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Preskusi mehanskih in fizikalnih lastnosti agregatov - 7. del: Določevanje prostorninske mase zrn kamene moke - Postopek s piknometrom

Tests for mechanical and physical properties of aggregates - Part 7: Determination of the particle density of filler - Pycnometer method

Prüfverfahren für mechanische und physikalische Eigenschaften von Gesteinskörnungen - Teil 7: Bestimmung der Rohdichte von Füller - Pyknometer-Verfahren

Essais pour déterminer les caractéristiques mécaniques et physiques des granulats - Partie 7: Détermination de la masse volumique réelle du filler - Méthode au pycnomètre

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EUROPEAN STANDARD
NORME EUROPÉENNE
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English Version

Tests for mechanical and physical properties of aggregates - Part 7: Determination of the particle density of filler - Pyknometer method

Essais pour déterminer les caractéristiques
mécaniques et physiques des granulats - Partie 7:
Détermination de la masse volumique réelle du filler -
Méthode au pycnomètre

Prüfverfahren für mechanische und physikalische
Eigenschaften von Gesteinskörnungen - Teil 7:
Bestimmung der Rohdichte von Füller - Pyknometer-
Verfahren

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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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

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European foreword

This document (prEN 1097-7:2020) has been prepared by Technical Committee CEN/TC 154 “Aggregates”, the secretariat of which is held by BSI.

This document is currently submitted to CEN Enquiry.

This document will supersede EN 1097-7:2008.

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

- a) Precision of the water bath temperature has been revised from $(25,0 \pm 0,1)$ °C to $(25,0 \pm 3,0)$ °C or $(25,0 \pm 0,5)$ °C depending on the liquid used. As a consequence, texts in Clauses 6.3, 8, A.2.4 and B.2.4 have been revised.
- b) In the Scope, a reference to methods for determination of particle density of aggregates has been added.
- c) The “European foreword”, “Normative references”, “Terms and definitions” and “Test report” clauses are updated.
- d) Terms and definitions have been clarified to correspond with definitions in EN 1097-6.
- e) The “Principle”, “Preparation of test portion” and “Procedure” clauses have been clarified.
- f) In the “Materials” clause, low viscosity white mineral oils have been added among the examples given in the note.
- g) In “Procedure”, the note about adding liquid carefully has been transformed into main text.
- h) In the Bibliography, EN 1097-6 has been added.

In this document, the Annexes A and B are normative and the Annex C is informative.

This document forms part of a series of tests for mechanical and physical 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 933 (all parts), *Tests for geometrical 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 1097 include:

- *Part 1: Determination of the resistance to wear (micro-Deval)*
- *Part 2: Methods for the determination of resistance to fragmentation*
- *Part 3: Determination of loose bulk density and voids*

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- *Part 4: Determination of the voids of dry compacted filler*
- *Part 5: Determination of the water content by drying in a ventilated oven*
- *Part 6: Determination of particle density and water absorption*
- *Part 8: Determination of the polished stone value*
- *Part 9: Determination of the resistance to wear by abrasion from studded tyres – Nordic test*
- *Part 10: Determination of water suction height*

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1 Scope

This document specifies the reference method used for type testing and in cases of dispute for the determination of the particle density of filler by means of a pycnometer. For other purposes, in particular factory production control, other methods can be used provided that an appropriate working relationship with the reference method has been established.

NOTE Methods for determination of particle density of aggregates are specified in EN 1097-6.

Annexes are included that specify the procedures for calibration of the pycnometer (Annex A) and determination of the density of the liquid used to determine the particle density of the filler (Annex B).

Annex C (informative) contains precision data.

WARNING – The use of this part of EN 1097 can involve hazardous materials, operations and equipment (such as liquids, 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 932-2:1999, *Tests for general properties of aggregates - Part 2: Methods for reducing laboratory samples*
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EN 932-5, *Tests for general properties of aggregates - Part 5: Common equipment and calibration*

ISO 3507, *Laboratory glassware - Pycnometers*

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/ui>

3.1

laboratory sample

sample intended for laboratory testing

3.2

test portion

sample used as a whole in a single test

prEN 1097-7:2020 (E)**3.3****test specimen**

sample used in a single determination when a test method requires more than one determination of a property

3.4**constant mass**

mass determined by successive weighings after drying performed at least 1 h apart and 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 (see 6.6) 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.

3.5**particle density of filler**

ρ_r

ratio obtained by dividing the pre-dried mass of a test specimen of filler by the volume it occupies in a liquid, including the volume of any internal sealed voids but excluding the volume of liquid in any liquid accessible voids

Note 1 to entry: The definition corresponds to the definition of the term 'pre-dried particle density' in EN 1097-6:2013.

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3.6**filler aggregate**

aggregate, most of which passes a 0,063 mm sieve

Note 1 to entry: Added fillers are filler aggregates of mineral origin that have been produced separately and can be added to construction materials to provide certain properties.

4 Principle

The pycnometer method is a method for determining the volume of irregularly formed samples, e.g. aggregate. When the mass of the sample is known, the density can be calculated from the ratio of mass to volume.

The principle is based on the replacement of a certain amount of liquid of known density with the test specimen. A pycnometer with known volume, containing the test specimen, is topped up with the liquid. The volume of this liquid is calculated as the mass of the liquid added divided by the liquid density. The volume of the test specimen is then calculated by subtraction of this volume from the pycnometer volume.

5 Materials

5.1 Suitable liquid, in which the filler does not dissolve and with which the filler does not react.

NOTE Water, denatured ethanol, redistilled kerosene, low viscosity white mineral oils or toluene have been found to be suitable for different types of filler.

5.2 Demineralized water, boiled and cooled, for calibration (see Annex A).

5.3 Acetone, for calibration (see Annex A).

6 Apparatus

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

6.2 Pyknometer, of nominal capacity 50 ml, conforming to ISO 3507. The stopper shall be concave at the underside, and shall include a thick-walled capillary (riser pipe) whose top has been ground to a level surface.

NOTE The pyknometer can be fitted with a thermometer.

6.3 Water bath, thermostatically controlled, capable of being maintained at $(25,0 \pm 3,0)$ °C if water is used as the liquid in the pyknometer, or $(25,0 \pm 0,5)$ °C for other liquids such as those mentioned in the note in 5.1.

6.4 Balance, accurate to the nearest 0,001 g for the determination.

6.5 Balance, accurate to the nearest 0,000 1 g for calibration (see Annex A).

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

6.7 Desiccator, filled with an appropriate amount of desiccant.

6.8 Vacuum desiccator.

6.9 Vacuum pump, capable of achieving a residual pressure of less than 3,0 kPa.

6.10 Spatula.

6.11 Test sieve, 0,125 mm and suitable receiver.

7 Preparation of test portion

The mass of the laboratory sample shall have at least 100 g of particles smaller than 0,125 mm.

Reduce the size of the laboratory sample in accordance with EN 932-2 to obtain a test portion which shall consist of three test specimens of filler particles smaller than 0,125 mm, each having a dry mass of approximately 25 g.

Dry the laboratory sample (or the reduced sample) in the oven at (110 ± 5) °C to constant mass and leave it to cool down in the desiccator (see 6.7) for at least 90 min. Check the test sample for the presence of lumps and, if present, pulverize them carefully with the spatula and mix the pulverized lumps.

Dry sieve the test sample on the 0,125 mm sieve. Retain all particles which pass the sieve.

Prepare three test specimens of the retained particles in accordance with EN 932-2:1999, Clause 11.

8 Procedure

Carry out the determination of density using three separate test specimens, using a calibrated pyknometer or pyknometers (see Annex A) and a liquid of a known density (see Annex B). Carry out all weighing with an accuracy of 0,001 g.

Proceed as follows for each of the three determinations.

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Weigh the clean and dry pyknometer with stopper (m_0). Fill the pyknometer with the test specimen, which takes about half the volume of the pyknometer, and weigh the pyknometer with filler (m_1). Add sufficient liquid to completely submerge the test specimen. Add the liquid very carefully allowing it to percolate through the filler in the pyknometer.

The amount of filler may be varied to optimize the volume ratio of filler and liquid, in order to reduce the influence of the temperature on the density of the pyknometer liquid. This amount added is calculated as $(m_1 - m_0)$ and may be held constant for next measurements of the same filler.

Put the stopper in the pyknometer, place the pyknometer in the vacuum desiccator and evacuate it with the vacuum pump in approximately 5 min to less than 3,0 kPa. Leave the pyknometer for at least 30 min in the vacuum desiccator at a pressure less than 3,0 kPa.

After restoring the air pressure in the desiccator, take the pyknometer out and fill it with liquid. Place the pyknometer without stopper in the water bath at $(25 \pm 3,0)^\circ\text{C}$ or $(25 \pm 0,5)^\circ\text{C}$ depending on the liquid used (see 6.3) so that the top protrudes between 2 mm to 3 mm above the water level in the bath. After 60 min, put the stopper in the pyknometer causing an amount of liquid to come out of the capillary.

Dry the top of the capillary and remove the pyknometer from the water bath. Quickly cool the pyknometer in cold (running) water, to prevent liquid expanding out of the capillary due to warm handling. Carefully dry the outside and weigh the pyknometer filled with test specimen and liquid (m_2).

9 Calculation and expression of results

Calculate the particle density of the filler, in megagrams per cubic metre, in accordance with the following formula:

$$\rho_f = \frac{m_1 - m_0}{V - \frac{m_2 - m_1}{\rho_l}} \quad (1)$$

where

ρ_f is the particle density of the filler at 25 °C, in megagrams per cubic metre;

m_0 is the mass of the empty pyknometer with stopper, in grams;

m_1 is the mass of the pyknometer with the dry filler test specimen, in grams;

m_2 is the mass of the pyknometer with the filler test specimen, topped up with liquid (see 5.1), in grams;

V is the volume of the pyknometer, in millilitres (see Annex A);

ρ_l is the density of liquid at 25 °C, in megagrams per cubic metre (see Annex B).

Calculate the particle density of the filler as the mean of the three determinations, and round off to the nearest 0,01 Mg/m³.

NOTE A statement on the precision of this test is given in Annex C.