SLOVENSKI STANDARD
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Bitumenske zmesi - Preskusne metode - 39. del: Ugotavljanje deleža veziva s sežigom

Bituminous mixtures - Test methods - Part 39: Binder content by ignition

Asphalt - Prüfverfahren - Teil 39: Bindemittelgehalt durch Thermoanalyse

Mélanges bitumineux - Méthodes d'essai - Partie 39 : Teneur en bitume

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Bituminous mixtures - Test methods - Part 39: Binder content by ignition

Mélanges bitumineux - Méthodes d'essai - Partie 39 : Teneur en bitume par calcination

Asphalt - Prüfverfahren - Teil 39: Bindemittelgehalt durch Thermoanalyse

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COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Contents

European foreword

This document (EN 12697-39:2020) 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of an identical text or by endorsement, at the latest by August 2020, and conflicting national standards shall be withdrawn at the latest by August 2020.

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 supersedes EN 12697-39:2012.

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

— the title no longer makes the method exclusively for hot mix asphalt;

— [ge] editorial update according to current standard template;

— [ge] references added to each formula in related text;

— [ge] NOTEs modified or adjusted to normal text where appropriate according to ISO/IEC Directives – Part 2:2016, 24.5;

— [Clause 2] titles for test methods EN 12697-series corrected.

A list of all parts in the EN 12697 series can be found on the CEN website.

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

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, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the 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, either on the complete mixture or on each of its component materials separately, 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 documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 — Part 1: Soluble binder content*

EN 12697-14, *Bituminous mixtures — Test methods — Part 14: Water content*

EN 12697-27, *Bituminous mixtures — Test methods — Part 27: Sampling*

EN 12697-28, *Bituminous mixtures — Test methods — Part 28: Preparation of samples for determining binder content, water content and grading*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at https://www.iso.org/obp/ui

3.1
binder

covers 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 to entry: The calibration value is a specific, material-dependent value that normally results from 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 to entry: Mineral aggregates will show varying losses 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 to entry: 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 1 to entry: 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 1 to entry: 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.

4 Principle

The test method determines the binder content of bituminous mixtures by ignition of the mixture in a furnace.

The corrected binder content is obtained by a calculation that includes a calibration term. Calibration terms are determined for particular asphalt mixtures or aggregates. Two test methods are described: Method A utilizes 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 significant change in the mixture, including changes in the constituent materials or their proportions.

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

NOTE 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 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 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).

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

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

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

5.1.3 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, by which the furnace shall be vented into a hood or to the outside and which, when properly set up, shall permit 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 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.

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

5.1.5 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, which 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 not 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 while still providing sufficient room to safely load and unload the sample. The baskets shall be capable of being nested.

5.3 Catch pan, made 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 ± 5) °C in the vicinity of the samples.

5.5 External balance, capable of weighing the mass of trays plus the catch pan and the 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.

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.

Table 1 — Size of sample

Nominal maximum aggregate size mm	Mass of sample g	Maximum constant mass limit g
4	1 000 to 1 400	0,15
5,6 or 6,3 or 8 or 10	1 000 to 1 600	0,15
11,2 or 12,5 or 14 or 16	1 000 to 1 700	0,20
20 or 22,4	1 000 to 2 400	0,25
31,5	1 000 to 3 000	0,30
40 or 45	1 000 to 4 000	0,40

When the mass of the test specimen exceeds the capacity of the equipment used, the test specimen may be divided into suitable increments, tested, and the results combined by weighted average for calculation of the corrected binder content.

NOTE Large samples of fine mixtures tend to result in incomplete ignition of the binder.

6.2 The sample shall be dried to constant mass in an oven at (110 ± 5) °C unless it is known that the sample does not contain water. Alternatively, the water content shall be determined in accordance with EN 12697-14 for later correction.

NOTE If a damp sample is tested, the water content will be driven off and could be counted as binder if no correction is made.

7 Procedure

7.1 General

7.1.1 Obtain the calibration value in accordance with Annex A for the specific mixture to be analysed either directly or from records. The calibration procedure (or re-calculation for calibration based on dry analysis of constituent aggregate) shall be carried out with a mixture before undertaking any acceptance testing, and repeated at regular intervals. It shall also be carried out each time there is a significant change in the mixture design. A significant change is classified as being:

- a change of source for any component material (particularly recycled asphalt pavement); and/or
- a change in the proportion of the component materials (other than the binder or recycled asphalt pavement) by 10 % or more of their original proportion; and/or
- a change in the proportion of any recycled asphalt pavement by 5 % or more of the original proportion; and/or
- a change of target binder content by 0,5 % or more.

NOTE 1 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.

NOTE 2 The analysis method by ignition can be affected by the type of aggregate in the mixture.

NOTE 3 Mineral aggregate (and organic fibres) will show varying losses 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. The calibration value for these components can be measured by determination on a bitumen/filler mortar (A.4).

7.1.2 The test shall be carried out using the same equipment used in the calibration, including furnace and sample baskets.

7.1.3 Pre-heat the ignition furnace. Record the furnace temperature at the start of the test.

7.1.4 Continue the procedure in accordance with either 7.2 (Method A) for furnaces with an internal balance or 7.3 (Method B) for furnaces without an internal balance.

NOTE When required, the aggregate grading can be determined in accordance with Annex B after the binder content has been obtained.

7.2 Test procedure for furnace with internal balance (Method A)

7.2.1 Weigh the sample at a temperature of (110 ± 5) °C on the external balance and record the sample mass $W_{s,w}$, in grams, to the nearest 0,1 g.

7.2.2 Place the available sample baskets in the catch pan in turn and without any appreciable loss of temperature. Distribute the relevant proportion of the sample as evenly as possibly over each sample basket before placing the next. Take care to keep the material away from the edges of the sample baskets. Use a spatula or trowel to level the sample.

To ensure consistent results, the temperature of the test portion after determining $W_{s,w}$ should not be allowed to cool from 110 °C for more than 3 min before loading into the furnace.

7.2.3 Weigh to 0,1 g and record the mass of the sample, sample basket(s) and catch pan W_{t+s}, in grams, on the external balance.

7.2.4 Place the sample baskets and catch pan in the furnace ensuring that they are not in contact with either the furnace wall or any temperature probe or other protrusion inside the furnace. Run the appropriate ignition programme after the oven has been pre-heated according to the manufacturer's instructions.

7.2.5 Allow the test to continue until the balance indicator displays show that the sample mass has stabilized and so that the range of 3 consecutive weighing, taken at 1 min intervals, is within the constant mass limit in Table 1. Remove the sample baskets, placing them on the heat-resistant surface, immediately covering with the heat guard and allow to cool to approximately room temperature to permit safe handling.

NOTE 1 Failure of the furnace scale to stabilize might indicate that the sample baskets are in contact with the furnace wall.

NOTE 2 The cooling time for samples removed from the furnace is dependent primarily on sample size and dispersion in the baskets. The time taken to cool to approximately room temperature is unlikely to be less than 30 min.

7.2.6 Record the loss of mass W_{loss}, in grams, from the internal balance. Then weigh and record the mass of the sample, sample baskets and catch pan W_{t+a}, in grams, to 0,1 g on the external balance.

W_{t+a} need not be measured if *B* is to be calculated using W_{loss} directly, but *B* should be calculated using W_s and W_a whenever used for reference purposes because the internal balance measures the mass loss at elevated temperature.

7.3 Procedure for furnace without internal balance (Method B)

7.3.1 Weigh to 0,1 g and record the mass of the sample basket(s) and catch pan W_t, in grams, at room temperature. Place the sample basket(s) in the catch pan and evenly distribute the specimen in the basket(s). Weigh to 0,1 g and record the mass of the sample, sample basket(s) and catch pan W_{t+s}, in grams.

7.3.2 Heat the sample in the furnace at the test temperature ± 5 °C for the time determined during the calibration ± 5 min.

7.3.3 Remove the sample from the furnace and allow it to cool to below 35 °C. Weigh to 0,1 g and record the mass of the sample, sample basket(s) and catch pan W_{t+a}, in grams.

7.3.4 Return the sample back into the furnace. After the furnace reaches the set point temperature, heat the sample for a further 15 min. Repeat 7.3.3 to determine the mass of the sample after the extended ignition period.

7.3.5 Repeat 7.3.4 until the change in the mass of the sample after 15 min of further ignition is less than the constant mass limit in Table 1.