



BSI Standards Publication

Bitumen and bituminous binders — Determination of water content in bituminous emulsions — Method using a drying balance

National foreword

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

Bitumes et liants bitumineux - Détermination de la teneur en eau des émulsions de bitume - Méthode par évaporation à la balance dessiccatrice

Bitumen und bitumenhaltige Bindemittel - Bestimmung des Wassergehaltes von Bitumenemulsionen - Verfahren mittels Trocknungswaage

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European foreword

This document (EN 16849:2016) 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of an identical text or by endorsement, at the latest by April 2017, and conflicting national standards shall be withdrawn at the latest by April 2017.

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1 Scope

This European Standard specifies a quick method for determining, by evaporation, the water content of bituminous road emulsions, with or without polymer added.

For bituminous emulsions without flux oil, bituminous emulsions containing vegetal flux oil, and bituminous emulsions containing up to 1,5 % mass of mineral flux oil in the emulsion, this European Standard, according to the selected operating conditions, is considered as an alternative method to the reference methods EN 1428 [1] and EN 1431 [2].

Above a mineral flux oil content of 1,5 % by mass, depending on the volatility of the flux oil:

- the present method can be used if the user can prove its reliability in comparison to EN 1428 or EN 1431.
- the present method can only be used by correcting the result by means of a previously established correlation with the reference method EN 1428 or EN 1431.

NOTE Polymer modified emulsions can behave differently in the test than unmodified emulsions; in case of doubt, the method is checked against EN 1428 or EN 1431.

In case of dispute, the water content should be determined according to EN 1428 or EN 1431.

WARNING — The use of this European Standard can 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 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*

3 Terms and definitions

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

3.1 water content

percentage in mass of water determined according to the test conditions described in this European Standard

3.2 mineral flux

flux which may be of carbochemical, petrochemical or petroleum origin or a mixture of those

3.3 vegetal flux

type of bio-flux derived exclusively from plant based (vegetal) product

4 Principle

The water contained in the emulsion is evaporated by regulated heating. The sample being directly placed on a balance integrated to the apparatus, the weight loss is automatically determined until a constant mass is obtained.

5 Reagents and material

Solvents appropriate for the cleaning of the laboratory material used.

6 Apparatus

Usual laboratory material and glassware, together with the following:

6.1 Drying balance, of sufficient capacity, capable of weighing to the nearest 0,001 g, equipped with a regulated heating device able to maintain a temperature at an accuracy ± 5 °C in the range of 110 °C to 150 °C, around the sample during the test.

6.2 Drying cups, in a material not affecting the behaviour of bituminous emulsions.

6.3 Absorbing glass fibre filter(s), to enable a homogeneous repartition of the sample and to avoid skinning effect.

7 Sampling

The product submitted to the test shall be sampled according to EN 58 and shall be prepared according to EN 12594.

8 Procedure

8.1 Control of the apparatus

Each new apparatus shall be calibrated for temperature and weighing accuracy before its first use. Once in service, an apparatus shall be calibrated at least yearly.

8.2 Test temperature and stop criterion

Test temperature shall be set at:

- 110 °C for mineral flux emulsions with up to 1,5 % by mass of flux and emulsions with 5 % by mass or more of vegetal flux;
- 130 °C for vegetal fluxed emulsions with less than 5 % by mass of flux;
- 150 °C for non-fluxed emulsions.

The stop of the test can be defined automatically or manually. The stop criterion shall be set at a value closest to about 1 mg/50 s.

8.3 Test

8.3.1 Switch on the drying balance (6.1). Set up the heating temperature and stop criterion at the desired values (see 8.2).

8.3.2 Place the absorbing filter (6.3) on the drying cup (6.2) and tare the balance.

8.3.3 Remove the absorbing filter and weigh $(4,0 \pm 0,5)$ g of emulsion, m_0 in the drying cup to the nearest 0,001 g. Distribute the emulsion uniformly on the cup surface in less than 30 s. Place the same absorbing filter (8.3.2) on top of the emulsion sample in order to ensure an even drying of the test sample.

NOTE To enable easy cleaning and re-use of the drying cup, it is also possible to pour the emulsion sample in-between two glass fibre filters.

8.3.4 Start up the apparatus for test.

8.3.5 The test is considered finished when the mass of the dehydrated binder is constant. The mass is considered constant when the stop criterion is satisfied (8.2).

Record the final mass m_f to the nearest 0,001 g.

Before any new measurement, open the balance and let it cool down to ambient temperature

9 Calculation

The water content (w) of the sample tested, expressed in percentage by mass, is calculated using the following formula:

$$w = \frac{m_0 - m_f}{m_0} \times 100$$

where

m_0 is the initial mass of emulsion (8.3.3);

m_f is the final mass (8.3.5).

NOTE some apparatus automatically calculates the water content and/or binder content when the test is finished.

10 Expression of results

Express the water content as a percentage by mass to the nearest 0,1 %.

11 Precision

NOTE The precision of the method was evaluated in the frame of a Round Robin test program conducted by TC336/WG2 [3] in accordance with ISO 5725-2 [4].

11.1 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 0,6 % by mass in absolute value, in only one case in twenty [3].

11.2 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 1,8 % by mass in absolute value, in only one case in twenty [3].

12 Test report

The test report shall contain at least the following information:

- a) a reference to this European Standard;
- b) the type and complete identification of the sample under test;
- c) test temperature and stop criterion
- d) the test result (see Clause 10); if used, the corrected water content according to the correlation with the reference method EN 1428 [1] or EN 1431 [2];
- e) any deviation, by agreement or otherwise, from the procedure specified;
- f) the date of the test.

Bibliography

- [1] EN 1428, *Bitumen and bituminous binders — Determination of water content in bituminous emulsions — Azeotropic distillation method*
- [2] EN 1431, *Bitumen and bituminous binders — Determination of residual binder and oil distillate from bitumen emulsions by distillation*
- [3] CEN TC336/WG2, Round Robin on bituminous emulsions, B. Eckmann, F. Le Cunff, M. Cresnar, S. Collins in the name of TC336/WG2 6th Eurasphalt & Eurobitume Congress, Paper 100, Prague 2016
- [4] ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

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BSI Group Headquarters

389 Chiswick High Road London W4 4AL UK