



BSI Standards Publication

# Bitumen and bituminous binders — Determination of residue on sieving of bituminous emulsions, and determination of storage stability by sieving

**National foreword**

This British Standard is the UK implementation of EN 1429:2013. It supersedes BS EN 1429:2009 which is withdrawn.

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## Bitumen and bituminous binders - Determination of residue on sieving of bituminous emulsions, and determination of storage stability by sieving

Bitumes et liants bitumineux - Détermination du résidu sur tamis des émulsions de bitume et détermination de la stabilité au stockage par tamisage

Bitumen und bitumenhaltige Bindemittel - Bestimmung des Siebrückstandes von Bitumenemulsionen und Bestimmung der Lagerbeständigkeit durch Sieben

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## Foreword

This document (EN 1429:2013) 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of an identical text or by endorsement, at the latest by December 2013, and conflicting national standards shall be withdrawn at the latest by December 2013.

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This document supersedes EN 1429:2009.

The main technical changes brought to EN 1429 are as follows:

- Less stringent precision requirements for the weighing scale to be used in 8.2 and Clause 9.
- More accurate definition of dilution procedures for viscous emulsions (8.2.9 and 8.3.8).
- Revision of procedure for determination of the storage stability by sieving (Clause 9).

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## 1 Scope

This European Standard specifies methods utilising sieving for the determination of the quantity of coarse particles of binder present in bitumen emulsions, and for the determination of storage stability.

## 2 Normative references

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

ISO 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

## 3 Terms and definitions

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

### 3.1 residue on sieving

mass fraction in % of particles retained on a sieve of a mesh size specified in this European Standard

### 3.2 storage stability

ability of a bituminous emulsion not to form more coarse particles within a period specified under an appropriate emulsion specification

Note 1 to entry: Storage stability is a different concept than settling tendency as defined and measured by EN 12847. Although both phenomena are often linked, an emulsion may settle without modification of particle size distribution (no coalescence of emulsion droplets).

Note 2 to entry: The purpose of the limits fixed by the emulsion specification is to ensure that there cannot be any disturbance of the workability of the bitumen emulsion under practical conditions.

## 4 Principle

A known mass of bituminous emulsion is filtered through either a prepared sieve with a mesh size of 0,500 mm or through two prepared sieves with mesh sizes of 0,500 mm and of 0,160 mm. The amount of binder retained on the sieves is weighed after washing and drying.

Storage stability is determined as the amount of binder retained on the sieve with a mesh size of 0,500 mm after a defined storage period ( $n$  days).

## 5 Reagents and materials

Use only reagents of recognised analytical grade and water conforming to grade 3 of EN ISO 3696.

The aqueous solutions (5.1) and (5.2) may be replaced by aqueous phases of the same ionic structure as the emulsion under test.

### 5.1 Solution $S_a$

Aqueous phase solution of nominal 0,01 mol/l of sodium hydroxide (NaOH) containing a nominal 1 % mass fraction sodium oleate. This solution, or the actual aqueous phase, may be used for the preparation of anionic emulsion samples.

### 5.2 Solution $S_c$

Aqueous phase solution of nominal 0,01 mol/l of hydrochloric acid (HCl) containing a nominal 1 % mass fraction cetyltrimethylammonium bromide. This solution, or the actual aqueous phase, may be used for the preparation of cationic emulsion samples.

The solution of 1 % of cetyltrimethylammonium bromide should be prepared slightly above 25 °C (but not higher than 30 °C). Then it should be stored at a temperature of  $(25 \pm 1)$  °C before test.

### 5.3 Efficient rinsing agent

Suitable solvent rinsing agents shall be used.

### 5.4 Ethanol

96 % minimum or methylated spirit 99 %.

## 6 Apparatus

Usual laboratory apparatus and glassware, together with the following:

**6.1 Sieve**, conforming to ISO 565, stainless steel or brass, with a frame diameter within the range of 75 mm to 100 mm and a mesh size of 0,500 mm.

**6.2 Sieve**, conforming to ISO 565, stainless steel or brass, with a frame diameter within the range of 75 mm to 100 mm and a mesh size of 0,160 mm.

**6.3 Sieve pans**, of corresponding diameter.

**6.4 Balance**, of sufficient capacity, capable of weighing to the nearest 1 g.

**6.5 Balance**, of sufficient capacity, capable of weighing to the nearest 0,01 g.

**6.6 Balance**, of sufficient capacity, capable of weighing to the nearest 0,001 g.

**6.7 Conical flask**, 200 ml capacity, with a ground stopper.

**6.8 Conical flask**, two, 250 ml capacity with a ground stopper.

**6.9 Conical flask**, 500 ml capacity with a ground stopper (see 9.1).

**6.10 Bottle**, of sufficient capacity, with a screw-in stopper.

**6.11 Container**, of sufficient capacity.

**6.12 Ventilated oven**, capable of maintaining a temperature of  $(105 \pm 5)$  °C around the sample.

**6.13 Desiccator.**

**6.14 Funnel.**

## 7 Sampling

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

The sample shall be divided into two portions; for referee purposes, both portions shall be tested (see Clause 11).

## 8 Procedure for the determination of residue on sieving

### 8.1 General

Carry out the procedure under normal laboratory conditions between 18 °C and 28 °C.

### 8.2 Residue on the 0,500 mm sieve

**8.2.1** Wash the 0,500 mm sieve (6.1) with rinsing agent (5.3) and then with ethanol or methylated spirit (5.4).

**8.2.2** Place the sieve on the sieve pan (6.3) and dry in the oven (6.12) for not less than 1 h.

**8.2.3** After drying, allow the sieve and the sieve pan to cool within the desiccator (6.13).

**8.2.4** Weigh the sieve and the sieve pan together. Record the mass,  $m_I$ , to the nearest 0,01 g (6.5).

**8.2.5** Wet the sieve cloth with solution  $S_a$  (5.1) or  $S_c$  (5.2) as appropriate.

**8.2.6** Drain the sieve thoroughly and place it on a funnel (6.14) which is mounted over the bottle (6.10).

**8.2.7** Weigh the container (6.11) and record the mass,  $m_C$ , to the nearest 1 g (6.4).

**8.2.8** Weigh  $(1\ 000 \pm 5)$  g of emulsion into the container and record the mass of emulsion,  $m_{E, 0,500}$ , to the nearest 1 g (6.4).

**8.2.9** Pour the emulsion through the wetted sieve and allow draining completely.

Discard the first 30 ml or 40 ml of the filtered emulsion, in order to avoid any possible modification in the properties of the emulsion resulting from the action of the  $S_a$  or  $S_c$  solution.

Clear as much as possible the container of the emulsion and reweigh the container. Record the mass,  $m_{C, R}$  to the nearest 1 g (6.4).

If the emulsion does not pass through the sieve in 5 min at normal laboratory conditions, it is permitted to deviate from this procedure by pre-heating and testing the laboratory sample at  $(60 \pm 5)$  °C and/or by diluting the emulsion with either  $S_a$  solution (5.1) or  $S_c$  solution (5.2) as appropriate, in order to determine residue on sieving. If a dilution is performed, an amount of  $(500 \pm 5)$  g, weighed to the nearest 1 g (6.4), of  $S_a$  or  $S_c$  solution shall be used.

If heating is required, all precautions should be taken to minimize water loss and/or skin formation during this operation.



In case dilution is performed, adequate adaptations of the procedure and of calculations shall be performed and documented in the test report. Except for the determination of the residue on the 0,160 mm sieve (8.2), the filtered emulsion shall not be used for any other emulsion test.

**8.2.10** Remove the bottle containing the filtered emulsion, and stopper it.

NOTE The emulsion filtered in this way will be used to carry out the second part of the test (see 8.3, when required) and, if not diluted, to carry out all other tests on the emulsion.

**8.2.11** Place the sieve on a funnel which is mounted over a 250 ml conical flask (6.8). Wash the residue on the sieve with solution  $S_a$  or  $S_c$  until the washings run clear. Finally wash with water.

**8.2.12** Drain the sieve thoroughly and place it on the sieve pan. Dry in the oven (6.12) for at least 2 h and then cool in the desiccator (6.13) for about 30 min.

**8.2.13** Weigh the sieve with its sieve pan and residue and record the mass,  $m_2$ , to the nearest 0,01 g (6.5).

### **8.3 Particles between 0,500 mm and 0,160 mm**

**8.3.1** Wash the 0,160 mm sieve (6.2) with rinsing agent (5.3) and then with ethanol or methylated spirit (5.4).

**8.3.2** Place the sieve on the sieve pan (6.3) and dry in the oven (6.12) for not less than 1 h.

**8.3.3** After drying, allow the sieve and the sieve pan to cool in the desiccator (6.13).

**8.3.4** Weigh the sieve and the sieve pan together. Record the mass,  $m_3$ , to the nearest 0,001 g (6.6).

**8.3.5** Wet the sieve cloth with solution  $S_a$  (5.1) or  $S_c$  (5.2) as appropriate.

**8.3.6** Drain it thoroughly and place it on a funnel which is mounted over a 250 ml conical flask (6.8).

**8.3.7** Gently stir the emulsion that was filtered through the 0,500 mm sieve (see 8.2.10).

**8.3.8** Place the 200 ml conical flask (6.7) on the balance and weigh into it 50 g,  $m_{E, 0,160}$  of filtered emulsion to the nearest 1 g (6.4), then add approximately 50 cm<sup>3</sup> of the  $S_a$  or  $S_c$  solution.

In case the emulsion has been diluted for determining the residue on the 0,500 mm sieve (8.2.9), weigh into the conical flask (6.7) 75 g,  $m_{E, 0,160}$  of filtered emulsion to the nearest 1 g (6.4) and add approximately 25 cm<sup>3</sup> of the  $S_a$  or  $S_c$  solution.

**8.3.9** Gently stir the diluted emulsion.

**8.3.10** Filter the diluted emulsion through the 0,160 mm sieve and proceed as described for filtering with 0,500 mm sieve. Wash the residue on the sieve with solution  $S_a$  or  $S_c$  solution until the washings run clear. Finally wash with water.

**8.3.11** Drain the sieve thoroughly and place it on the sieve pan. Dry in the oven (6.12) for at least 2 h and then cool in the desiccator (6.13) for approximately 30 min.

**8.3.12** Weigh the sieve with its sieve pan and residue and record the mass,  $m_4$ , to the nearest 0,001 g (6.6).

## 9 Procedure for the determination of the storage stability by sieving after $n$ days storage period

9.1 Carefully rinse a conical flask (6.8) with solution  $S_a$  (5.1) or  $S_c$  (5.2) as appropriate. For very viscous emulsions which are slow to pass through the aperture of the 0,500 mm sieve, use a 500 ml conical flask (6.9).

9.2 Weigh enough of the bituminous emulsion prepared according to Clause 7 (approximately 250 g) into the conical flask. Record the mass  $m_{E,n \text{ days}}$  to the nearest 1 g (6.4).

9.3 Store the closed flask in a dark room at ambient temperature for  $n$  days.

Following storage, measure the residue on sieving as described below.

9.4 Wash the 0,500 mm sieve (6.1) with rinsing agent (5.3) and then with ethanol or methylated spirit (5.4).

9.5 Place the sieve on the sieve pan (6.3) and dry in the oven (6.12) for not less than 1 h.

9.6 After drying, allow the sieve and the sieve pan to cool in the desiccator (6.13).

9.7 Weigh the sieve and the sieve pan together. Record the mass,  $m_5$ , to the nearest 0,01 g (6.5).

9.8 Wet the sieve cloth with solution  $S_a$  (5.1) or  $S_c$  (5.2) as appropriate.

9.9 Drain it thoroughly and place it on a funnel which is mounted over the bottle (6.10).

9.10 Pour the emulsion through the wetted sieve. For very viscous emulsions which have been stored in a 500 ml flask, first dilute the emulsion by adding approximately 200 ml of the  $S_a$  or  $S_c$  solution.

9.11 Rinse the conical flask with solution  $S_a$  or  $S_c$  over the sieve until the washings run clear. Drain the flask thoroughly. If a significant amount of emulsion stays adhering to the walls of the flask, report "Broken emulsion" in the test report under f).

9.12 Wash the residue on the sieve with solution  $S_a$  or  $S_c$  until the washings run clear. Finally wash with water. Drain the sieve thoroughly.

9.13 Place the sieve on the sieve pan. Dry the sieve and the sieve pan in the oven (6.12) for at least 2 h and then cool in the desiccator (6.13) for approximately 30 min.

9.14 Weigh the sieve with its sieve pan and residue and record the mass,  $m_6$ , to the nearest 0,01 g (6.5).

## 10 Calculation

### 10.1 For residue on the 0,500 mm sieve

For the residue on the 0,500 mm sieve ( $R_{0,500}$ ), calculate its mass fraction in %, using the following formula:

$$R_{0,500} = \frac{(m_2 - m_1)}{m_{E, 0,500} - (m_{C,R} - m_C)} \times 100 \quad (1)$$

where

$m_1$  is in grams as defined in 8.2.4;

$m_2$  is in grams as defined in 8.2.13;

$m_{E, 0,500}$  is in grams as defined in 8.2.8;

$m_{C, R}$  is in grams as defined in 8.2.9;

$m_C$  is in grams as defined in 8.2.7.

## 10.2 For particles between 0,500 mm and 0,160 mm

For the residue on the 0,160 mm sieve ( $R_{0,160}$ ), calculate its mass fraction in %, using the following formula:

$$R_{0,160} = \frac{(m_4 - m_3)}{m_{E, 0,160}} \times 100 \quad (2)$$

where

$m_3$  is in grams as defined in 8.3.4;

$m_4$  is in grams as defined in 8.3.12;

$m_{E, 0,160}$  is in grams as defined in 8.3.8.

## 10.3 For residue on the 0,500 mm sieve after $n$ days storage

For the residue after  $n$  days storage ( $R_n$  days; 0,500), calculate its mass fraction in %, using the following formula:

$$R_{n \text{ days}; 0,500} = \frac{(m_6 - m_5)}{m_{E, n \text{ days}}} \times 100 \quad (3)$$

where

$m_5$  is in grams as defined in 9.7;

$m_6$  is in grams as defined in 9.14;

$m_{E, n \text{ days}}$  is in grams as defined in 9.2.

## 11 Expression of results

Express the residue on sieving as a mass fraction in % to the nearest 0,01 % or, for referee purposes, as the arithmetic mean of two tests.

Duplicate tests are required for referee purposes only. In the case of routine internal production control, it is permitted to perform a single test.

## 12 Precision

### 12.1 Repeatability

The difference between two test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed a mass fraction of 0,03 % in only one case in twenty.

### 12.2 Reproducibility

The difference between two single and independent test results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed a mass fraction of 0,08 % in only one case in twenty.

## 13 Test report

The test report shall contain at least the following information:

- a) type and complete identification of the sample under test;
- b) reference to this European Standard;
- c) conditioning of the sample at  $(60 \pm 5)$  °C, if performed;
- d) dilution of the sample, if performed;
- e) result of the test (Clause 11);
- f) "broken emulsion" if a significant amount of emulsion stays adhering to the walls of the flask after the procedure described in 9.11;
- g) number of tests performed;
- h) any deviation, by agreement or otherwise, from the procedure specified;
- i) date of the test.

## Bibliography

EN 12847, *Bitumen and bituminous binders — Determination of settling tendency of bituminous emulsions*





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