SLOVENSKI STANDARD
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Preskusi geometričnih lastnosti agregatov - 9. del: Ugotavljanje finih delcev - Preskus z metilen modrim

Tests for geometrical properties of aggregates - Part 9: Assessment of fines - Methylene blue test

Prüfverfahren für geometrische Eigenschaften von Gesteinskörnungen - Teil 9: Beurteilung von Feinanteilen - Methylenblau-Verfahren

Essais pour déterminer les caractéristiques géométriques des granulats - Partie 9 : Qualification des fines - Essais au bleu de méthylène

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COMITÉ EUROPÉEN DE NORMALISATION
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European foreword

This document (EN 933-9:2022) has been prepared by Technical Committee CEN/TC 154 “Aggregates”, the secretariat of which is held by BSI.

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of an identical text or by endorsement, at the latest by August 2022, and conflicting national standards shall be withdrawn at the latest by August 2022.

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 933-9:2009+A1:2013.

In comparison with the previous edition, the following technical modifications have been made:

a) Normative references has been extended with EN 933-2 which is referenced in 6.1.7 Test sieves;

b) Terms and definitions has been extended with the definition of laboratory sample;

c) Clause 6 Apparatus has been restructured into two subclauses (6.1 general and 6.2 special), due to special apparatus (tinted glass bottle) used in former Annex C;

d) the test sieves in 6.1.7 are extended with 0,125 mm, which is used in normative Annex A;

e) the note in former Clause 6.4 about alternative types of mixers has been transformed to main text;

f) pre-drying temperature has been increased to (110 ± 5) °C for natural and manufactured aggregates (Clause 7) and the clause has been divided into General, Preparation without pre-drying and Preparation with pre-drying;

g) the procedure has been clarified with restructured text and an illustrating figure (Clause 8);

h) the Scope, the Terms and definitions and the Test report content have been adapted to the current rules and the text has been clarified;

i) the order of annexes has been changed, to place the normative annexes first;

j) the note in former Annex D, containing recommendations about checking of MB_{K}, has been transformed to main text in a new Clause C.1 General;

k) Annex A and former Annexes C and D have been restructured;

l) the titles of Annex A and former Annex D have been shortened;

m) the row numbers in Annex E have been deleted.

This document forms part of a series of tests for geometrical properties of aggregates. Test methods for other properties of aggregates are covered by the following European Standards:

— EN 932 (all parts), *Tests for general properties of aggregates*

— EN 1097 (all parts), *Tests for mechanical and physical properties of aggregates*

— EN 1367 (all parts), *Tests for thermal and weathering properties of aggregates*

— EN 1744 (all parts), *Tests for chemical properties of aggregates*

— EN 13179 (all parts), *Tests for filler aggregate used in bituminous mixtures*

The other parts of EN 933 include:

— *Part 1: Determination of particle size distribution — Sieving method*

— *Part 2: Determination of particle size distribution — Test sieves, nominal size of apertures*

— *Part 3: Determination of particle shape — Flakiness index*

— *Part 4: Determination of particle shape — Shape index*

— *Part 5: Determination of percentage of crushed and broken surfaces in coarse aggregate particles*

— *Part 6: Assessment of surface characteristics — Flow coefficient of aggregates*

— *Part 7: Determination of shell content — Percentage of shells in coarse aggregates*

— *Part 8: Assessment of fines — Sand equivalent test*

— *Part 10: Assessment of fines — Grading of filler aggregates (air jet sieving)*

— *Part 11: Classification test for the constituents of coarse recycled aggregate*

Any feedback and questions on this document should be directed to the users' national standards body. A complete listing of these bodies can be found on the CEN website.

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, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

1 Scope

This document specifies the reference method used for type testing and in cases of dispute, for the determination of the methylene blue value of the size 0/2 mm fraction in fine aggregates or all-in aggregates (*MB*). It also specifies the reference method for the determination of the methylene blue value of the size 0/0,125 mm fraction (MB_F) in normative Annex A. Other methods can be used for other purposes, such as factory production control, provided that an appropriate working relationship with the suitable reference method has been established.

Annex B specifies the preparation of 10 g/l methylene blue solution and Annex C specifies the procedure for the determination of the methylene blue value of kaolinite (MB_k). Annexes B and C are normative.

A conformity check, adding a single quantity of dye solution equivalent to a specified limiting value and which can be used as part of a production control process, is described in informative Annex D.

An example of a test data sheet is given in informative Annex E.

WARNING – The use of this part of EN 933 can involve hazardous materials, operations and equipment (such as dust, noise and heavy lifts). It does not purport to address all of the safety or environmental problems associated with its use. It is the responsibility of users of this document to take appropriate measures to ensure the safety and health of personnel and the environment prior to application of the standard, and fulfil statutory and regulatory requirements for this purpose.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 932-2, *Tests for general properties of aggregates - Part 2: Methods for reducing laboratory samples*

EN 932-5, *Tests for general properties of aggregates - Part 5: Common equipment and calibration*

EN 933-2, *Tests for geometrical properties of aggregates - Part 2: Determination of particle size distribution - Test sieves, nominal size of apertures*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp/ui

— IEC Electropedia: available at https://www.electropedia.org/

3.1
laboratory sample
sample intended for laboratory testing

3.2
subsample
sample obtained by means of a sample reduction procedure

3.3
test portion
d_0/D_0
sample used as a whole in a single test

3.4
fines
particle size fraction of an aggregate which passes the 0,063 mm sieve

3.5
particle size fraction
d_i/D_i
fraction of an aggregate passing the larger (D_i) of two sieves and retained on the smaller (d_i)

Note 1 to entry: The lower limit can be zero.

3.6
constant mass
mass determined after successive weighings at least 1 h apart not differing by more than 0,1 %

Note 1 to entry: In many cases constant mass can be achieved after a test portion has been dried for a pre-determined period in a specified oven at (110 ± 5) °C. Test laboratories can determine the time required to achieve constant mass for specific types and sizes of sample dependent upon the drying capacity of the oven used.

4 Principle

Increments of a methylene blue solution are added successively to a test portion suspended in water. The adsorption of dye solution, by potential clay minerals in the test portion, is checked after each addition of solution. This is done by carrying out a stain test on filter paper to detect the presence of free dye, which indicates that adsorption is complete.

When the presence of free dye is confirmed, the methylene blue value (MB or MB_F) is calculated and expressed as grams of dye adsorbed per kilogram of the size fraction tested.

NOTE A conformity check, adding a single quantity of dye solution equivalent to a specified limiting value, and which can be used as part of a production control process, is described in Annex D.

5 Reagents

5.1 Dye solution, solution of standard or technical quality methylene blue, (10,0 ± 0,1) g/l (see Annex B). The solution shall not be used more than 28 days after preparation. It shall be stored in a dark place.

5.2 Distilled or demineralized water.

5.3 Kaolinite, of known methylene blue value MB_K (see Annex C).

NOTE Kaolinite of MB_K value between 1 g and 2 g per 100 g of kaolinite is preferable in order to avoid excessive use of dye.

6 Apparatus

All apparatus, unless otherwise stated, shall conform to the general requirements of EN 932-5.

6.1 Apparatus for general purposes

6.1.1 Burette, pipette, micro pipette or bottle top dispenser, capable of delivering a volume of 5 ml and 2 ml with an accuracy of 0,2 ml.

6.1.2 Filter paper, quantitative and ash-free (<0,010 %); 95 g/m²; thickness 0,20 mm; filtration speed 75 s; pore size 8 µm.

6.1.3 Glass rod, length 300 mm; diameter 8 mm.

6.1.4 Impeller agitator, capable of controlled variable rotation rates up to (600 ± 60) r/min with three or four impeller blades of (75 ± 10) mm diameter. Other types of mixers that have been shown to produce the same results may be used.

6.1.5 Balance, accurate to 0,1 % of the mass of the test portion.

6.1.6 Timer.

6.1.7 Test sieves, 0,125 mm and 2 mm, with apertures as specified in EN 933-2, with guard sieve (if necessary).

6.1.8 Sieve brush.

6.1.9 Beaker, glass or plastic, capacity of about 1 l or about 2 l.

6.1.10 Flask, glass, capacity of 1 l.

6.1.11 Ventilated oven, capable of maintaining a temperature of (110 ± 5) °C.

6.1.12 Thermometer, accurate to 1 °C.

6.1.13 Spatula.

6.1.14 Desiccator.

6.2 Special apparatus for the preparation of 10 g/l methylene blue solution (Annex B)

6.2.1 Tinted glass bottle.

7 Preparation of test portion

7.1 General

The test portion shall consist of at least 200 g of particle size fraction 0/2 mm. It may be prepared using pre-dried or not pre-dried material.

When testing a non-pre-dried material, a correction of the mass with respect to the water content is necessary. Thus, the water content shall be determined in this case.

The preparation without pre-drying is described in 7.2 and the preparation with pre-drying is described in 7.3.

All masses shall be determined to the nearest 1 g.

7.2 Preparation without pre-drying

Reduce the laboratory sample in accordance with EN 932-2 to produce two subsamples, each containing at least 200 g of particle size fraction 0/2 mm.

NOTE 1 One subsample is aimed for determining the water content in order to calculate the dry mass M_1 of the test portion. In this way the test portion used for the *MB* determination is not exposed for any heating.

Sieve the subsamples on a 2 mm sieve, protected if necessary by a guard sieve, e.g. 4 mm, and using a sieve brush to ensure effective separation and collection of all particles in the size 0/2 mm fraction. Discard any particles retained on the 2 mm sieve.

Determine the water content *W* by weighing one of the sieved subsamples, *M.* Dry it to constant mass, and weigh it again as *M*'. Calculate and record *W* according to Formula (1), and discard this subsample.

$$W = 100 \times \frac{(M - M')}{M'} \quad (1)$$

where

W is the water content, in percent (%);

M is the mass of the sieved subsample, in grams (g);

M' is the mass of the sieved subsample dried to constant mass, in grams (g).

NOTE 2 The water content determination can be achieved by other means than drying in a ventilated oven, such as drying in a microwave for example.

Take the second sieved subsample and, if necessary, achieve further reduction in accordance with EN 932-2. Sample reduction shall yield a test portion size which is larger than [200 × (1 + *W*/100)] g, but not of an exact predetermined value.

Weigh the test portion as M_0 and calculate M_1 (dry mass), according to Formula (2):

$$M_1 = \frac{M_0}{1 + (W/100)} \quad (2)$$

where

M_1 is the dry mass of the test portion, in grams (g);

M_0 is the mass of the test portion, in grams (g);

W is the water content determined on the other subsample, in percent (%).

7.3 Preparation with pre-drying

Reduce the laboratory sample in accordance with EN 932-2 to yield a test portion size which is larger than the minimum, but not of an exact predetermined value.

Dry the sample to constant mass, at (110 ± 5) °C in the case of natural and manufactured aggregates, and at less than 45 °C in the case of other type of aggregates.

Sieve the sample on a 2 mm sieve, protected if necessary by a guard sieve, e.g. 4 mm, and using a sieve brush to ensure effective separation and collection of all particles in the size 0/2 mm fraction. Discard any particles retained on the 2 mm sieve.

Weigh the obtained test portion as M_1.

8 Procedure

8.1 Preparation of suspension

Place (500 ± 5) ml of distilled or demineralized water in the beaker (6.1.9) and add the test portion while stirring well with the spatula (6.1.13).

Stir the dye solution (5.1) or alternatively mix it thoroughly. Fill the burette (6.1.1) with dye solution and return the stock of dye solution to a dark place.

Set the agitator (6.1.4) to a speed of 600 r/min and position the impeller about 10 mm above the base of the beaker. Switch on the agitator and start the timer (6.1.6), agitating the contents of the beaker for 5 min at (600 ± 60) r/min.

Reduce the speed of the agitator to (400 ± 40) r/min and agitate continuously for the remainder of the test.

If insufficient fines are present in the test portion to obtain a halo (see 8.2), kaolinite shall be added together with additional dye solution as follows:

— add to the beaker (30,0 ± 0,1) g of kaolinite (5.3), dried at (110 ± 5) °C to constant mass;

— add V' ml of dye solution to the beaker, where $V' = 30\ MB_K$, i.e. the volume of dye solution adsorbed by 30 g of kaolinite.

8.2 Stain test

Place a filter paper (6.2) on top of an empty beaker, or some other suitable support, so that most of its surface is not in contact with any solid or liquid.

Add a dose of 5 ml of dye solution into the beaker with the suspension and agitate at (400 ± 40) r/min for at least 1 min.

Apply a drop of the dyed suspension on the filter paper (6.1.2) by means of the glass rod (6.1.3). The stain formed on the filter paper is composed of a central deposit of material, of a generally solid blue colour, surrounded by a colourless wet zone. The amount of drop applied shall be such that the diameter of the deposit is between 8 mm and 12 mm. If this is not fulfilled, a new drop shall be applied.

The test result is said to be positive if, in the wet zone, a halo consisting of a light blue ring of about 1 mm is formed around the central deposit and persists for 5 min. This detects the presence of free dye, which indicates that adsorption is complete.

If the halo does not appear after the addition of the initial 5 ml of dye solution, add a further 5 ml of dye solution, continue agitating for 1 min, and carry out another stain test.