SLOVENSKI STANDARD
oSIST prEN 933-9:2020

01-november-2020

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

Ta slovenski standard je istoveten z: **prEN 933-9**

ICS:

91.100.15	Mineralni materiali in izdelki	Mineral materials and products

oSIST prEN 933-9:2020 **en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

DRAFT
prEN 933-9

September 2020

ICS 91.100.15

Will supersede EN 933-9:2009+A1:2013

English Version

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This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 154.

If this draft becomes a European Standard, CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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European foreword

This document (prEN 933-9:2020) has been prepared by Technical Committee CEN/TC 154 “Aggregates”, the secretariat of which is held by BSI.

This document is currently submitted to the CEN Enquiry.

This document will supersede EN 933-9:2009+A1:2013.

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

a) Pre-drying temperature has been increased for natural and manufactured aggregates (Clause 7);

b) The procedure of stain tests has been clarified (8.3);

c) The test report content has been adapted to the current rules (Clause 10);

d) The note in Annex D, containing recommendations, has been transformed to main text (D.9).

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 particles in coarse and all-in natural aggregates*

— *Part 6: Assessment of surface characteristics — Flow coefficient for coarse 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 fillers (air jet sieving)*

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

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 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 0/0,125 mm fraction (MB_F) in 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.

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 B.

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

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*

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:

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at https://www.iso.org/obp

3.1
subsample
sample obtained by means of a sample reduction procedure

3.2
test portion
***d*$_0$/*D*$_0$**
sample used as a whole in a single test

3.3
fines
particle size fraction of an aggregate which passes the 0,063 mm sieve

3.4
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.

3.5
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 solution of methylene blue are added successively to a suspension of the test portion in water. The adsorption of dye solution by the test portion is checked after each addition of solution by carrying out a stain test on filter paper to detect the presence of free dye.

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 B.

5 Reagents

5.1 Dye solution, solution of standard or technical quality methylene blue, (10,0 ± 0,1) g/l (see Annex C). The maximum period of use of the solution shall be 28 days. It shall be stored away from light.

5.2 Distilled or demineralized water.

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

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 shall conform to the general requirements of EN 932-5.

6.1 Burette, with capacity of either 100 ml or 50 ml and graduation of either 1/10 ml or 1/5 ml, or one 5 ml and one 2 ml micro-pipette.

6.2 Filter paper, quantitative and ash-free (<0,010 %); 95 g/m^2; thickness 0,20 mm; filtration speed 75 s; pore size 8 µm.

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

6.4 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.

NOTE Alternative types of mixer can be used if it can be shown that results obtained agree with results produced using an impeller agitator as specified above.

6.5 Balance, readable to 0,1 % of the mass to be weighed.

6.6 Stopwatch or **stopclock**, readable to 1 s.

6.7 Test sieve, 2 mm aperture, with guard sieve (if necessary).

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

6.9 Flask, glass, capacity of 1 l.

6.10 Ventilated oven, thermostatically controlled to maintain a temperature of (110 ± 5) °C.

6.11 Thermometer, readable to 1 °C.

6.12 Spatula.

6.13 Desiccator.

7 Preparation of test portions

The laboratory samples shall be reduced in accordance with EN 932-2 to produce two subsamples, each containing at least 200 g of 0/2 mm particle size. Sieve each subsample on a 2 mm sieve, protected if necessary by a guard sieve, using a sieve brush to ensure effective separation and collection of all particles in the 0/2 mm fraction. Discard any particles retained on the 2 mm sieve.

The subsamples may be pre-dried at a temperature of (110 ± 5) °C in the case of natural and manufactured aggregates, and less than 45 °C in the case of other type of aggregates, to facilitate the sieving operation.

Weigh one of the subsamples as M. Dry it to constant mass, then weigh it again as M'. Determine and record the water content of this subsample as W (%) = 100 × (M − M') / M'. Discard this subsample.

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

Take the other subsample and, if necessary, achieve further reduction in accordance with EN 932-2 to obtain a test portion of mass at least [200 × (1 + W / 100)] g. The mass of the test portion shall be larger than [200 × (1 + W / 100)] g but not of an exact predetermined value. Weigh the test portion as M_0 and determine its dry mass M_1 to the nearest 1 g according to the following formula:

$$M_1 = M_0 / (1 + W / 100) \quad (1)$$

8 Procedure

8.1 Description of the stain test

After each injection of dye, the stain test consists of taking a drop of suspension by means of the glass rod and depositing it on the filter paper. The stain that is formed is composed of a central deposit of material, of a generally solid blue colour, surrounded by a colourless wet zone.

The amount of drop taken shall be such that the diameter of the deposit is between 8 mm and 12 mm.

The test is deemed positive if, in the wet zone, a halo consisting of a persistent light blue ring of about 1 mm is formed around the central deposit.

NOTE As the end-point is approached, the halo will appear, but can then disappear again, because the clay minerals can take some time to complete their adsorption of the dye. For this reason, the end-point is confirmed by repeating the stain test at 1 min intervals for 5 min without adding more dye solution.

8.2 Preparation of suspension

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

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

Set the agitator 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 stopwatch, agitating the contents of the beaker for 5 min at (600 ± 60) r/min and subsequently (see 8.3) agitate continuously at (400 ± 40) r/min for the remainder of the test.

If insufficient fines are present in the test portion to obtain a halo, kaolinite should 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 $V' = 30\,MB_K$ is the volume of dye solution adsorbed by 30 g of kaolinite.

8.3 Determination of the quantity of dye adsorbed

Place the 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.

After agitating for 5 min at (600 ± 60) r/min, inject a dose of 5 ml of dye solution (see 5.1) into the beaker; agitate at (400 ± 40) r/min for at least 1 min and carry out a stain test (see 8.1) on the filter paper. If after the addition of this initial 5 ml of dye solution the halo does not appear, add a further 5 ml of dye solution, continue agitating for 1 min, and carry out another stain test. If a halo still does not appear, continue agitating, making additions of dye and doing stain tests in this manner until a halo is observed.

When this stage is reached, continue agitating and without further additions of dye solution, perform stain tests at 1 min intervals.

From this point, if the halo disappears during the following 4 min, add a further 5 ml of dye solution.

If the halo disappears during the following fifth minute, add only 2 ml of dye solution. In either case, continue agitating and doing stain tests until a halo persists for 5 min, corresponding to six consecutive spots starting from the addition of methylene blue (Figure 1).

Record the total volume of dye solution V_1 added to produce a halo that persists for 5 min, to the nearest 1 ml.

Containers used in methylene blue tests should be reserved specifically for that test. Containers should be cleansed thoroughly with water as soon as the tests are completed and traces of any detergents used should be removed by thorough rinsing.

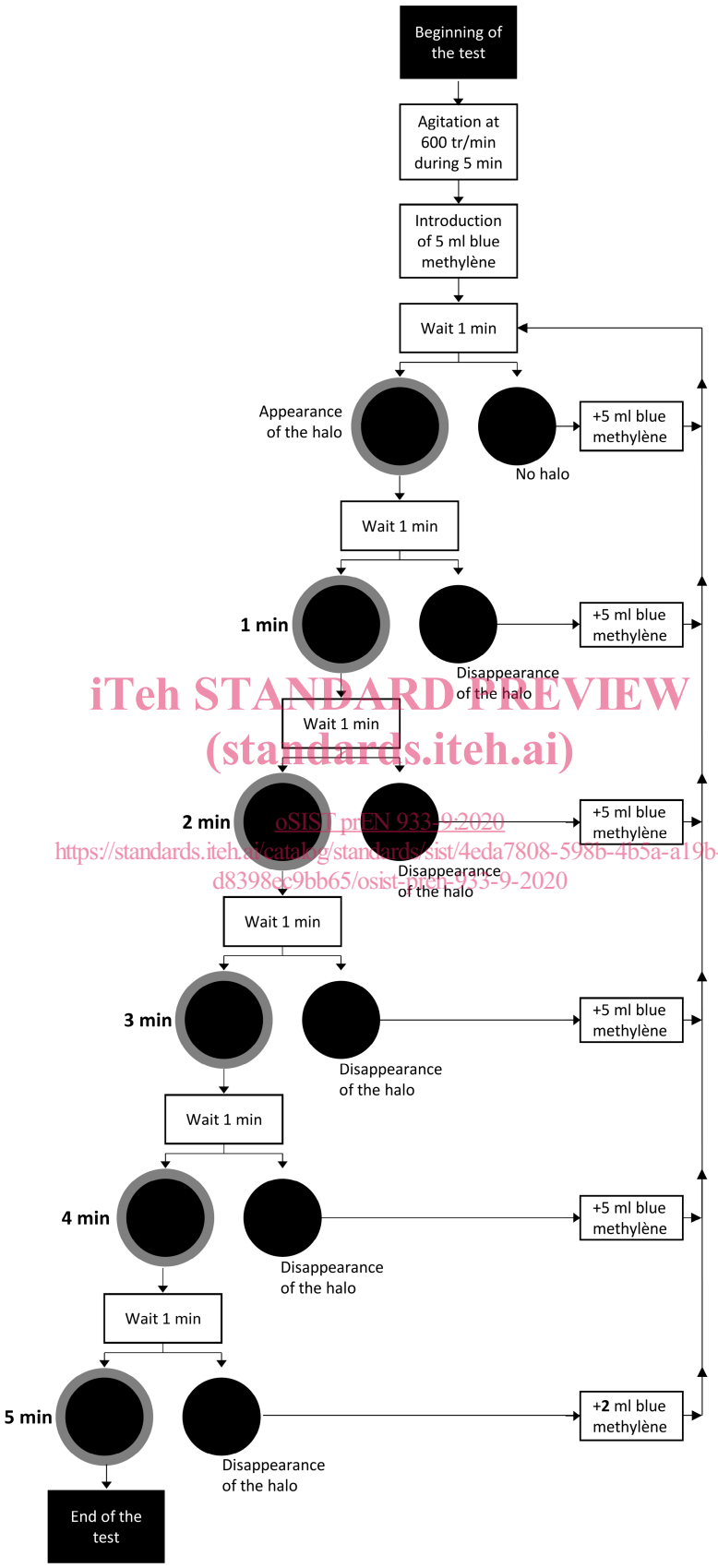

Figure 1 — Flow chart of test procedure