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

Prüfverfahren für mechanische und physikalische Eigenschaften von Gesteinskörnungen - Teil 7: Bestimmung der Rohdichte von Füllern - 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 COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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European foreword

This document (EN 1097-7:2022) 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of an identical text or by endorsement, at the latest by April 2023, and conflicting national standards shall be withdrawn at the latest by April 2023.

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 1097-7:2008.

This document has been prepared under a Standardization Request given to CEN by the European Commission and the European Free Trade Association.

In comparison with the previous edition EN 1097-7:2008, the following technical modifications have been made:

a) in the Scope, a reference to methods for determination of particle density of aggregates has been added;

b) Clause 3 “Terms and definitions” has been clarified to correspond with definitions in EN 1097-6. The definition of size fraction d_i/D_i has been added;

c) in Clause 5 “Materials”, low viscosity white mineral oils have been added among the examples of suitable liquids given in the note;

d) in 6.3, the required accuracy of the water bath temperature has been reduced from (25,0 ± 0,1) °C to either (25,0 ± 3,0) °C or (25,0 ± 0,5) °C depending on the liquid used. Texts in Clause 8, A.2.5 and B.2.4 have been revised accordingly;

e) the required accuracy of the balance for the pyknometer volume determination has been reduced from 0,000 1 g to 0,001 g, since water is always used for this purpose. The texts in 6.4, 6.5 and A.2.2 have been revised accordingly;

f) in Clauses 7 and 8, the required masses of the test portion and the test specimens have been revised. The possibility to vary the filler amount, and thus the ratio of filler and liquid, has been added in order to reduce the influence of the temperature on the density of the pyknometer liquid;

g) the required number of test specimens has been reduced from three to two, provided that the two test results differ less than 0,025 Mg/m^3. The texts in Clauses 7, 8 and 9 have been revised accordingly;

h) in Clause 8 “Procedure”, the note about adding liquid carefully has been transformed into main text;

i) in Clause 10 “Test report”, the density of the liquid used for the determination is moved from Required data to Optional data;

j) the title of Annex A has been revised to “Determination of the pyknometer volume” since *calibration* was not the correct term. All texts referring to *calibration* have been corrected accordingly. A new paragraph (A.2.4) has been added to describe the evacuation of the pyknometer by means of vacuum;

k) the title of Annex B has been shortened and B.1 has been clarified regarding the use of Annex B;

l) Annex C has been removed since the precision data given cannot be tracked down any longer. Consequently, the note about precision statement in Clause 9 has been revised;

m) in the Bibliography, EN 1097-6 and EN 1936 have been added and the national standards have been deleted.

In addition, the whole document has been updated and the “Principle”, “Preparation of test portion”, “Procedure” and Annex A have been clarified.

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 the EN 1097 series 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*

— 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*

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, Türkiye 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 particle density of filler by means of a pyknometer. 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.

Annex A specifies the procedure for determination of the pyknometer volume. Annex B specifies the procedure for determination of the density of the liquid used to determine the particle density of the filler. Annexes A and B are normative.

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*

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

ISO 3507, *Laboratory glassware — Pyknometers*

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
test portion
sample used as a whole in a single test

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_f
ratio obtained by dividing the pre-dried mass of a test specimen of filler by the volume it occupies in a liquid, including the volumes 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.

3.6
filler aggregate
aggregate, most of which passes a 0,063 mm sieve, which can be added to construction materials to provide certain properties

Note 1 to entry: Added fillers are filler aggregates of mineral origin that have been produced separately.

3.7
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)

4 Principle

The pyknometer 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 pyknometer 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 pyknometer 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 pyknometer volume determination (see Annex A).

5.3 Acetone, for pyknometer volume determination (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 ± 3,0) °C if water is used as the liquid in the pyknometer, or (25,0 ± 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 filler density determinations when water is used as the liquid, and for the pyknometer volume determination (see Annex A).

6.5 Balance, accurate to the nearest 0,000 1 g, for filler density determinations and for the liquid density determination when other liquids than water are used (see Annex B). This balance may also be used for determination of the pyknometer volume.

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 laboratory sample shall contain at least 100 g of the size fraction 0/0,125 mm.

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

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.

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

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

8 Procedure

Carry out the density determination using two test specimens, by means of a pyknometer or pyknometers of a known volume (see Annex A) and a liquid of a known density (see Annex B). Carry out all weighings with an accuracy of 0,001 g (see 6.4).

Proceed as follows for each of the two determinations.

Weigh the clean and dry pyknometer with stopper, and record as m_0. Fill the pyknometer with the test specimen, which takes about half the volume of the pyknometer. Weigh the pyknometer with filler and record as 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 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 ± 3,0) °C or (25 ± 0,5) °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 Formula (1):

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

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 two determinations, and round off to the nearest 0,01 Mg/m^3.

If the two individual test results differ more than 0,025 Mg/m^3, a third test shall be carried out. If the difference between at least two of the three measurements is within the precision limit of 0,025 Mg/m^3,