SLOVENSKI STANDARD
SIST EN 12697-39:2005

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Bitumenske zmesi ï Preskusne metode za vroļ e asfaltne zmesi ï 39. del: Deleġ veziva ob ġarenju

Bituminous mixtures - Test methods for hot mix asphalt - Part 39: Binder content by ignition

Asphalt - Prüfverfahren für Heißasphalt - Teil 39: Bindemittelgehalt durch Thermoanalyse

Mélanges bitumineux - Méthodes d'essai pour mélange hydrocarboné a chaud - Partie 39: Détermination de la teneur en liant par calcination

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Bituminous mixtures - Test methods for hot mix asphalt - Part 39: Binder content by ignition

Mélanges bitumineux - Méthodes d'essai pour mélange hydrocarboné à chaud - Partie 39: Détermination de la teneur en liant par calcination

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

Management Centre: rue de Stassart, 36 B-1050 Brussels

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Foreword

This document (EN 12697-39:2004) 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of an identical text or by endorsement, at the latest by April 2005, and conflicting national standards shall be withdrawn at the latest by April 2005.

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

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 Marshall specimens*

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 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 a 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*

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

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

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

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

No existing European Standard is superseded.

WARNING — The temperature of the oven and the different accessories is extremely high during the ignition method. Special care shall be taken when handling the equipment and the samples baskets etc. should be placed, shielded and marked in a way that any unpremeditated contact is avoided.

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

1 Scope

This document describes a test method for the determination of the binder content of samples of bituminous mixtures by ignition. As such, it is an alternative to the more traditional method of extracting the binder using solvents. The method can be used for evaluation of mixture composition because the remaining aggregate can be used for determining aggregate gradation and density provided excessive breakdown of the aggregate particles does not occur at the temperature reached. The results can be used for process control or checks on the compliance of mixtures. However, the need for calibration of a mixture or its component materials before an analysis can be carried out makes this method easier to use with regularly used mixtures rather than with an extensive range of different mixtures from different aggregate sources. The test method is equally suitable for the analysis of mixtures containing unmodified or modified binders because the method has to be calibrated for each mixture being checked when calibration on mixtures is used. In case of doubt/dispute, the determination of the calibration value based on laboratory-prepared bituminous mixtures (see A.1 and A.2) is the reference method.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 12597, *Bitumen and bituminous binders – Terminology.*

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

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

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.*

ISO 5725 (all parts), *Accuracy (trueness and precision) of measurement methods and results.*

3 Terms and definitions

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

3.1
binder
covers both bitumen and bituminous binder as described in EN 12597

3.2
corrected binder content
calculated binder content after correction by the calibration value in order to compensate for components in the asphalt material itself that, due to the high temperatures during ignition, could give rise to misinterpretations

3.3
calibration value
mean difference between the actual and measured binder contents for a particular bituminous mixture, in per cent, as measured from three samples

NOTE 1 The calibration value is a specific, material-dependent value that normally is due to a loss of mass during the ignition from constituents in the mixture other than the binder. The principle of the binder content by ignition is based upon a pre-determination of the corrections for the constituents used in the mixture, primarily the aggregate.

NOTE 2 Mineral aggregate will show varying loss of mass during the test depending on their origin (petrographic composition). Examples of components that give rise to high corrections are limestone, hydrated lime and cellulose fibres.

NOTE 3 Methods for determining the calibration values are given in Annex A (normative).

3.4
target temperature
temperature to which the furnace would raise the sample in the test without the rise in temperature due to the exothermic reaction of burning the binder

NOTE The target temperature (usually 540 °C) is determined during the calibration.

3.5
test completion time
time from when the temperature in the furnace returns to the target temperature after the initial rise above that temperature to when the test is completed with the sample having nominally reached constant mass

NOTE If a furnace with an internal balance is used, the completion time is when the loss of mass between individual readings taken at 1 min intervals for three consecutive minutes is less than a constant mass limit. If a furnace without an internal balance is used, the completion time is when the change in the mass of the sample after further ignition for 15 min is less than a constant mass limit.

3.6
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 reflected 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 object. Quantitative measures of precision depend critically on the stipulated conditions. Repeatability and reproducibility conditions are particular sets of extreme conditions.

3.7
repeatability
precision under repeatability conditions

3.8
repeatability conditions
conditions where 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

3.9
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 is r.

3.10
reproducibility
precision under reproducibility conditions

3.11
reproducibility conditions
conditions where test results are obtained with the same method on identical test items in different laboratories with different operators using different equipment

3.12
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 is *R*.

3.13
single test result
value obtained by applying the standard test method fully, once to a single specimen

NOTE The single test result may be the mean of two or more observations or the result of a calculation from a set of observations as specified by the standardised test method.

4 Principle

The test method determines the binder content of bituminous mixtures by ignition of the mixture in a furnace. The binder content is obtained by a calculation that includes a calibration term. Calibration terms are determined for particular mixtures or aggregates. Two test methods are described; Method A utilises a furnace with an internal balance; Method B permits the use of a furnace and external balance. Re-determination (re-calibration or re-calculation depending on the calibration method chosen from Annex A) shall be undertaken for each change in the mixture, including changes in the constituent materials or their proportions.

NOTE 1 The ignition process should have a controlled rise in temperature in order to avoid excessive heating of the mineral aggregate that can break down the aggregate particles depending on the petrographic composition.

NOTE 2 The calculation of the calibration value based on parallel analysis with extraction methods (A.1 and A.3) is appropriate for mixtures for which the intended proportions of the constituent materials are not known.

5 Apparatus

5.1 Furnace

5.1.1 A furnace capable of burning all the binder with the features detailed in **5.1.2** to **5.1.6** for Method A and **5.1.2** to **5.1.4** for Method B.

NOTE 1 The temperature required to burn all the binder will depend on the technology used and is determined as part of the calibration procedure (see Annex A).

NOTE 2 The furnace should not have to operate at its maximum capacity in order to allow flexibility and to ensure long service.

5.1.2 A sample chamber having an internal capacity capable of taking the sample without touching the sides and with the maximum dimension not greater than twice the minimum dimension.

NOTE Larger chamber sizes may expedite testing by allowing larger sample basket(s) and thus the material to be tested can be placed in thinner layers.

5.1.3 An automatic lock that shall not allow the door to be opened until the completion of the test procedure, and a warning system to indicate the end of the pre-programmed temperature cycle.

5.1.4 System for reducing furnace emissions. The furnace shall be vented into a hood or to the outside and, when properly set up, shall have no noticeable odours escaping into the laboratory. The furnace shall have a fan with the capability to pull sufficient air through the furnace to expedite the test and to reduce the escape of smoke into the laboratory.

NOTE 1 The method for reducing furnace emissions can comprise a filter and a post combustion chamber that is designed to eliminate the toxic residues produced by burning the binder.

NOTE 2 If mechanical ventilation is used, the air flow should be adjusted so as not to affect the operation of the equipment (e.g. forced air extraction system may result in a loss of fines and the generation of fumes from the furnace).

5.1.5 An internal balance, capable of detecting mass variations of ± 0,1 g in the sample within the baskets. The balance shall be thermally isolated from the furnace chamber.

5.1.6 Data collection system and a warning system. The warning system shall be capable of being set to a value such that the loss in mass between individual readings taken at 1 min intervals for three consecutive minutes at end of the pre-programmed temperature cycle is no higher than the values stated in Table 1.

5.2 Metal baskets manufactured from perforated sheet of tempered stainless steel or other suitable material that permits adequate air flow through the sample and retains the majority of the sample throughout the test. The dimensions shall be specified by the furnace manufacturer to provide the maximum surface area for the sample whilst still providing sufficient room to safely load and unload the sample. Sets of baskets shall be nested.

5.3 Catch pan of stainless steel with dimensions sized to accommodate the metal baskets specified in **5.2**.

5.4 Oven with convection or forced draft, capable of maintaining a temperature of (110 ± 2) °C in the vicinity of the samples.

5.5 External balance capable to weigh the mass of trays plus test sample according to Table 1 to ± 0,1 g.

5.6 Safety equipment, including safety glasses or face shield, high temperature gloves, and long sleeved jacket.

5.7 Heat-resistant surface, capable of withstanding 650 °C and able to act as a heat sink that can speed the cooling of the sample baskets, and protective cage, capable of completely surrounding the sample baskets and preventing accidental physical contact with them.

NOTE An appropriate sign warning of the danger of `Very Hot Surfaces' should be attached to the protective cage.

5.8 Pan, larger than the sample basket(s), for transferring samples after ignition.

5.9 Spatulas

5.10 Bowls

5.11 Wire brushes

6 Preparatory treatment of laboratory samples of bituminous mixtures

6.1 Sampling shall be performed in accordance with EN 12697-28 to achieve a sample size as given in Table 1. The mass of the sample in the baskets during the test shall be the same as the mass used for the calibration to ± 100 g.