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Bitumen and bituminous binders — Determination of breaking behaviour

Part 1: Determination of breaking value of cationic bituminous emulsions, mineral filler method

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National foreword

This British Standard is the UK implementation of EN 13075-1:2016. It supersedes BS EN 13075-1:2009/BS 2000-494:2009 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PTI/13, Petroleum Testing and Terminology.

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

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Bitumen and bituminous binders - Determination of breaking behaviour - Part 1: Determination of breaking value of cationic bituminous emulsions, mineral filler method

Bitumes et liants bitumineux - Détermination du comportement à la rupture - Partie 1 : Détermination de l'indice de rupture des émulsions cationiques de bitume, méthode des fines minérales

Bitumen und bitumenhaltige Bindemittel - Bestimmung des Brechverhaltens - Teil 1: Bestimmung des Brechwertes kationischer Bitumenemulsionen, Verfahren mit Feinmineralstoff

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European foreword

This document (EN 13075-1:2016) 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of an identical text or by endorsement, at the latest by May 2017, and conflicting national standards shall be withdrawn at the latest by May 2017.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 13075-1:2009.

In comparison with EN 13075-1:2009, the following significant changes have been made:

— Clause 5 : three reference fillers (Forshammer, Sikaisol and Caolin Q92) may be used;

— Subclauses 6.4.3 and 6.4.4: calibration range of the filler feeder and accuracy requirements for the timer are extended so as to cover the case of slow breaking emulsions;

— Subclause 6.4.8 is discarded since this clause is not called by the test procedure (Clause 8) and since requirements on temperature control are already stated in 6.4.1 (oven) and 6.4.6 (temperature bath or climatic chamber);

— Subclauses 8.2 and 8.3: more accurate and more complete description of the test procedure. Calculation and reporting (Clause 12) of the actual filler feeding rate;

— Clause 9: New factors for the conversion of measured breaking values into "Forshammer equivalents". The conversion factors for Sikaisol and Caolin Q92 have been established from an extensive Round Robin test program conducted by TC336/WG2 in 2014 (see bibliographic reference N°2);

— Subclause 11.2: A value for Reproducibility is now stated, as an outcome of the TC336/WG2 Round Robin;

— 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 breaking value of cationic bituminous emulsions.

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 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 term and definition applies.

3.1
breaking value "*BV*"
dimensionless number corresponding to the mass of reference filler, in grams, needed to coagulate 100 g of bitumen emulsion

4 Principle

A reference filler is added at a uniform rate to a specified quantity of stirred cationic bitumen emulsion. When the emulsion has broken completely, the mass of added filler is determined by weighing. The mass of filler (in grams) multiplied by 100 and divided by the mass of emulsion (in grams) is the breaking value.

NOTE The cationic or anionic nature of an emulsion can be determined with EN 1430 [1].

5 Reagents and materials

5.1 Reference fillers.

The Forshammer filler[1)], the Sikaisol filler[1)] or the Caolin Q92 filler[1)] shall be used as reference fillers while applying the conversion factors given in Clause 9. The characteristics of these fillers are given in Annex A.

In the event of dispute, the same filler and the same procedure (manual or semi-automatic) shall be used by the testing laboratories.

5.2 Cleaning agents, as used conventionally in laboratories.

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1) This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN of the products 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

6.1 General

Usual laboratory apparatus and glassware, together with specific equipment that is described below depending on the used procedure (semi-automatic or manual). An explanatory sketch of the equipment set-up is shown in Figure 1 for the semi-automatic procedure.

6.2 Equipment for semi-automatic procedure

6.2.1 Stirrer motor, as shown in Figure 1, with an output power of at least 25 W, and a speed of (260 ± 60) r/min.

6.2.2 Stirrer, as shown in Figure 2, having the dimensions given in Figure 3, Figure 4 and Figure 5. In Figure 3, the dimensions are given as an example.

6.2.3 Metal cans, cylindrical, of approximate capacity 500 ml, height 95 mm and diameter 90 mm.

6.3 Equipment for manual procedure

6.3.1 Enamelled or stainless steel dish, having approximately 20 cm inside diameter and 10 cm high.

6.3.2 Spatula, nickel or stainless steel, approximately 20 cm long.

6.4 Equipment for both procedures

6.4.1 Oven, capable of maintaining a temperature of (110 ± 5) °C.

6.4.2 Conical-shaped funnel, capable of supplying a continuous sufficient flow of filler to the filler feeder.

6.4.3 Adjustable filler feeder, to be placed at the outlet of the filler holding funnel and capable of feeding the filler at a rate of (0,35 ± 0,10) g/s. This equipment shall be calibrated. The calibration shall be achieved by weighing the mass of the filler poured during a period of time between 100 s and 600 s depending on the anticipated test duration and measured with an accuracy of 0,2 s.

The feeding rate, q, in g/s, shall be calculated, using Formula (1):

$$q = \frac{m_f}{t} \tag{1}$$

where

m_f is the mass of filler, in grams;

t is the analysis time, in seconds.

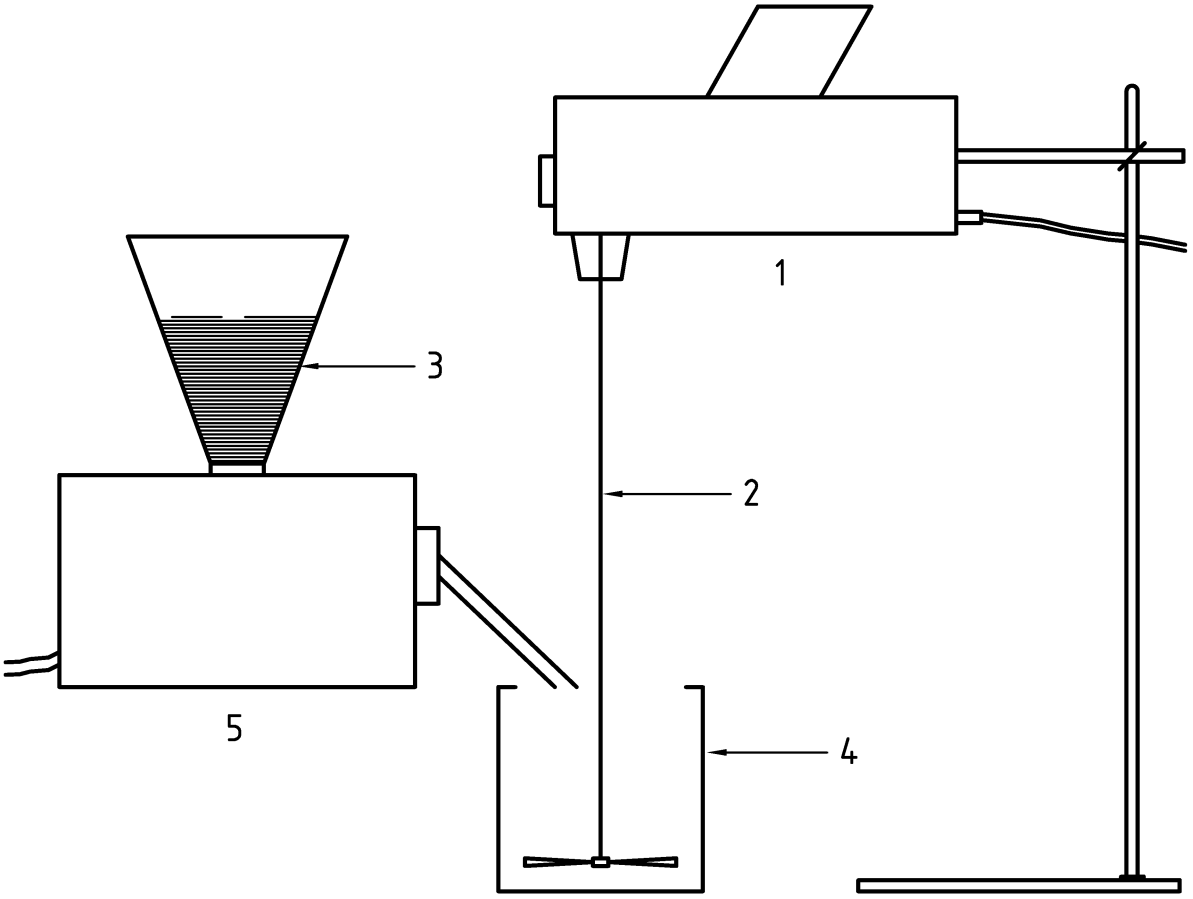
6.4.4 Timer or stop watch, with an accuracy of 0,2 s or better over a time interval equal to or higher than 600 s.

6.4.5 Bottles, of approximate capacity 500 ml made of a material that will not react with the emulsion, having tight fitting lids.

6.4.6 Constant temperature bath and/or climatic chamber, capable of maintaining the sample in the can at (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 plastic bottles from moving in the water bath.

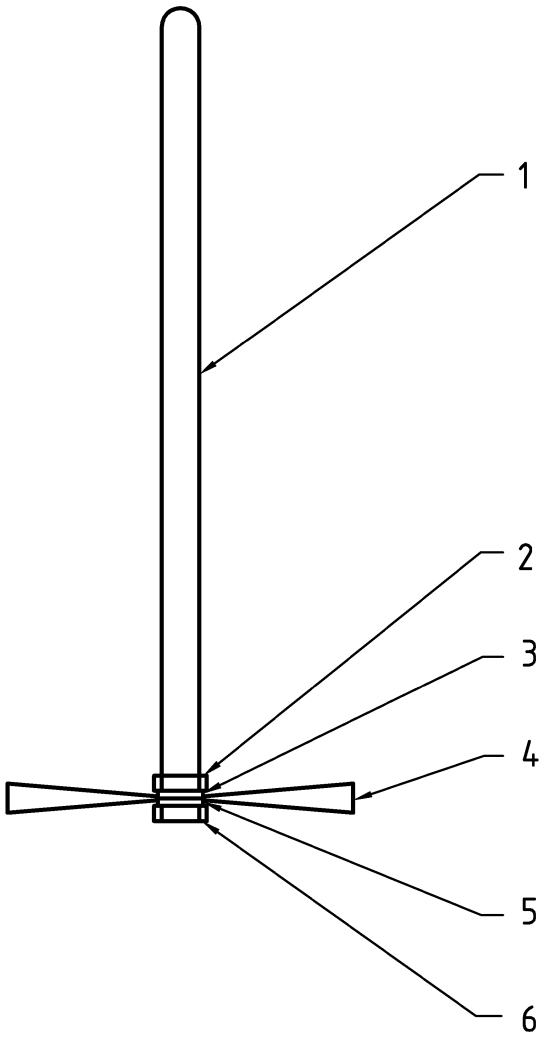
6.4.7 Balance, having a suitable range, capable of weighing the samples in Clause 8 to the nearest 0,1 g.



Key

1 stirrer motor
2 stirrer
3 filler
4 sample can
5 feeder

Figure 1 — Explanatory sketch of equipment for determination of breaking value of bituminous emulsion

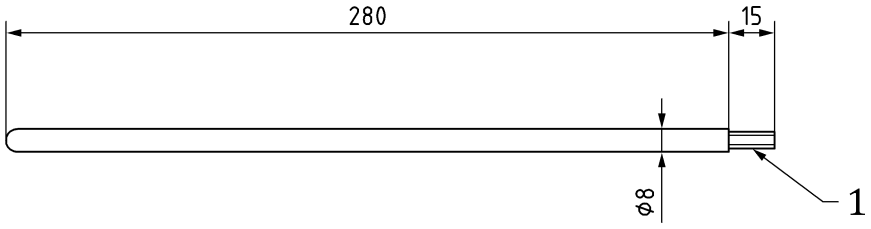


Key

1 stirrer rod
2 nut
3 washer
4 stirrer blades
5 washer
6 nut

Figure 2 — Design of stirrer

Dimensions in millimetres



Key

1 M6 metric thread

Figure 3— Example stirrer rod (informative)

Dimensions in millimetres

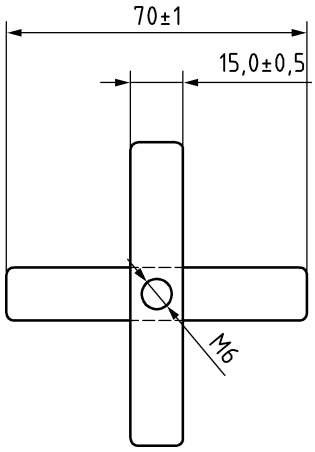


Figure 4 — Stirrer blades

Dimensions in millimetres

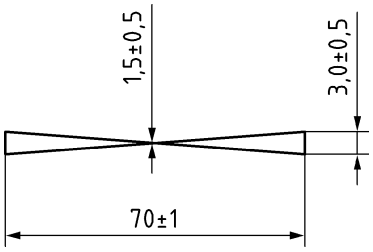


Figure 5 — Stirrer blades geometry

7 Sampling

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

8 Procedure

8.1 General

Carry out the procedure under normal laboratory conditions (18 °C to 28 °C). However, before carrying out any one of the two procedures:

— dry the quantities of filler, required for the test, in the oven (6.4.1) at a temperature of (110 ± 5) °C until constant mass and cool to ambient temperature in a desiccator;

— pour a portion of emulsion (250 ± 10) g into a bottle (6.4.5) and secure the lid;

— pour the required quantities of filler in a container and close the container;

— place the bottle with the emulsion and the container with the filler in the constant temperature bath or climatic chamber (6.4.6) for a minimum of 1,5 h;

— transfer the required quantity of filler from the container into the hopper of the adjustable filler feeder.

8.2 Semi-automatic procedure

Weigh the metal can (6.2.3) containing the stirrer (6.2.2) to the nearest 0,1 g (m_1).

Transfer (100 ± 1) g, weighed to the nearest 0,1 g (m_e) of the emulsion sample from the bottle (6.4.5) to the weighed metal can.

Place the metal can under the stirrer motor (6.2.1) and connect the stirrer (6.2.2) to the stirrer motor.

Start the stirrer motor. Ensure that the stirrer blades rotate at (260 ± 60) r/min and are below the surface of the emulsion during the test. Then, start the filler feeder and the timer simultaneously.

Rotate the metal can slowly (approximately 5 r/min) by hand in the opposite direction to the stirrer in order to ensure homogeneity of mixing.

The mixture becomes thicker as the filler is added and the emulsion is considered broken when the mix comes off completely (or substantially) from the metal can. At this point, stop the filler feeder and the timer and then the stirrer motor. Note the actual filler feeding time (t_a) to the nearest 0,2 s.

Weigh the metal can containing the broken emulsion and the stirrer to the nearest 0,1 g (m_2).

Calculate the actual feeding rate using Formula (2):

$$q_a = \frac{\left(m_2 - m_1 - m_e\right)}{t_a} \tag{2}$$

where

m_2 is the mass of the metal can containing the broken emulsion and the stirrer, in grams;

m_1 is the mass of the metal can containing the stirrer, in grams;

m_e is the mass of the emulsion, in grams;

t_a is the actual filler feeding time, in seconds.

Verify that q_a = (0,35 ± 0,10) g/s. If not, redo the calibration of the filler feeder according to 6.4.3 and redo the test.

Repeat the test with a second portion of emulsion taken from the same bottle using a second metal can and stirrer.

8.3 Manual procedure

Weigh the dish (6.3.1) containing the spatula (6.3.2) to the nearest 0,1 g (m_1).

Transfer (100 ± 1) g, weighed to the nearest 0,1 g (m_e) of the emulsion sample from the bottle (6.4.5) to the weighed dish containing the spatula.

Start the filler addition and the timer simultaneously. Thoroughly mix the emulsion and the filler by stirring at a steady rate of 1 r/s, using the spatula.

The mixture becomes thicker as the filler is added and the emulsion is considered broken when the mix comes off completely (or substantially) from the dish. Stop stirring and filler addition and the timer at this point. Note the actual filler feeding time (t_a) to the nearest 0,2 s.

Weigh the dish containing the broken emulsion and the spatula to the nearest 0,1 g (m_2).

Calculate the actual feeding rate using Formula (2)

where

m_2 is the mass of the dish containing the broken emulsion and the spatula, in grams;

m_1 is the mass of the dish containing the spatula, in grams;

m_e is the mass of the emulsion, in grams;

t_a is the actual filler feeding time, in seconds.

Verify that q_a = (0,35 ± 0,10) g/s. If not, redo the calibration of the filler feeder according to 6.4.3 and redo the test.

Repeat the test with a second portion of emulsion taken from the same bottle using a second dish and spatula.

9 Calculation

Calculate the breaking value, *BV* for 100 g of emulsion, using Formula (3):

$$BV = \frac{100 \times m_f}{m_e} \tag{3}$$

where

m_f = $m_2 - m_e - m_1$ is the added mass of filler in grams;

m_e is the mass of emulsion in grams.

The result shall be converted into the "Forshammer equivalent" value using the following conversion factors [2]:

— when using Forshammer filler: $BV_{\text{Forshammer}}$ = 1 x *BV*;

— when using Sikaisol filler: $BV_{\text{Forshammer}}$ = 1,3 x *BV*;

— when using Caolin Q92 filler: $BV_{\text{Forshammer}}$ = 1,2 x *BV*.

10 Expression of results

Express the individual breaking values to the nearest 0,1.

Express the result as the arithmetic mean of the two individual results of the breaking value, to the nearest integer.

11 Precision

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

11.1 Repeatability, *r*

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 10 % of the mean value, in only one case in twenty.

11.2 Reproducibility, R

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 40 % of the mean value, in only one case in twenty.

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) procedure used (semi-automatic or manual);

d) actual feeding rate and rotational speed (Clause 8);

e) filler used;

f) result of the test and individual breaking values, BV Foshammer (see Clause 9 and Clause 10);

g) any deviation, by agreement or otherwise, from the procedure described;

h) 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):

— SiO_2: from 74,3 % to 76,8 %

— Al_2O_3: from 14,1 % to 14,8 %

— K_2O: from 3,6 % to 4,7 %

— Na_2O: from 4,2 % to 5,2 %

The particle size distribution envelope, according to EN 933-10 [4], is shown in Figure A.1 and defined by:

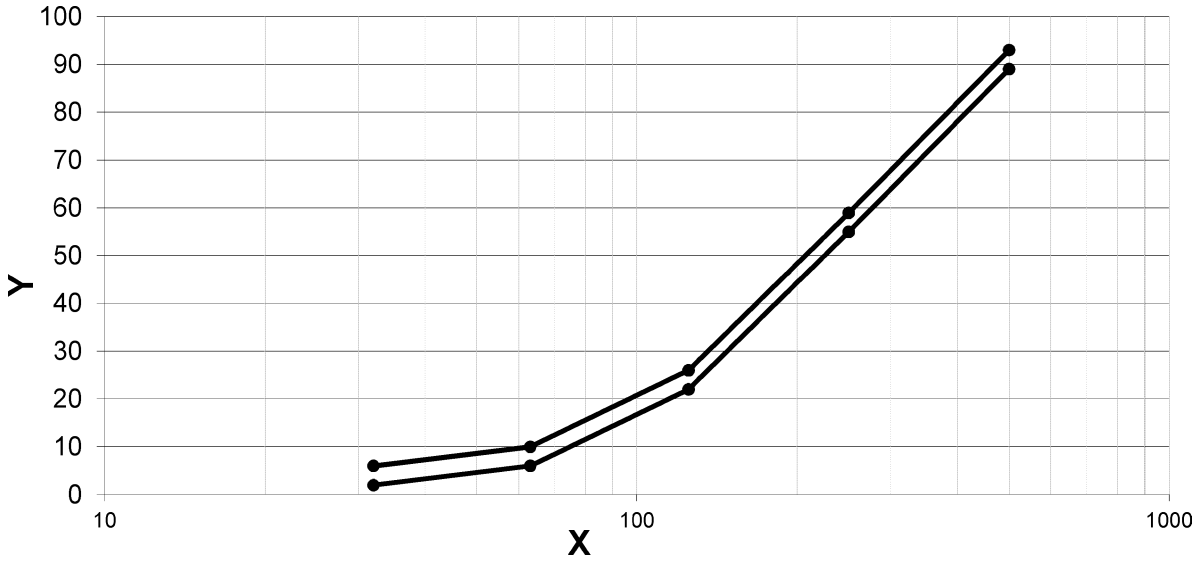
— 89 % to 93 % passing the 0,500 mm sieve;

— 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;

— SiO_2 content greater than 98 %;

— density of (2650 ± 20) kg/m^3;

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

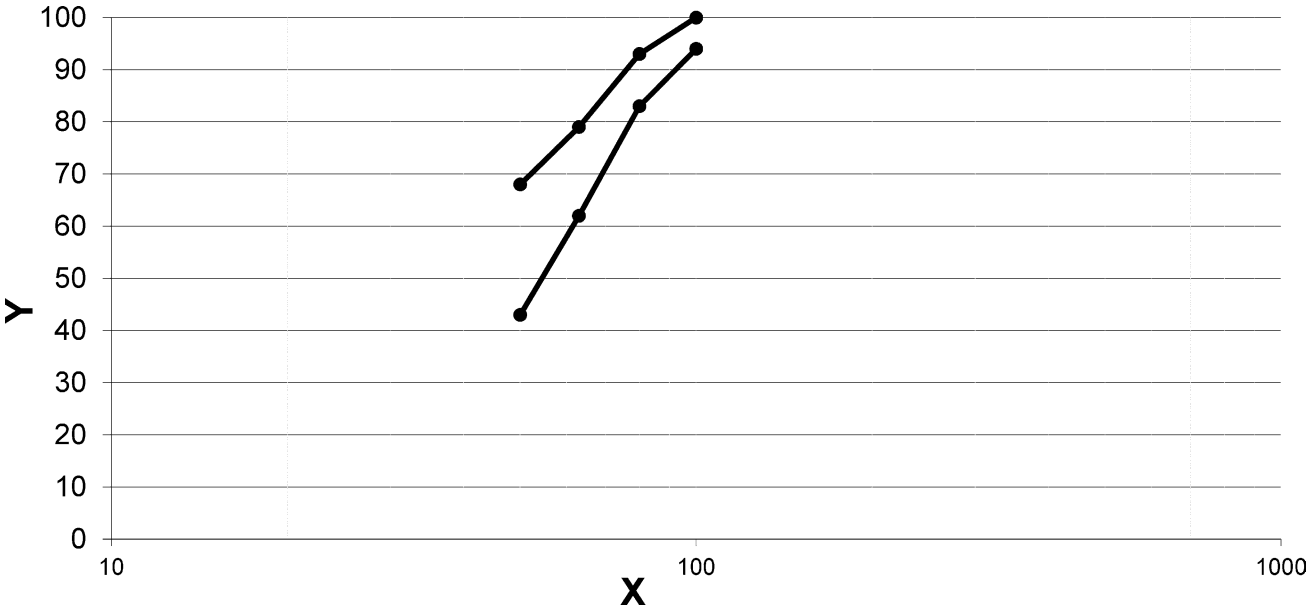
— 94 % to 100 % passing the 0,100 mm sieve;

— 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 %

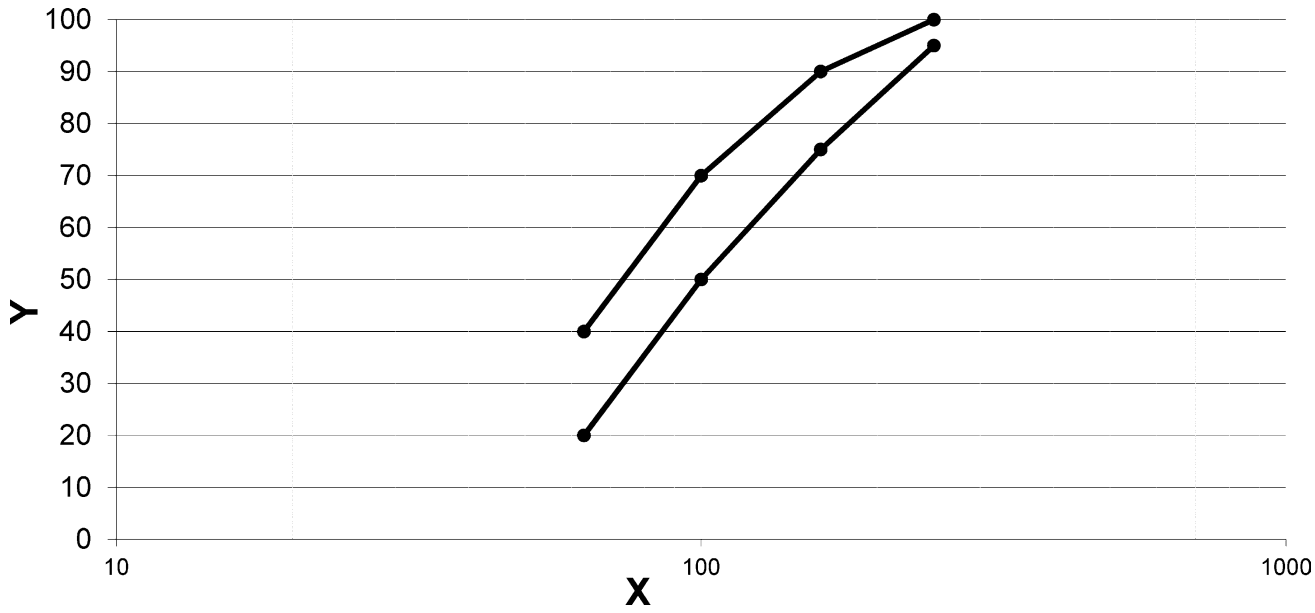
The particle size distribution envelope, according to EN 933-1 [5], is shown in Figure A.3 and defined by:

— 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

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[1] EN 1430, *Bitumen and bituminous binders - Determination of particle polarity of bituminous emulsions*

[2] CEN TC 336/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

[3] 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*

[4] EN 933-10, *Tests for geometrical properties of aggregates - Part 10: Assessment of fines - Grading of filler aggregates (air jet sieving)*

[5] EN 933-1, *Tests for geometrical properties of aggregates - Part 1: Determination of particle size distribution - Sieving method*

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BSI Group Headquarters

389 Chiswick High Road London W4 4AL UK

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