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Bitumen and bituminous binders — Determination of water content in bituminous emulsions — Azeotropic distillation method

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Bitumen and bituminous binders - Determination of water content in bituminous emulsions - Azeotropic distillation method

Bitumes et liants bitumineux - Détermination de la teneur en eau dans les émulsions de bitume - Méthode de distillation azéotropique

Bitumen und bitumenhaltige Bindemittel - Bestimmung des Wassergehaltes von Bitumenemulsionen - Azeotropisches Destillationsverfahren

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Foreword

This document (EN 1428:2012) 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of an identical text or by endorsement, at the latest by July 2012, and conflicting national standards shall be withdrawn at the latest by July 2012.

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1 Scope

This European Standard specifies a method for the determination of the water content in bituminous emulsions by means of distillation.

WARNING — The use of this standard 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 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 ISO 3696, *Water for analytical laboratory use — Specification and test methods (ISO 3696)*

ISO 5272, *Toluene for industrial use — Specifications*

ISO 5280, *Xylene for industrial use — Specification*

3 Terms and definitions

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

3.1
water content
mass percentage of water determined in accordance with the method specified in this standard

4 Principle

The water contained in a bituminous emulsion is distilled over by means of a carrier vapour from a water immiscible solvent-carrier liquid.

Condensed solvent-carrier liquid and water are separated continuously in a graduated receiver; the water settles in the graduated section of the receiver and the condensed solvent-carrier liquid returns to the flask.

5 Reagents and materials

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

5.1 Distillation regulators, with a higher density than the liquid under test such as, for instance, anhydrous anti-bumping granules (e.g. boiling sand), glass beads.

5.2 Suitable solvent-carrier liquid.

Xylene conforming to ISO 5280 is recommended for routine tests. Other petroleum distillates, conforming to the following distillation requirements: 98% distils between 120°C and 250ºC, are suitable.

In the event of dispute, toluene conforming to ISO 5272 shall be used.

NOTE In selecting a solvent-carrier liquid, attention is drawn to any relevant safety regulations.

6 Apparatus

Usual laboratory apparatus and glassware, together with the following:

6.1 Distillation apparatus (see Figure 1) consisting of the following: flask, receiver and condenser being connected with suitable ground glass joints.

6.1.1 Flask, 500 ml round bottomed with a short neck capable of supporting the reflux tube on the receiver.

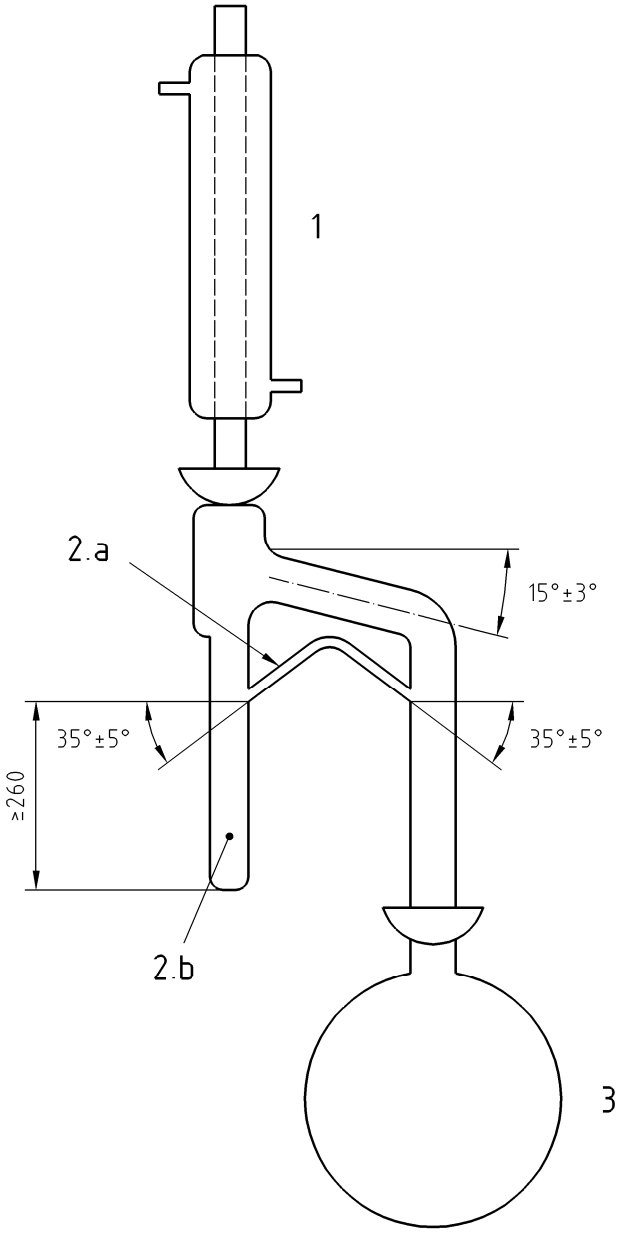
6.1.2 Receiver, with a 25 ml nominal capacity graduated to 0,1 ml.

6.1.3 Vertical condenser, water-cooled with a minimum length of jacket of 300 mm.

6.1.4 Heater.

NOTE An electric flask heater with electronic power regulation is recommended in order to obtain controlled distillation.

Dimension in millimetres



1. Reflux condenser
2. Receiver
   a With or without this tube
   b Capacity: 25 ml
3. Round bottomed flask

Figure 1 – Typical assembly

6.2 Stainless steel wire, with looped-end or fitted with a rubber stopper.

NOTE Other materials or devices are acceptable provided they allow dislodging the water (8.2.7) without interfering with the measurement.

6.3 Balance, of sufficient capacity, accurate to 0,01 g.

7 Sampling

The test material shall be sampled in accordance with EN 58. Preparation of test samples shall be made in accordance with EN 12594.

The prepared sample shall be divided into two test portions.

For referee purposes, both portions shall be tested (see Clause 10, 2nd paragraph).

8 Procedure

8.1 Verification of apparatus

When necessary the apparatus shall be verified in accordance with the procedure given in Annex A.

The verification procedure shall be carried out on new apparatus before using it for the first time and existing apparatus shall be checked annually.

8.2 Test

8.2.1 Carry out the procedure under normal laboratory conditions, at a temperature of (23 ± 5) °C.

8.2.2 Before commencing a test, ensure that the distillation apparatus is clean and dry.

8.2.3 Pour 100 ml to 200 ml of solvent-carrier liquid (5.2) added with distillation regulator (5.1) into the round bottomed flask (6.1.1).

8.2.4 Weigh a sample of the emulsion under test, of such a size that the distillation of 15 ml to 25 ml of water can be expected, into the round bottomed flask (6.1.1).

NOTE Adequate measures should be taken so as to prevent splashes of emulsion onto the neck of the flask, (e.g. rod, funnel, ...).

8.2.5 Assemble the apparatus and insert a loose plug of cotton wool in the top of the condenser tube (6.1.3) to prevent the condensation of atmospheric moisture in the condenser tube.

8.2.6 Heat the flask to boiling point, adjusting the rate of boiling so that condensate falls from the end of the condenser at a rate of two to five drops per second.

8.2.7 If there is water in the condenser tube or adhering to the sides of the receiver, dislodge it with the stainless steel wire (6.2). Continue distillation until the volume of water in the receiver is constant and no water is visible other than in the receiver.

8.2.8 Rinse the condenser with solvent-carrier liquid (5.2) from a wash bottle.

8.2.9 Allow a clear interface to develop between the water and solvent layers in the receiver then read off the volume of water to the nearest 0,1 ml.

9 Calculation

Calculate the water content of the material under test (w), as a mass percentage, using the following equation:

$$w = \frac{m_W}{m_E} \times 100$$

where

m_W is the mass of the water distilled from the test material, in grams, and is equal to the volume of water, in millilitres collected in the graduated receiver;

m_E is the mass of emulsion used for the test, in grams.

10 Expression of results

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

11 Precision

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 term, in the normal and correct operation of the test method, exceed 1 % mass fraction in absolute value, in only one case in twenty.

11.2 Reproducibility

The difference between two single and independent 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 2 % mass fraction in absolute value, in only one case in twenty.

In the case of an emulsion containing a flux, the reproducibility is 3 % mass fraction in absolute value.

12 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) type of solvent used;

d) result of the test (see Clause 10);

e) number of tests performed;

f) any deviation, by agreement or otherwise, from the procedure specified;

g) date of the test.

Annex A
(normative)

Verification of apparatus

A.1 A given apparatus assembly shall be considered satisfactory if a result within permissible limits is obtained after a known quantity of water (5) is added to the solvent-carrier liquid (5.2) and the mixture is tested in accordance with 8.2.

A.2 The results shall be judged acceptable if the permissible limits given in Clause A.3 for the 25 ml graduated receiver are not exceeded.

Results outside the permissible limits shall be investigated and the causes eliminated before proceeding.

Repeat the verification until a result within the permissible limits is obtained.

A.3 The quantity (q_W) of water added to the flask at ambient temperature shall be (15,00 ± 0,02) g to (25,00 ± 0,02) g. The permissible limits of water recovered at ambient temperature are q_W ± 1,66 %.

NOTE For this procedure, 1 ml of water may be taken as having a mass of 1 g.

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