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Bitumen and bituminous binders — Determination of breaking behaviour

Part 2: Determination of fines mixing time of cationic bituminous emulsions

National foreword

This British Standard is the UK implementation of EN 13075-2:2016. It supersedes BS EN 13075-2:2009/BS 2000-495:2009 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PTI/15, Natural Gas & Gas Analysis.

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

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English Version

Bitumen and bituminous binders - Determination of breaking behaviour - Part 2: Determination of fines mixing time of cationic bituminous emulsions

Bitumes et liants bitumineux - Détermination du
comportement à la rupture - Partie 2 : Détermination
de la durée de miscibilité des fines dans les émulsions
cationiques de bitume

Bitumen und bitumenhaltige Bindemittel -
Bestimmung des Brechverhaltens - Teil 2: Bestimmung
der Mischzeit von Feinanteilen in kationischen
Bitumenemulsionen

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European foreword

This document (EN 13075-2:2016) 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of an identical text or by endorsement, at the latest by May 2017, and conflicting national standards shall be withdrawn at the latest by May 2017.

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

This document supersedes EN 13075-2:2009.

In comparison with EN 13075-2:2009, the following significant changes have been made:

- Clause 5: three reference fillers (Forshammer, Sikaisol and Caolin Q92) may be used;
- Subclause 6.10 is discarded since this clause is not referenced in the test procedure (Clause 8) and since requirements on temperature control are already stated in 6.1 (oven) and 6.7 (temperature bath or climatic chamber);
- Subclause 6.2 and Clause 8: the conical shaped funnel is no longer required as filler is to be directly added via a spoon from a simple ad hoc container. Clause 6.2 now defines the different containers needed by the test procedure. Clause 8 is modified accordingly;
- Clause 10: since no supporting evidence for the values previously stated in the 2009 version could be found, repeatability and reproducibility figures are withdrawn;
- Annex A: more complete (e.g. particle size distribution envelopes) and more homogeneous description of the three reference fillers.

EN 13075 consists of the following parts under the general title "*Bitumen and bituminous binders - Determination of breaking behaviour*":

- *Part 1: Determination of breaking value of cationic bituminous emulsions, mineral filler method*
- *Part 2: Determination of fines mixing time of cationic bituminous emulsions*

According to the CEN-CENELEC Internal Regulations, the national standards organisations 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 fines mixing time of diluted cationic bituminous emulsions, under standardized conditions.

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*

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 term and definition applies.

3.1 fines mixing time

time in seconds for the mixability of a mixture of mineral filler and bitumen emulsion without noticeable breaking effect under the conditions specified in this European Standard

4 Principle

A specified quantity of filler is added at a uniform rate, with stirring, to a specified quantity of bitumen emulsion diluted with water. Stirring is continued until the mixture becomes pasty and forms lumps that do not adhere to the walls of the pan. This, combined with a noticeable increase in stirring power indicates the end of mixability. The time to reach this point (breaking status) is the fines mixing time.

5 Reagents and materials

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

5.1 Reference fillers.

The Forshammer filler¹⁾, the Sikaisol filler¹⁾ or the Caolin Q92 filler¹⁾ shall be used as reference fillers. The characteristics of these fillers are given in Annex A.

In the event of dispute, the same filler shall be used by the testing laboratories.

5.2 Cleaning agents, as used conventionally in laboratories.

1) This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN of the product named. Equivalent products may be used if they can be shown to lead to the same results, or if a correlation between the products has been established.

6 Apparatus

Usual laboratory apparatus and glassware, together with the following.

- 6.1 **Oven**, capable of maintaining a temperature of (110 ± 5) °C.
- 6.2 **Containers** of appropriate capacity for storing and holding the quantities of filler required in Clause 8.
- 6.3 **Spatula**, nickel or stainless steel, 20 cm long.
- 6.4 **Spatula**, spoon shaped for adding filler.
- 6.5 **Stop watch**, with an accuracy of 0,2 s or better over a time interval of 500 s.
- 6.6 **Bottles**, of approximate capacity 500 ml made of a material that will not react with the emulsion, having tight fitting lids, for storing emulsion samples and water.
- 6.7 **Constant temperature bath and/or climatic chamber**, capable of maintaining a temperature of (25 ± 1) °C.

If the bath is used to condition the emulsion sample bottles, it should be equipped with a frame or device to prevent the bottles from moving in the water bath.

- 6.8 **Balance**, capable of weighing up to 1 000 g with an accuracy of 0,1 g or better.
- 6.9 **Enamelled or stainless steel dish**, of suitable volume, capable of containing 150 g of diluted emulsion and 150 g of filler.

7 Sampling

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

8 Procedure

Carry out the procedure under normal laboratory conditions (18 °C to 28 °C).

Dry the quantities of filler, required for the test, in the oven (6.1) at a temperature of (110 ± 5) °C until constant mass is reached and let it cool to ambient temperature in a desiccator.

Pour a portion of emulsion (250 ± 10) g and water (150 ± 10) g into separate bottles (6.6) and secure the lid. Place the required quantity of filler in a closed container (6.2). Place the bottles with the emulsion and the water and the container with the filler in the constant temperature bath or climatic chamber (6.7) for a minimum of 1,5 h.

Transfer $(100,0 \pm 0,5)$ g of the emulsion in the dish (6.9) then add $(50,0 \pm 0,5)$ g of water while agitating with the spatula (6.3).

Transfer (150 ± 1) g of filler in a second container (6.2).

Start the stop watch (6.5), and while mixing with the spatula (6.3) at constant rate of 1 r/s, add filler with the spoon shaped spatula (6.4) from the container (6.2) to the diluted emulsion, by portions of 10 g per 5 s, so that within the 75 s, the overall (150 ± 1) g filler is poured.

NOTE To control the portion of filler, the container could be placed on the balance.

Continue mixing and measure the time until the emulsion breaks (see Clause 4).

If the bitumen emulsion does not break within 300 s, the test procedure shall be stopped, and the result of the test “> 300 s” shall be reported.

It may happen that lumps form as soon as the filler contacts the diluted emulsion, but the body of the emulsion neither breaks nor reaches the “pasty” stage within 300 s. In such cases, the emulsion is deemed to have not broken. The test procedure shall be stopped and the result of the test “> 300 s – formation of lumps” shall be reported.

Repeat the test with a new test portion taken from the same laboratory sample using clean apparatus.

9 Expression of results

Express the individual mixing time values (in seconds) to the nearest integer.

Express the result as the arithmetic mean of the two individual results of mixing time, to the nearest integer.

10 Precision

A European round robin test has not been carried out.

11 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) reference to the used filler;
- d) result of the test and individual mixing time values (see Clause 8 and Clause 9);
- e) any deviation, by agreement or otherwise, from the procedure described;
- f) date of the test.

Annex A (normative)

Characteristics of the reference fillers

A.1 Characteristics of the Forshammer filler

The Forshammer filler is a mixture of:

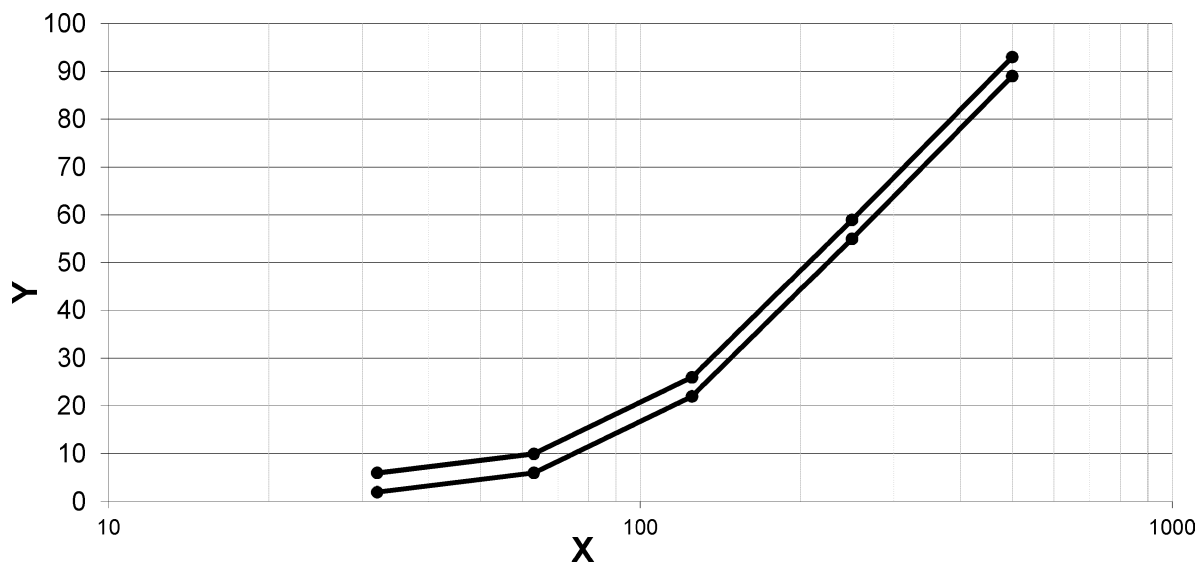
- 65 % Feldspath
- 30 % Quartz
- 5 % Mica

It has following compositional properties (X-ray fluorescence):

- SiO₂: from 74,3 % to 76,8 %
- Al₂O₃: from 14,1 % to 14,8 %
- K₂O: from 3,6 % to 4,7 %
- Na₂O: from 4,2 % to 5,2 %

The particle size distribution envelope, according to EN 933-10 [1], is shown in Figure A.1 and defined by:

- 89 % to 93 % passing the 0,500 mm sieve;
- 55 % to 59 % passing the 0,250 mm sieve;
- 22 % to 26 % passing the 0,125 mm sieve;
- 6 % to 10 % passing the 0,063 mm sieve;
- 2 % to 6 % passing the 0,032 mm sieve.



Key

- X sieves, in µm
- Y % passing, in weight

Figure A.1 — Gradation of the Forshammer filler

A.2 Characteristics of the Sikaisol filler

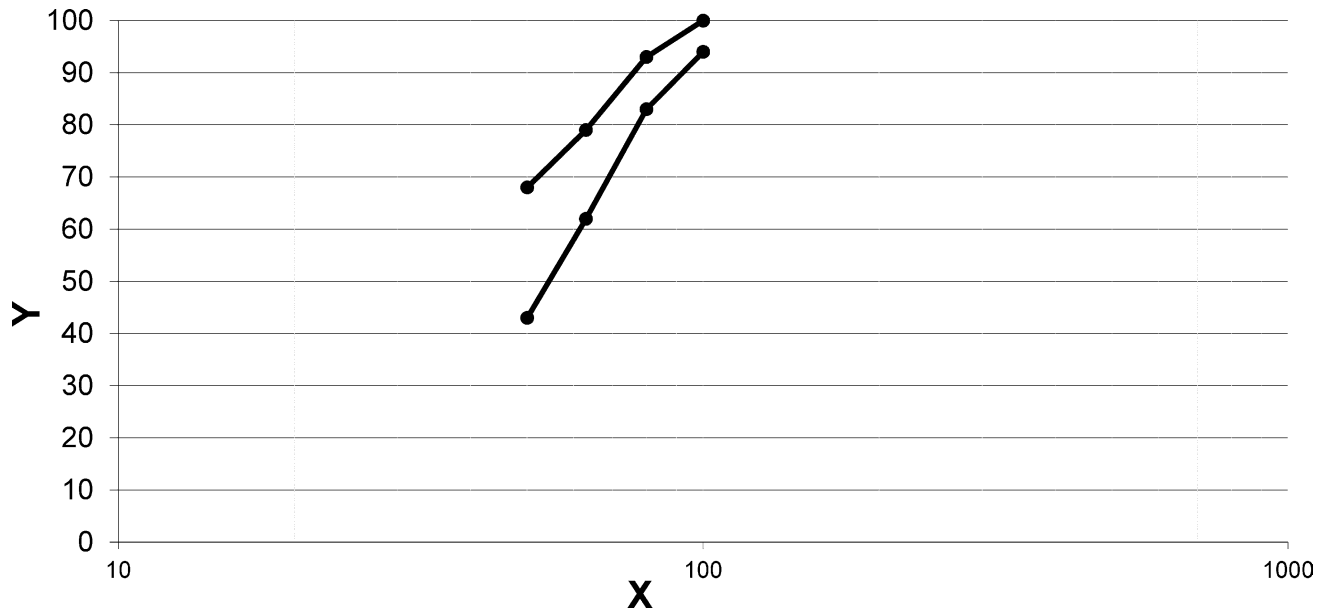
The Sikaisol filler has the following properties:

- natural fine silica, uncrushed;
- SiO₂ content greater than 98 %;
- density of (2650 ± 20) kg/m³;

The particle size distribution envelope is shown in Figure A.2 and defined by:

- 94 % to 100 % passing the 0,100 mm sieve;
- 83 % to 93 % passing the 0,080 mm sieve;
- 62 % to 79 % passing the 0,063 mm sieve;
- 43 % to 68 % passing the 0,050 mm sieve.

The Sikaisol filler is no longer produced, however since it may still be available at production sites and laboratories, this filler has been maintained as a possible reference filler in this standard.



Key

- X sieves, in μm
- Y % passing, in weight

Figure A.2 — Gradation limits of the Sikaisol filler

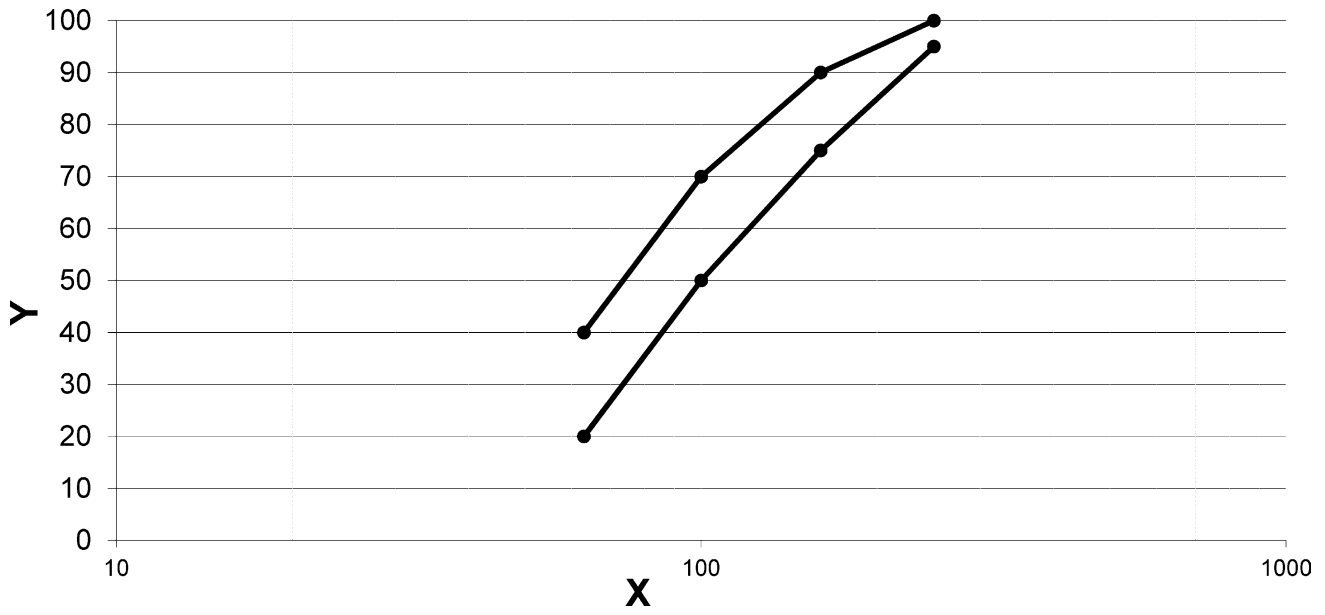
A.3 Characteristics of the Caolin Q92 filler

The Caolin Q92 filler has the following properties (X-Ray fluorescence):

- SiO_2 : from 84,0 % to 95,0 %
- Al_2O_3 : from 3,5 % to 7,0 %
- K_2O : from 2,0 % to 5,0 %

The particle size distribution envelope, according to EN 933-1 [2], is shown in Figure A.3 and defined by:

- 95 % to 100 % passing the 0,250 mm sieve;
- 75 % to 90 % passing the 0,160 mm sieve;
- 50 % to 70 % passing the 0,100 mm sieve;
- 20 % to 40 % passing the 0,063 mm sieve.



Key
X sieves, in μm
Y % passing, in weight

Figure A.3 — Gradation of the Caolin Q92 filler

Bibliography

- [1] EN 933-10, *Tests for geometrical properties of aggregates - Part 10: Assessment of fines - Grading of filler aggregates (air jet sieving)*
- [2] EN 933-1, *Tests for geometrical properties of aggregates - Part 1: Determination of particle size distribution - Sieving method*

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