

English Version

## Bitumen and bituminous binders - Determination of adhesivity of bituminous emulsions by water immersion test

Bitumes et liants bitumineux - Détermination de l'adhésivité des émulsions de bitume par l'essai d'immersion dans l'eau

Bitumen und bitumenhaltige Bindemittel - Bestimmung des Haftverhaltens von Bitumenemulsionen bei Wasserlagerung

This European Standard was approved by CEN on 12 February 2011.

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## Foreword

This document (EN 13614:2011) has been prepared by Technical Committee CEN/TC 336 “Bituminous binders”, the secretariat of which is held by AFNOR/BNPé.

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of an identical text or by endorsement, at the latest by September 2011, and conflicting national standards shall be withdrawn at the latest by September 2011.

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 13614:2004.

This revised version (EN 13614:2011) makes a distinction between “immediate” adhesivity and water effect on binder adhesion. Compliance to the adhesivity requirements set by EN 13808 is verified according to the procedure defined for testing water effect on binder adhesion.

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, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.



1 Scope

This European Standard specifies a method for determining the adhesion of a bituminous emulsion coated onto aggregate when immersed in water.

The method considers two different aspects of adhesivity, i.e. immediate adhesivity and water effect on binder adhesion.

The method may be used with a reference aggregate. In that case, it measures the intrinsic adhesion behaviour of a bituminous emulsion. The method may also be used with a specific aggregate as used on a job site.

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

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

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

EN 13808, *Bitumen and bituminous binders — Framework for specifying cationic bituminous emulsions*

EN ISO 3696, *Water for analytical laboratory use — Specification and test methods (ISO 3696:1987)*

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 the adhesion

**3.3**  
**immediate adhesivity**  
qualitative assessment of the ability of the binder from a bituminous emulsion to resist the action of water just after coating of the aggregate

**3.4**  
**water effect on binder adhesion**  
qualitative assessment of the ability of the binder from a bituminous emulsion to resist the action of water after coating of the aggregate and a period of curing



## 4 Principle

The bituminous emulsion is mixed thoroughly with the considered aggregate under specified conditions.

When testing immediate adhesivity, the mixture is immediately washed under running water and the percentage of the aggregate surface covered with binder is assessed visually under specified conditions.

When testing water effect on binder adhesion, the mixture is first left to cure and then immersed under water under specified conditions. The percentage of the aggregate surface covered with binder is assessed visually under specified conditions.

Conformity to the “adhesivity” requirement specified in EN 13808 is to be assessed while measuring the water effect on binder adhesion with a reference aggregate.

## 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).

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

**5.2 Water**, distilled or deionised, conforming to EN ISO 3696.

**5.3 Cleaning agents**, conventionally used in a laboratory.

## 6 Apparatus

**6.1 Ventilated oven**, capable of maintaining a temperature of  $(60 \pm 3) ^\circ\text{C}$ .

**6.2 Spatula**.

**6.3 Two heat resistant dishes**, diameter approximately 15 cm to 20 cm.

**6.4 Timer**, accurate to at least 1 s over 60 s.

**6.5 Two beakers**, approximately 500 ml capacity.

**6.6 Set of five watch glasses**, diameter of at least 15 cm.

**6.7 Balance**, of sufficient capacity, accurate to  $\pm 1$  g.

**6.8 Measuring cylinder**, 250 ml to 500 ml capacity.

**6.9 Thermometer**, of adequate range, allowing measuring the specified temperatures with an accuracy of  $\pm 1 ^\circ\text{C}$ .

**6.10 Ventilated oven**, capable of maintaining a temperature of  $(110 \pm 5) ^\circ\text{C}$ .

## 7 Sampling

Sample the test material in accordance with EN 58. Prepare the test samples in accordance with EN 12594.



## **8 Procedure**

### **8.1 General**

Carry out the procedure under normal laboratory conditions ( $23 \pm 5$  °C).

If the intrinsic adhesivity of the emulsion is controlled, the procedure shall be carried out with the reference aggregate(s). The reference aggregate(s) shall be washed with water (5.2) and dried in the ventilated oven (6.10) at ( $110 \pm 5$ ) °C for about 2 h.

If the adhesivity behaviour of a specific emulsion/aggregate combination is controlled for a specific job site, the aggregate shall be used in its job site conditions (5.1).

### **8.2 Determination of immediate adhesivity**

**8.2.1** Weigh ( $100 \pm 5$ ) g of aggregate (5.1) into a dish (6.3) and ( $150 \pm 5$ ) g of emulsion into another dish (6.3).

**8.2.2** Pour the aggregate into the emulsion and allow contacting for ( $60 \pm 5$ ) s without stirring; the dish may be gently shaken to disperse any air bubbles which might prevent proper moistening of the aggregate.

**8.2.3** Remove excess emulsion by carefully tilting the dish and wash the aggregate at room temperature, holding the dish tilted under a slow stream of water (5.2) until the water runs clear.

**8.2.4** Introduce the coated aggregate into a beaker (6.5) and cover with approximately 300 ml of water (5.2) at room temperature.

**8.2.5** Immediately assess the surface coated with the film of binder and grade it according to the scheme indicated in Clause 9.

### **8.3 Water effect on binder adhesion**

#### **8.3.1 General**

This procedure shall be used, with reference aggregate(s), to check conformity to the intrinsic “adhesivity” requirement specified in EN 13808. The reference aggregate(s) is (are) then washed with water (5.2) and dried in the ventilated oven (6.10) at ( $110 \pm 5$ ) °C for about 2 h.

It may also be used to check water effect on binder adhesion for a specific emulsion/aggregate combination used on a specific job site. The aggregate shall then be used in its job site conditions (5.1).

**8.3.2** Weigh ( $200 \pm 5$ ) g of aggregate (5.1) into a dish (6.3) and an amount of emulsion corresponding to ( $10 \pm 1$ ) g of bituminous binder into another dish (6.3).

**8.3.3** Pour the aggregate into the emulsion and thoroughly mix by means of a spatula (6.2).

**8.3.4** If full coating (100 % of coverage) is not achieved, restart the procedure as from 8.3.1 by increasing the amount of residual bitumen to ( $20 \pm 1$ ) g. The amount of residual bitumen actually used shall be mentioned in the test report (Clause 11 f)).

**8.3.5** Spread the mixture on a watch-glass (6.6) and place it in the ventilated oven (6.10) at ( $60 \pm 3$ ) °C for ( $24 \pm 1$ ) h.



**8.3.6** Transfer the mixture in a beaker (6.5), pour approximately 300 ml of water (5.2), heated to  $(60 \pm 3) ^\circ\text{C}$  onto the mixture and cover with a watch-glass (6.6). Place the beaker in the ventilated oven (6.10), at  $(60 \pm 3) ^\circ\text{C}$  for  $(20 \pm 4)$  h.

**8.3.7** Assess the surface coated with the film of binder and grade it according to the scheme indicated in Clause 9.

## 9 Expression of results

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

- 100: all the surface is coated;
- 90: more than approximately 90 % of the surface is coated;
- 75: approximately 75 % to 90 % of the surface is coated;
- 50: approximately 50 % to 75 % of the surface is coated;
- < 50: less than approximately 50 % of the surface is coated;
- 0: the binder is separate from the aggregates, except for some faint marks.

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

NOTE 1 A guidance for the grading of the surface covered with binder is given, in the form of indicative sketches, in Annex A.

NOTE 2 If necessary, to facilitate the assessment, it should be compared with an untreated and immersed aggregate.

## 10 Precision

The method is qualitative and it is not possible to quantify the precision. However, tests carried out by the same operator have shown that the same result is generally achieved for any given bitumen emulsion.

## 11 Test report

The test report shall contain at least the following information:

- a) reference to this European Standard;
- b) type and complete identification of the emulsion sample under test;
- c) type and complete identification (reference aggregate or other aggregate) of the aggregate and fraction used (either 6/10 mm or 8/11 mm);
- d) test used : immediate adhesivity or water effect on binder adhesion;
- e) result of the test (see Clause 9);
- f) in case of a test performed according to 8.2, the corresponding mass of binder used;
- g) any deviation, by agreement or otherwise, from the procedure described;
- h) date of the test.

**Annex A**  
(informative)

**Guidance for the marking of coated surface area**



a)



b)



c)



d)



e)



f)

**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 coated aggregates**



## Bibliography

- [1] EN 12597, *Bitumen and bituminous binders — Terminology*
- [2] EN 13043, *Aggregates for bituminous mixtures and surface treatments of roads, airfields and other trafficked areas*