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Bitumen and bituminous binders — Determination of adhesivity of cut-back and fluxed bituminous binders by water immersion test — Aggregate method

National foreword

This British Standard is the UK implementation of EN 15626:2016. It supersedes BS EN 15626:2009 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee B/510/19, Bitumen and related products.

A list of organizations represented on this committee can be obtained on request to its secretary.

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ISBN 978 0 580 91270 2

ICS 93.080.20

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 July 2016.

Amendments issued since publication

Date	Text affected
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EUROPEAN STANDARD

EN 15626

NORME EUROPÉENNE

EUROPÄISCHE NORM

July 2016

ICS 93.080.20

Supersedes EN 15626:2009

English Version

Bitumen and bituminous binders - Determination of adhesivity of cut-back and fluxed bituminous binders by water immersion test - Aggregate method

Bitumes et liants bitumineux - Détermination de l'adhésivité des liants bitumineux fluidifiés et fluxés par l'essai d'immersion dans l'eau - Méthode utilisant des granulats

Bitumen und bitumenhaltige Bindemittel - Bestimmung des Haftverhaltens von verschnittenen und gefluxten bitumenhaltigen Bindemitteln bei Wasserlagerung - Verfahren mit Gesteinskörnung

This European Standard was approved by CEN on 9 April 2016.

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CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

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European foreword

This document (EN 15626:2016) has been prepared by Technical Committee CEN/TC 336 “Bituminous binders”, the secretariat of which is held by AFNOR.

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of an identical text or by endorsement, at the latest by January 2017, and conflicting national standards shall be withdrawn at the latest by January 2017.

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

This document supersedes EN 15626:2009.

In comparison to EN 15626:2009, the following main changes have been made:

- a sentence on environmental issues in the WARNING was added under Clause 1 (Scope);
- normative references were updated throughout the text (EN 13357 was updated as EN 12846-2, EN 13702-1 as EN 13702, EN 14896 as EN 13302, and prEN 15322 as EN 15322).
- NOTES in 5.1, 8.1.3 and Clause 9 were converted into plain text.

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

1 Scope

This European Standard specifies a method for the determination of the adhesivity of cut-back and fluxed bituminous binders coated onto aggregate when immersed in water.

WARNING — The use of this document may involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use. For environmental reasons and to reduce emissions to air, water and soil, it is recommended to limit the use of products, solvents and energy to the minimum required for a valid test result.

2 Normative references

The following referenced 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 58, *Bitumen and bituminous binders — Sampling bituminous binders*

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

EN 12846-2, *Bitumen and bituminous binders — Determination of efflux time by the efflux viscometer — Part 2: Cut-back and fluxed bituminous binders*

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

EN 13302, *Bitumen and bituminous binders — Determination of dynamic viscosity of bituminous binder using a rotating spindle apparatus*

EN 13702, *Bitumen and bituminous binders — Determination of dynamic viscosity of modified bitumen by cone and plate method*

EN 15322, *Bitumen and bituminous binders — Framework for specifying cut-back and fluxed bituminous binders*

EN ISO 3696, *Water for analytical laboratory use — Specification and test methods (ISO 3696)*

3 Terms and definitions

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

3.1 adhesion

ability of a binder to coat the surface of an aggregate and to remain bonded over time in the presence of water

3.2 adhesivity

qualitative assessment of the measurement of adhesion

3.3

ambient temperature

temperature between 18 °C and 28 °C

4 Principle

The binder is mixed thoroughly with a dry and clean reference aggregate under specified temperature conditions. After specified pre-conditioning procedures depending on the viscosity of the binder, the mixture is immersed in water in a glass container. After a given time under specified conditions, the percentage of the aggregate surface covered with binder is assessed visually.

5 Reagents and materials

5.1 Reference aggregate, as light in colour as possible, or aggregate from a specific job site, which either passes through a sieve having a mesh size of 10 mm and is retained on a sieve having a mesh size of 6,3 mm (sieve sizes belonging to the “basic set plus set 2” sizes specified in EN 13043), or passes through a sieve having a mesh size of 11 mm and is retained on a sieve having a mesh size of 8 mm (sieve sizes belonging to the “basic set plus set 1” sizes specified in EN 13043).

Each country should define petrographically its own reference aggregates, for instance, in a national informative annex.

5.2 Water, distilled or deionized, conforming to EN ISO 3696, water quality 3.

5.3 Cleaning agents, conventionally used in a laboratory.

6 Apparatus

6.1 Ventilated oven, capable of maintaining a temperature of (60 ± 3) °C.

6.2 Ventilated oven, capable of maintaining a temperature at ± 5 °C for temperatures ranging from 30 °C to 150 °C.

Temperature shall be checked **in the surroundings** of the sample.

6.3 Spatula.

6.4 Heat resistant dishes, diameter approximately 150 mm to 200 mm.

6.5 Stop watch, accurate to at least 1 s over 60 s.

6.6 Two beakers, approximate 400 ml capacity.

6.7 Watch glasses, diameter approximately 100 mm to 150 mm.

6.8 Balance, capable of reading up to 500 g, and enabling weighing to ± 1 g.

6.9 Measuring cylinder, 250 ml to 500 ml capacity.

6.10 Thermometers, of adequate range, allowing to measure the specified temperatures with an accuracy of ± 1 °C.

6.11 Heating plate, or any other heating device allowing to maintain temperature without overheating.

7 Sampling

The material under test shall be sampled in accordance with EN 58 and prepared in accordance with EN 12594.

8 Procedure

8.1 Preparation of aggregates and binders

8.1.1 Wash the aggregate (5.1) with water (5.2) and dry it in the ventilated oven (6.2) for a minimum of 12 h at (110 ± 5) °C.

8.1.2 The quantities of aggregates needed for the tests to be carried out under 8.2 and 8.3 shall be weighed into adequate containers by batches of (200 ± 2) g and/or (400 ± 4) g.

Depending on the binder to be tested, bring the aggregates and the dish(es) to be used for the mixing procedure to the temperature indicated in Table 1 and maintain this temperature for about 2 h.

8.1.3 A binder sample of a minimum amount of 200 g shall be prepared in accordance with EN 12594 and brought to the temperature indicated in Table 1. The efflux time shall be determined in accordance with EN 12846-2 and dynamic viscosity at 60 °C in accordance with EN 13302 or EN 13702. Closed containers shall be used and care shall be taken to avoid local overheating and any loss of volatile constituents.

Leave the binder in the oven for the minimum time to ensure that it reaches the temperature given in Table 1.

Binder formulated with an adhesion improver should be tested under the representative conditions of its use, i.e. after one or several days of storage in a closed container at the typical storage temperature for that binder.

Table 1 — Heating temperatures

Binder	Viscosity classes as specified according to EN 15322	Heating temperature for the binder and the aggregates °C ± 5 °C
Low viscosity cut-back and fluxed bituminous binders of low viscosity	Efflux time 4 mm 25 °C	
	15 s to 25 s	23 (ambient)
	26 s to 50 s	30
	51 s to 100 s	40
	101 s to 200 s	50
	Efflux time 10 mm 25 °C	
	15 s to 50 s	75
	51 s to 150 s	80
	151 s to 300 s	90
	301 s to 500 s	95
	Efflux time 10 mm 40 °C	
	50 s to 100 s	100
101 s to 200 s	110	
201 s to 400 s	115	
401 s to 500 s	120	
High viscosity cut-back and fluxed bituminous binders of high viscosity	Dynamic viscosity at 60 °C	
	10 Pa s to 30 Pa s	125
	31 Pa s to 50 Pa s	130
	51 Pa s to 80 Pa s	135
	> 80 Pa s	140

8.2 Procedure for “high viscosity” binders

8.2.1 This procedure shall be applied for cut-back and fluxed bituminous binders with an efflux time 10 mm 40 °C (EN 12846-2) higher than 500 s or with a dynamic viscosity at 60 °C (EN 13302 or EN 13702) higher than 10 Pa · s.

8.2.2 Pour (10 ± 1) g of binder prepared as indicated in 8.1.3 into the pre-heated dish and place the dish onto the heating plate (6.11) maintained at the temperature specified in 8.1.3. Pour (200 ± 2) g of aggregates prepared as indicated in 8.1.2 onto the binder and mix with a spatula until coating is completed, but no longer. The dish shall remain on the heating plate during mixing. Remove the dish from the heating plate and ensure by periodic stirring that aggregates remain properly coated until binder drainage stops. Once at ambient temperature put the mix in a beaker (6.6) and cover it with a watch-glass (6.7); let it rest for (30 ± 5) min.

8.2.3 Pour approximately 300 ml of water (5.2) that has been pre-heated to $(60 \pm 3)^\circ\text{C}$ into the beaker; cover the beaker with a watch-glass and place it in the ventilated oven at $(60 \pm 3)^\circ\text{C}$ for (20 ± 4) h (6.1).

8.2.4 Assess the surface coated with the film of binder according to the grading scheme given in Clause 9.

8.3 Procedure for “low viscosity” cut-back and fluxed bituminous binders

8.3.1 This procedure is to be applied for cut-back and fluxed bituminous binders with an efflux time 10 mm 40°C (EN 12846-2) lower or equal to 500 s or with a dynamic viscosity at 60°C (EN 13302 or EN 13702) lower or equal to 10 Pa · s.

8.3.2 Pour (20 ± 2) g of binder prepared as indicated in 8.1.3 into the pre-heated dish and place the dish onto the heating plate (6.11) maintained at the temperature specified in 8.1.3. Pour (400 ± 4) g of aggregates prepared as indicated in 8.1.2 onto the binder and mix with a spatula until coating is completed but no longer. The dish shall remain on the heating plate during mixing. Remove the dish from the heating plate and ensure by periodic stirring that aggregates remain properly coated until binder drainage stops.

Once at ambient temperature, separate the mix into approximately two equal parts:

8.3.3 Put the first part in a beaker (6.6) and cover it with a watch-glass; let it rest for (30 ± 5) min. Then pour approximately 300 ml of water (5.2) at ambient temperature into the beaker; cover the beaker with a watch-glass and let it rest for (20 ± 4) h at ambient temperature.

8.3.4 Spread the second part on a watch-glass (6.7) and place it in the ventilated oven (6.1) for (24 ± 1) h at $(60 \pm 3)^\circ\text{C}$. Care shall be taken to properly spread out the coated aggregates on the watch-glass to ensure a single layer. Then transfer the mixture in a beaker (6.6), pour roughly 300 ml of water (5.2) that has been pre-heated to $(60 \pm 3)^\circ\text{C}$ into the beaker; cover the beaker with a watch-glass and place it in the ventilated oven at $(60 \pm 3)^\circ\text{C}$ for (20 ± 4) h (6.1).

With low viscosity binders, in particular those incorporating non- or low-volatile flux oils, binder drainage can occur during the dry curing process at 60°C . If that is the case, it shall be mentioned in the test report (11) and only the rating obtained following 8.3.3 shall be retained.

8.3.5 Assess for both parts the surface coated with the film of binder according to the grading scheme given in Clause 9.

9 Expression of results

The surface coated with the film of binder is graded according to the following scheme:

- a) 100: the surface remains fully coated;
- b) 90: more than approximately 90 % of the surface remains coated;
- c) 75: approximately 75 % to 90 % of the surface remains coated;
- d) 50: approximately 50 % to 75 % of the surface remains coated;
- e) < 50: less than 50 % of the surface remains coated;
- f) 0: binder is totally separated from the aggregates: only few globules remain.

For “low” viscosity cut-back and fluxed bituminous binders, the grading to be retained for checking the compliance with the requirements of EN 15322 is the lower rating obtained for the two procedures.

To facilitate the assessment, it may be helpful to compare with an untreated and immersed aggregate.

The simple coloration of the aggregate surface by adsorbed light binder fractions shall not qualify this surface as a coated surface.

10 Precision

The method is qualitative and it is not possible to quantify the precision.

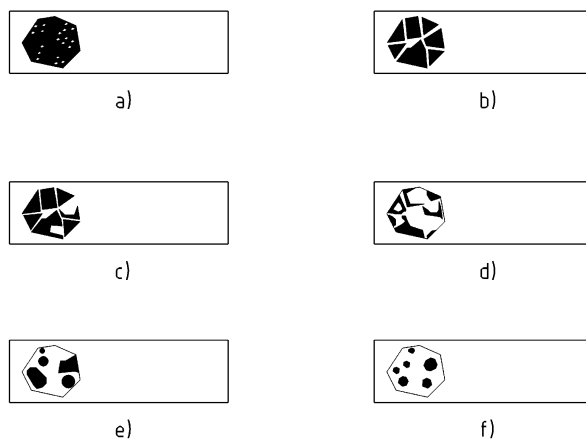
11 Test reports

The test report shall contain at least the following information:

- a) reference to this European Standard;
- b) type and complete identification of the sample under test;
- c) type and identification of the aggregates and fraction used (either 6/10 mm or 8/11 mm);
- d) in the case of a binder containing an adhesion improver, the type of additive (if known), applied binder storage conditions (duration and temperature);
- e) applied binder and aggregate mixing temperatures (Table 1 and 8.1.3);
- f) result of the test (see Clause 9);
- g) any deviation, by agreement or otherwise, from the procedure specified;
- h) date of the test;
- i) name of the operator.

Annex A (informative)

Guidance for the marking of coated surface area



Key

- a) mark 100
- b) mark 90
- c) mark 75
- d) mark 50
- e) mark < 50
- f) mark 0

Figure A.1 — Typical cases of partially or totally coated aggregates

Bibliography

- [1] EN 12597, *Bitumen and bituminous binders — Terminology*

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BSI Group Headquarters

389 Chiswick High Road London W4 4AL UK