RATIFIED EUROPEAN TEXT

EN933-9:1998

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English version

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

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

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

This European Standard was approved by CEN on 1 October 1998.

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CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

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

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

Contents

Foreword

This European Standard 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of an identical text or by endorsement, at the latest by April 1999, and conflicting national standards shall be withdrawn at the latest by December 1999.

This Standard forms part of a series of tests for geometrical properties of aggregates. Test methods for other properties of aggregates will be covered by Parts of the following European Standards:

prEN 932	Tests for general properties of aggregates
prEN 1097	Tests for mechanical and physical properties of aggregates
prEN 1367	Tests for thermal and weathering properties of aggregates
prEN 1744	Tests for chemical properties of aggregates
prEN 13179	Tests for filler aggregate used in bituminous mixtures

The other parts of EN 933 will be:

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 for coarse aggregates
Part 7: Determination of shell content - Percentage of shells in coarse aggregates
Part 8: Assessment of fines - Sand equivalent
Part 10: Assessment of fines - Grading of fillers (air jet sieving)

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom

1 Scope

This European Standard specifies a method for the determination of the methylene blue value of the 0/2 mm fraction in fine aggregates or all-in aggregates (*MB*). A procedure for the determination of the methylene blue value of the 0/0,125 mm fraction (MB_F) is specified in annex A.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references, the latest edition of the publication referred to applies.

prEN 932-2	Tests for general properties of aggregates - Part 2: Methods for reducing laboratory samples
prEN 932-5	Tests for general properties of aggregates - Part 5: Common equipment and calibration

3 Definitions

For the purposes of this standard, the following definitions apply.

3.1 subsample: Sample obtained by means of a sample reduction procedure.

3.2 test portion: The sample used as a whole in a single test.

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

3.4 particle size fraction: Fraction of an aggregate passing the larger of two sieves and retained on the smaller.

NOTE: The lower limit can be zero.

3.5 constant mass: Successive weighings after drying at least 1 h apart not differing by more than 0,1 %.

NOTE: In many cases constant mass can be achieved after a test specimen 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 may 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 demineralised 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 prEN 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 75s; 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) min^{-1} 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 1s.

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

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

6.9 Flask, glass, capacity 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 prEN 932-2 to produce a subsample containing at least 200 g of 0/2 mm particle size.

Dry the subsample at (110 ± 5) °C to constant mass and allow to cool.

Sieve the dry subsample on a 2 mm sieve protected if necessary by a guard sieve, and 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 and, if necessary, reduce the fraction passing the 2 mm sieve in accordance with prEN 932-2 to obtain a test portion of mass at least 200 g. The mass of the test portion shall be larger than 200 g but not of an exact predetermined value.

Weigh the test portion and record the mass to the nearest 1 g as M_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 which 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 to be 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 demineralised water in the beaker and add the dried 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 min^{-1} 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) min^{-1} and subsequently (see **8.3**) agitate continuously at (400 ± 40) r/min for the remainder of the test.

Switch on the agitator and start the stopwatch, agitating the contents of the beaker for 5 min at (600 ± 60) min^{-1} 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) min^{-1}, inject a dose of 5 ml of dye solution (see **5.1**) into the beaker; agitate at (400 ± 40) min^{-1} 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.

If the halo disappears during the first 4 min, add a further 5 ml of dye solution. If the halo disappears during the 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.

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

NOTE : Containers should be cleansed thoroughly with water as soon as the tests are completed. Traces of any detergents used should be removed by thorough rinsing. It is recommended that containers used in methylene blue tests are reserved specifically for that test.

9 Calculation and expression of results

The methylene blue value, MB, expressed in grams of dye per kilogram of the 0/2 mm fraction is given by the following equation:

$$MB = \frac{V_1}{M_1} \cdot 10$$

where:

M_1 is the mass of the test portion, in grams;
V_1 is the total volume of dye solution injected, in millilitres.

Record the MB value to the nearest 0,1 g of dye per kilogram of the 0/2 mm fraction.

If the test is carried out with the addition of kaolinite, the above equation becomes:

$$MB=\frac{V_1-V'}{M_1}\cdot 10$$

where: V' is the volume of dye solution adsorbed by the kaolinite, in millilitres.

NOTE 1: The factor 10 in the above equations converts the volume of dye solution used to the mass of dye adsorbed per kilogram of the size fraction tested.

NOTE 2: An example of a test data sheet is given in annex **E**.

10 Test report

The test report shall include the information referred to in **10.1** and can include the information referred to in **10.2**.

10.1 Required data

a) reference to this European Standard;
b) identity of laboratory;
c) identification of the sample;
d) description of the material tested;
e) *MB* value;
f) date of receipt of sample;
g) sampling certificate, if available.

10.2 Optional data

a) name and location of the sample source;
b) description of the sample reduction procedure.
c) date of test.

Annex A (normative)

Procedure for the determination of the methylene blue value (MB_F) of the 0/0,125 mm fraction

A.1 Prepare test portions as specified in clause **7** and follow the test procedure in clause **8**, but with a test portion mass M_1 of (30,0 ± 0,1) g of the 0/0,125 mm fraction.

A.2 Calculate the methylene blue value (MB_F) in grams of dye per kilogram of the 0/0,125 mm fraction as:

$$MB_F = \frac{V_1}{M_1} \cdot 10$$

where:

M_1 is the mass of the test portion, in grams;
V_1 is the total volume of dye solution added, in millilitres.

A.3 Record the MB_F value to the nearest 0,1g of dye per kilogram of the 0/0,125 mm fraction.

A.4 Test reports shall include appropriate information in accordance with clause 10.

Annex B (informative)

Test of conformity in relation to a specified MB value

A check on conformity with a specified *MB* value can be carried out by making a single addition of dye solution in the following manner.

If the specified *MB* value expressed as grams of dye per kilogram of 0/2 mm fraction is 'MB_1' then the volume of dye solution to be injected at one time, V_2, is given by the following equation:

$$V_2 = \frac{MB_1 \cdot M_1}{10} + V'$$

where:

M_1 is the mass of the test portion, in grams;
MB_1 is the specified *MB* value, in grams of dye per kilogram of 0/2 mm fraction;
V' is the volume of dye solution in millilitres adsorbed by any added kaolinite.

After preparation of a test portion in accordance with clause **7**, the suspension should be prepared using the test portion, the water and, if necessary the kaolinite, all in accordance with **8.2**, but including V_2 ml of dye solution.

The stain test should be carried out after stirring the suspension for 8 min at (400 ± 40) min^{-1}. If the stain test (see **8.1**) is positive, the sand can be assumed to conform to the specification.

If this stain test is negative however, the complete determination described in **8.3** should be carried out.

Annex C (normative)

Preparation of 10 g/l methylene blue solution

C.1 Prepare the 10 g/l dye solution following the procedure given in **C.1.1** to **C.1.7**.

C.1.1 Use methylene blue; ($C_{16}H_{18}ClN_3S$, nH_2O (n = 2 to 3) purity ≥ 98,5 %).

C.1.2 Determine the water content W of the methylene blue powder as follows:

Weigh approximately 5 g of methylene blue powder and record the mass to the nearest 0,01 g as M_h.

Dry this powder at (100 ± 5) °C to constant mass. Cool in the desiccator, and then weigh immediately after taking out of the desiccator. Record the dry mass to the nearest 0,01 g as M_g.

NOTE: At temperatures above 105 °C, methylene blue powder can be modified.

Calculate and record the water content W to the nearest decimal place from the following equation:

$$W = \frac{Mh - Mg}{Mg} \cdot 100$$

where:

M_h is the mass of the methylene blue powder, in grams;
M_g is the mass of the dried methylene blue powder, in grams.

The water content shall be determined for the preparation of every new batch of dye solution.

C.1.3 Take a mass of methylene blue powder of ((100 + W)/10) g ± 0,01 g (equivalent to 10 g of dry powder).

C.1.4 Warm 500 ml to 700 ml of distilled or demineralised water in a beaker to a temperature not exceeding 40 °C.

C.1.5 Agitate the contents of the beaker whilst slowly pouring the methylene blue powder into the warm water. Continue to agitate for 45 min, until complete dissolution of the powder, and then allow to cool to 20 °C.

C.1.6 Pour into a flask of capacity 1 l, rinsing with distilled or demineralised water to ensure complete transfer of all dye into the flask. Make sure that the flask and the water are at a temperature of (20 ± 1)°C to conform with the calibration of the flask and add more distilled or demineralised water to the 1 l graduation mark.

C.1.7 Shake the flask to ensure complete dissolution of the powder and pour into a conservation bottle in tinted glass.

C.2 The following details shall be marked on the conservation bottle:

a) 10 g/l methylene blue solution;
b) date of preparation;
c) limit date of use.

C.3 Methylene blue solution shall not be used more than 28 days after preparation. The stock of dye solution shall be stored in a dark place.

Annex D (normative)

Procedure for the determination of the methylene blue value of kaolinite (MB_K)

D.1 Dry the kaolinite at (110 ± 5) °C to constant mass.

D.2 Weigh (30,0 ± 0,1) g of dry kaolinite.

D.3 Pour the (30,0 ±0,1) g of kaolinite into the beaker **(6.8)** together with 500 ml of demineralised or distilled water.

D.4 Agitate for 5 min at (600 ± 60) min^{-1} with the impeller about 10 mm above the base of the container and subsequently agitate continuously at (400 ± 40) min^{-1} for the remainder of this determination.

D.5 Inject a dose of 5 ml of 10 g/l dye solution into the beaker and, after at least 1 min of agitating at (400 ± 40) min^{-1}, carry out a stain test (see **8.1**) on the filter paper.

D.6 If necessary continue to add dye solution in 5 ml doses until a positive result is obtained without adding any more solution. Leave the adsorption of blue, which is not instantaneous, to proceed while carrying out stain tests each minute.

If the light blue ring disappears on the fifth stain, further increments of 2 ml of dye shall be added.

Each addition shall be followed by tests carried out at intervals of 1 min.

These operations shall be repeated until the test remains positive for 5 consecutive min. The determination is then complete.

D.7 Record the total volume of dye solution adsorbed as V' in millilitres.

D.8 Calculate and record the methylene blue value of the kaolinite to the nearest 0,1 g of dye per 100 g of kaolinite from the following equation:

$$MB_K = V'/30$$

where:

V' is the total volume of dye solution adsorbed, in millilitres.

NOTE: A test on kaolinite of known MB_K value should be carried out at regular intervals to check the constancy of results. This procedure should also be used to check a new dye solution.

Annex E (informative)
Example of a test data sheet

EN 933-9	Laboratory :
	Date :
Identification of the sample :	
	Operator :

E.1 Dry mass of test portion 0/2 mm (to nearest gram) M_1	M_1 = g
E.2 Volume of solution adsorbed by kaolinite (if used) V	V = ml
E.3 Total amount of dye solution added V_1	V_1 = ml
E.4 *MB* value, expressed in grams of dye per kilogram of the 0/2 mm fraction (see clause 9)	*MB* =