.1 Apparatus for the extraction of the soluble bitumen

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

〈A1〉 NOTE The use of the hot extraction methods in EN 12697-1 may harden the binder and hence affect the results from subsequent tests. 〈A1〉

5.2 Apparatus for the clarification of the bitumen solution

For clarification of 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.

NOTE If an asphalt analyser is used for the extraction of soluble bitumen, the use of a centrifuge is not required.

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

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

where

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

n is the number of revolutions, expressed as revolution 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 this document.

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 appropriate size.

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

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.

5.3 Distillation apparatus

NOTE The distillation apparatus, a typical rotary evaporator, is shown in Figure 1.

5.3.1 Rotary evaporator, incorporating a rotating evaporating flask which can be operated under vacuum. Some models have an inclined condenser as shown in Figure 1, but other models using vertical condensers may be used. The apparatus shall:

a) accept a 1 l capacity evaporating flask;

b) have a drive motor and speed control capable of rotating the evaporating flask at (75 ± 15) r/min;

c) be capable of operating at P_2 kPa pressure where P_2 is taken from Table 1 for the solvent to be used;

d) have an evaporation capacity of solvent at a bath temperature of $(T_1 + 5)$ °C of at least 0,85 l/h when the flask is rotated at 75 r/min where T_1 is taken from Table 1 for the solvent to be used.

5.3.2 A 1 l, pressure resistant, evaporating flask, of heat-resistant glass fitted with a ground-glass joint.

5.3.3 Oil bath for 1 l evaporating flask, capable of raising the temperature of the oil to T_3 °C where T_3 is taken from Table 1 for the solvent to be used. A high temperature silicone oil is recommended because many other oils may be irreversibly damaged above 150 °C.

5.3.4 Vacuum pump, capable of reducing the absolute pressure in a leak-proof system to P_2 kPa where P_2 is taken from Table 1 for the solvent to be used. An oil sealed vacuum pump, running in the gas ballast mode, is recommended.

5.3.5 Two pressure gauges, capable of indicating the level of reduced pressure in the distillation apparatus; one with an absolute range from 0 kPa to 100 kPa with an accuracy of ± 0,5 kPa (0 mbar to 1 000 mbar ± 5 mbar) and one with an absolute range from 0 kPa to 5 kPa with an accuracy of ± 0,1 kPa (0 mbar to 50 mbar ± 1,0 mbar). Alternatively, a single gauge covering the required range with the specified accuracy may be used.

5.3.6 [A1] Thermometer, capable of covering the range of temperatures specified in Table 1 according to the solvent used with an accuracy of ± 0,5 °C. [A1]

5.3.7 A suitable container for bituminous solutions.

NOTE 1 A suitable container can be a flat-bottomed glass container of 2 l or 3 l capacity.

NOTE 2 A Winchester bottle is suitable.

6 Solvent and other materials

6.1 Dichloromethane (methylene chloride) or other suitable solvent

NOTE 1 Possible solvents include toluene, tetrachloroethylene, trichlorethylene, xylene, [A1] 1,1,1-Trichlorethane [A1] and benzene, although bitumen is less soluble in [A1] 1,1,1-Trichlorethane [A1] than in the other solvents. The use of other alternative solvents will require determination of the equivalent distillation conditions for inclusion in Table 1.