SLOVENSKI STANDARD
oSIST prEN 16346:2022

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Bitumen in bitumenska veziva - Ugotavljanje stopnje stabilnosti in neposredne obstojnosti kationskih bitumenskih emulzij

Bitumen and bituminous binders - Determination of breaking behaviour and immediate adhesivity of cationic bituminous emulsions

Bitumen und bitumenhaltige Bindemittel - Bestimmung der Brechzeit und des kurzfristigen Haftverhaltens von kationischen Bitumenemulsionen

Bitumes et liants bitumineux - Détermination du comportement à la rupture et de l'adhésivité immédiate des émulsions bitumineuses cationiques

Ta slovenski standard je istoveten z: **prEN 16346**

ICS:

75.140	Voski, bitumni in drugi naftni proizvodi	Waxes, bituminous materials and other petroleum products
91.100.50	Veziva. Tesnilni materiali	Binders. Sealing materials

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English Version

Bitumen and bituminous binders - Determination of breaking behaviour and immediate adhesivity of cationic bituminous emulsions

Bitumes et liants bitumineux - Détermination du comportement à la rupture et de l'adhésivité immédiate des émulsions bitumineuses cationiques

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This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 336.

If this draft becomes a European Standard, CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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

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European foreword

This document (prEN 16346:2022) has been prepared by Technical Committee CEN/TC 336 “Bituminous Binders”, the secretariat of which is held by AFNOR.

This document is currently submitted to the CEN Enquiry.

This document will supersede CEN/TS 16346:2012.

The main changes with respect to the previous document are listed below:

— change of the name of the document, new version does not include the 2/4 aggregate size as the document now allows the assessment of various aggregate sizes;

— instead of defining the amount of emulsion which is to be mixed with aggregate, the residual binder amount was defined (8.2.3.);

— assessment of the breaking behaviour (3.3) is now evaluated via wash-out water appearance in up to eight beakers (8.3.1.) instead of using a slow stream of water;

— immediate adhesivity (3.4) and immediate adhesivity after drying (3.5) are now assessed in a quantitative way instead of qualitative way by using nomograms;

— time when the immediate adhesivity (3.4) is assessed is fixed at 10 minutes (8.3.1);

— new procedure for evaluating the adhesivity after a certain rest time was introduced. This procedure is named “immediate adhesivity after drying” (3.5);

— aggregate (5.2) and washed-out aggregate (8.3.1, 8.4.4) is to be dried until a constant mass (3.6) is reached instead of a drying period of about 2 hours;

— new aggregate sizes were introduced (5.1) to have a higher level of versatility;

— the maximum mixing time to obtain the full breaking (3.1) is restricted to 45 seconds (8.2.4);

— a possibility to use a camera for depicting the actual state of wash-out water in beakers was introduced (8.3.1, 8.4.4);

— pictures depicting different results of the assessment of the breaking behaviour (3.3) were included (Clause 9).

1 Scope

This document specifies a method for the determination of the breaking and immediate adhesivity behaviour of cationic bituminous emulsions in contact with aggregate. The method applies to emulsions used for surface dressing and similar applications and can be used for formulation as well as for production control purposes.

WARNING — The use of this document can involve hazardous materials, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this document to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

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 58, *Bitumen and bituminous binders - Sampling bituminous binders*

EN 12594, *Bitumen and bituminous binders - Preparation of test samples*

EN 13043, *Aggregates for bituminous mixtures and surface treatments for roads, airfields and other trafficked areas*

EN 1428, *Bitumen and bituminous binders - Determination of water content in bituminous emulsions - Azeotropic distillation method*

EN 1431, *Bitumen and bituminous binders - Determination of residual binder and oil distillate from bitumen emulsions by distillation*

EN 16849, *Bitumen and bituminous binders - Determination of water content in bituminous emulsions - Method using a drying balance*

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:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
full breaking
stage at which, during the mixing process between aggregate particles and emulsion conducted as specified in (8.2), all aggregate particles have agglomerated into a single compact mass

3.2
breaking time
time in seconds, counted from the start of the mixing process, until the full breaking (3.1) is reached

3.3
breaking behaviour
appreciation of the actual degree of breaking of the emulsion after the mixture has reached the full breaking stage

Note 1 to entry: The breaking behaviour is measured by the number of successive washings of the final mixture that are necessary until the water runs clear.

3.4
immediate adhesivity
ability of the binder from a bituminous emulsion to resist the action of water just after aggregate coating

Note 1 to entry: Immediate adhesivity is assessed in a quantitative way (loss of mass after washing with water).

3.5
immediate adhesivity after drying
ability of the binder from a bituminous emulsion to resist the action of water after aggregate coating and after a certain resting time of the coated aggregate was applied before washing

Note 1 to entry: Immediate adhesivity after drying is assessed in a quantitative way (loss of mass after washing with water).

3.6
constant mass
a weight which, on subsequent weighing after drying for at least one hour, does not change by more than 0,1 %

4 Principle

Prescribed quantities of emulsion and aggregate are mixed under specified conditions. The necessary time to obtain a single agglomerated mass is the measure of the breaking time of the emulsion (3.2). If full breaking (3.1) is not obtained after 45 s, mixing is stopped. After 10 minutes, the final mixture is washed with water and the percentage of residual binder remaining on aggregates is assessed through weighing (3.4).

If needed or required, the procedure is repeated while spreading coated aggregate evenly and applying the washing procedure after the curing period of 60 minutes (3.5). This procedure is usually performed when it is not possible to obtain clear water after 8 successive washings or when the evolution of adhesivity over time is studied. If not specifically requested, this part is optional.

Depending on the result, additional tests may be conducted at shorter or longer curing periods, e.g. 30 or 90 minutes. Such deviations shall be noted in the report.

5 Reagents and material

5.1 Aggregate, being either reference aggregate or aggregate to be used on a specific job site. Reference aggregate, which passes through a sieve having a mesh size of 4 mm or 5,6 mm and is retained on a sieve having a mesh size of 2 mm (sieve sizes belonging to the “basic set plus set 1” are specified in EN 13043). Alternatively, aggregate which passes through a sieve having a mesh size of 6,3 mm or 8 mm and is retained on a sieve having a mesh size of 2 mm, 4 mm or 5 mm (sieve sizes belonging to the “basic set plus set 2 and set 1” are specified in EN 13043) can be utilized.

NOTE Each country can define petrographically its own reference aggregates, for instance, in a national specification document.

It is not allowed to use aggregate in its job site condition, e.g. containing moisture, fines, being contaminated, etc.

5.2 Water, it is recommended to use distilled or deionised water conforming to grade 3 of EN ISO 3696:1995 [2].

5.3 Cleaning agents, conventionally used in a laboratory.

6 Apparatus

6.1 Mixing bowls (at least 2), semi-spherical, acid resistant metal, diameter (150 ± 5) mm.

6.2 Spoon-shaped, rigid mixing **spatula.**

6.3 Timer, accurate to at least 0,5 s over 60 s.

6.4 Beakers (at least 9), approximately 500 ml capacity.

6.5 Balance, of sufficient capacity, with a maximum permissible error of ± 0,1 g.

6.6 Ventilated oven, capable of maintaining a temperature of (110 ± 5) °C.

6.7 Rigid metal plates, of sufficient dimensions to allow spreading of 200 g of aggregate in the form of a monolayer (5.1).

6.8 Sieves, stainless or brass, with a mesh size which can retain the used aggregate.

6.9 Camera, camera with a sufficient image resolution (use of a camera is optional).

7 Sampling

Sample the emulsion to be tested in accordance with EN 58. Prepare the test samples in accordance with EN 12594.

8 Procedure

8.1 Prerequisite

Carry out the procedure under normal laboratory conditions (23 ± 5) °C. Very viscous emulsions (class 12 as specified in EN 13808) may be tested at a temperature up to 40 °C but only if this is necessary to adequately carry out the procedure. The actual emulsion temperature shall be mentioned in the test report (11, b).

Wash aggregate with water and dry it in a ventilated oven (6.6) at (110 ± 5) °C until constant mass (3.6) is reached. Let the aggregate cool down to a laboratory temperature. Sieve the aggregate as to obtain the testing aggregate size (5.1).

Determine the water content of the emulsion W according to EN 1428, EN 1431 or EN 16849. Express W to the nearest 0,1 %.

The test procedures described in 8.3 and 8.4 shall be repeated on a second sample of the same emulsion and of the same aggregate.

8.2 Mixing procedure

8.2.1 Take a dry and clean mixing bowl (6.1) and weigh it. Record its mass m_{C0} to the nearest 0,1 g.

Take a dry and clean mixing spatula (6.2) and weigh it. Record its mass m_{S0} to the nearest 0,1 g.

Weigh the mixing bowl and the spatula and record the total mass m_0 to the nearest 0,1 g.

8.2.2 Weigh (200 ± 5) g of aggregate (5.1) into the mixing bowl. Record the aggregate mass m_G to the nearest 0,1 g. Dig a conical shaped cavity in the centre of the aggregate heap.

8.2.3 Pour rapidly, into the previously formed cavity, an amount of emulsion m_E corresponding to (13,5 ± 0,7) g of the residual binder.

Record the mass of emulsion used m_E to the nearest 0,1 g.

The amount m_E of emulsion, in grams, is calculated using Formula (1):

$$m_E = 100 \times m_B / (100 - W) \quad (1)$$

where:

m_B is the mass of the residual binder, in grams, required for the test;

W is the water content of the emulsion, in %, as defined (8.1).

NOTE For emulsion containing 69,0 % of the residual binder, the calculated amount of the emulsion allowing requested quantity of the residual binder of (13,5 ± 0,7) g is (19,6 ± 1,0) g.

8.2.4 Start immediately the timer (6.3) and the mixing process using the spatula (6.2). Mixing shall be done while holding the mixing bowl inclined to about 45° in one hand and the spatula in the other. The motion of the spatula shall start from the wall of the mixing bowl and drag the aggregate towards its centre in a circular movement starting from the top. Emulsion and aggregate shall be mixed thoroughly.

Continue mixing without interruption until full breaking (3.1) is obtained and record immediately the breaking time in seconds (3.2). If full breaking (agglomeration into a single compact mass) is not obtained after 45 seconds, mixing shall be stopped and this shall be mentioned in the test report (11) by specifying "breaking time > 45 s".

8.2.5 Remove the adhering aggregates as much as possible from the mixing spatula and put them back into the mixing bowl.

8.3 Determination of the breaking behaviour and immediate adhesivity

8.3.1 (10 ± 0,5) minutes after the start of the timing device, gently fill the mixing bowl with 500 ml of water (5.2). The spout of the beaker should be at the top edge of the mixing bowl. Once filled, wait for (5 ± 1) s, then pour the water from the mixing bowl into a second beaker while passing it over a sieve able to retain the tested aggregate size (6.8). Re-introduce the aggregate possibly retained on the sieve into the mixing bowl.

Repeat this procedure until the wash-out water gets clear and, by default, up to eight successive wash-outs. Record the number of washing N_0 to obtain clear water (9). If the wash-out water is not clear after 8 successive washings it is possible to test immediate adhesivity after drying (3.5) when required or needed.

It is recommended to provide eight beakers in order to gather as much information as possible on the evolution of the wash-out water. It may be sufficient to compare a given beaker with a reference beaker with clear water.

Take care not to lose any aggregate particles (coated or uncoated) during the different test phases leading to the drying phase.

8.3.2 Dry the mixing bowl inclusive aggregate-emulsion mixture and the spatula in the ventilated oven (6.6) at (110 ± 5) °C until a constant mass is reached. Let it cool down to a laboratory temperature and weigh all mass *(m_1)* to the nearest 0,1 g.

Determine the mass % of adhering bituminous binder *(q_{a0})* according to Formula (2):

$$q_{a0} = \frac{m_1 - (m_0 + m_G)}{m_E \times \frac{(100 - W)}{100}} \times 100 \quad (2)$$

where:

m_0 is the combined mass of the mixing bowl and the spatula in grams, as defined in 8.2.1;

m_G is the mass of dry aggregates, in grams, as defined in 8.2.2;

m_E is the mass of emulsion, in grams, as defined in 8.2.3;

m_1 is the mass of the mixing bowl with the coated aggregates and the spatula after drying in a ventilated oven, in grams, as defined in 8.3.2;

W is the water content of the emulsion, in %, as defined in 8.1.

Express quantitative adhesivity q_{a0}, as a mass percentage, to the nearest 0,1 %.

NOTE Binder that adheres to the mixing bowl and the spatula does not significantly influence the level of the immediate adhesivity. Therefore, this binder is not considered in the formula.

8.4 Determination of the breaking behaviour and immediate adhesivity after drying

This part of the procedure is optional and can be performed if needed or required. This is usually the case when the procedure described in 8.3 does not lead to clear water after 8 wash-outs ($N_0 > 8$).

8.4.1 Take a dry and clean mixing bowl (6.1) and weigh it. Record its mass *(m_{C0})* to the nearest 0,1 g.

Take a dry and clean mixing spatula (6.2) and weigh it. Record its mass *(m_{S0})* to the nearest 0,1 g.

Take a dry and clean metal plate (6.7) and weigh it. Record its mass *(m_{P0})* to the nearest 0,1 g.

Weigh together the mixing bowl, plate and spatula and note the total mass *(m_{00})* to the nearest 0,1 g.

Then perform the operations described in paragraphs 8.2.2 to 8.2.4.

8.4.2 Immediately after mixing pour the coated aggregate particles on the rigid metal plate (6.7) and using the mixing spatula spread them evenly in the form of a monolayer of material.

8.4.3 (60 ± 3) minutes after starting the timer, put the coated aggregate particles back into the mixing bowl with the aid of the mixing spatula while taking care not to lose any aggregate particles.

8.4.4 Apply the successive wash-out procedure as described in 8.3.1. Record the number of washings to obtain clear water *(N_1)* (9).