



**SLOVENSKI STANDARD**  
**SIST EN 933-9:2009**

**01-oktober-2009**

**BUXca Yý U**  
**SIST EN 933-9:1999**

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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 - Essai au bleu de méthylène

**Ta slovenski standard je istoveten z: EN 933-9:2009**

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**ICS:**

91.100.15 Mineralni materiali in izdelki Mineral materials and products

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EUROPEAN STANDARD  
NORME EUROPÉENNE  
EUROPÄISCHE NORM

**EN 933-9**

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## 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 12 June 2009.

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## Foreword

This document (EN 933-9:2009) 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of an identical text or by endorsement, at the latest by January 2010, and conflicting national standards shall be withdrawn at the latest by January 2010.

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

This document supersedes EN 933-9:1998.

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:

- EN 932, *Tests for general properties of aggregates*
- EN 1097, *Tests for mechanical and physical properties of aggregates*
- EN 1367, *Tests for thermal and weathering properties of aggregates*
- EN 1744, *Tests for chemical properties of aggregates*
- EN 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 test
- 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, Bulgaria, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and the United Kingdom.

**EN 933-9:2009 (E)****1 Scope**

This standard describes 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 describes the reference method for the determination of the methylene blue value of the 0/0,125 mm fraction (MB<sub>F</sub>) in Annex A. For other purposes, in particular factory production control, other methods may be used provided that an appropriate working relationship with the suitable reference method has been established.

**2 Normative references**

The following referenced documents are indispensable for the application 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.

**3.1****subsample**

sample obtained by means of a sample reduction procedure

**3.2****test portion**

sample used as a whole in a single test

**3.3****finer**

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  $(110 \pm 5) ^\circ\text{C}$ . 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,  $(10,0 \pm 0,1)$  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.

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## 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 ( $< 0,010$  %);  $95 \text{ g/m}^2$ ; thickness 0,20 mm; filtration speed 75 s; pore size  $8 \mu\text{m}$ .

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

**6.4** *Impeller agitator*, capable of controlled variable rotation rates up to  $(600 \pm 60) \text{ min}^{-1}$  with three or four impeller blades of  $(75 \pm 10)$  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 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  $(110 \pm 5) ^\circ\text{C}$ .

**6.11** *Thermometer*, readable to  $1 ^\circ\text{C}$ .

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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, 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.

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  $W$  (%) =  $100 \times (M - M')/M'$ . Discard this subsample.

NOTE The determination of the water content may 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  $[200 \times (1 + W/100)]$  g. The mass of the test portion shall be larger than  $[200 \times (1 + W/100)]$  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:

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

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**8 Procedure****8.1 Description of the stain test**

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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  $(500 \pm 5)$  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  $600 \text{ min}^{-1}$  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  $(600 \pm 60) \text{ min}^{-1}$  and subsequently (see 8.3) agitate continuously at  $(400 \pm 40) \text{ r/min}$  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  $(30,0 \pm 0,1)$  g of kaolinite (5.3), dried at  $(110 \pm 5)$  °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  $(600 \pm 60)$  min<sup>-1</sup>, inject a dose of 5 ml of dye solution (see 5.1) into the beaker; agitate at  $(400 \pm 40)$  min<sup>-1</sup> 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.

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## 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:

$$MB = \frac{V_1}{M_1} \cdot \times 10 \quad (2)$$

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:

$$MB = \frac{V_1 - V'}{M_1} \times 10 \quad (3)$$

where