

# **Tests for mechanical and physical properties of aggregates —**

## **Part 7: Determination of the particle density of filler — Pyknometer method**

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ICS 91.100.15

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### Summary of pages

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 6, an inside back cover and a back cover.

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This British Standard, having been prepared under the direction of the Sector Committee for Building and Civil Engineering, was published under the authority of the Standards Committee and comes into effect on 15 October 1999

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ISBN 0 580 32771 X

### Amendments issued since publication

Amd. No.	Date	Comments

EUROPEAN STANDARD

EN 1097-7

NORME EUROPÉENNE

EUROPÄISCHE NORM

July 1999

ICS 91.100.15

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 Dichte von Füller —  
Pyknometer-Verfahren

This European Standard was approved by CEN on 11 June 1999.

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Ref. No. EN 1097-7:1999 E

## Foreword

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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of an identical text or by endorsement, at the latest by January 2000, and conflicting national standards shall be withdrawn at the latest by December 2003.

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.

This European Standard forms part of a series of standards for tests for mechanical and physical 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 933, *Tests for geometrical properties of aggregates.*

EN 1367, *Tests for thermal and weathering properties of aggregates.*

EN 1744, *Tests for chemical properties of aggregates.*

prEN 13179, *Tests for filler aggregate used in bituminous mixtures.*

The other parts of EN 1097 will be:

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, *Method for the determination of the resistance to wear by abrasion from studded tyres: Nordic test.*

Part 10, *Water suction height.*

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## 1 Scope

This European Standard specifies the procedure for determining the particle density of filler by means of a pycnometer.

## 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.

EN 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.*

prEN 1097-4, *Tests for mechanical and physical properties of aggregates — Part 4: Determination of the voids of dry compacted filler.*

ISO 3507:1976, *Pycnometers.*

## 3 Terms and definitions

For the purposes of this standard, the following terms and definitions apply:

### 3.1

#### **laboratory sample**

reduced sample derived from a bulk sample 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**

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 portion has been dried for a pre-determined period in a specified oven (see 6.6) 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.

### 3.5

#### **particle density of filler**

mass per volume unit of filler excluding any trapped air

### 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

## 4 Principle

The pycnometer method is a well known 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.

The principle is based on the replacement of a certain amount of liquid of known density with the test portion. A pycnometer with known volume, containing the test portion, is topped up with the liquid. The volume of this liquid is calculated by dividing the mass of the liquid added by the liquid density. The volume of the test portion 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 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 prEN 932-5.

6.2 *Pycnometer*, 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 pycnometer can be fitted with a thermometer.

6.3 *Water bath*, capable of being maintained at  $(25,0 \pm 0,1)^\circ\text{C}$ .

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

6.5 *Balance*, accurate to the nearest 0,0001 g for calibration (see annex A).

6.6 *Drying oven*, thermostatically controlled to maintain a temperature of  $(110 \pm 5)^\circ\text{C}$ .

**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  $(3,0 \pm 0,3)$  kPa.

**6.10** *Spatula*.

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

## 7 Preparation of test portion

Reduce the size of the laboratory sample in accordance with EN 932-2. The test portion before drying shall have a minimum mass of 50 g.

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

Dry sieve the filler using the 0,125 mm sieve. Retain all the particles which pass the sieve.

## 8 Procedure

Carry out the determination of density using three separate test specimens, using a calibrated pycnometer or pycnometers (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.

Weigh the clean and dry pycnometer with stopper ( $m_0$ ). Fill the pycnometer with  $(10 \pm 1)$  g of filler taken from the test portion and weigh it again ( $m_1$ ). Add sufficient liquid to completely submerge the test specimen.

Put the stopper in the pycnometer, place the pycnometer in the vacuum desiccator and evacuate it with the vacuum pump in approximately 5 min to  $(3,0 \pm 0,3)$  kPa. Leave the pycnometer for 30 min in the vacuum desiccator at  $(3,0 \pm 0,3)$  kPa.

After restoring the air pressure in the desiccator, take the pycnometer out and fill it with liquid. Place the pycnometer without stopper in the water bath at  $(25,0 \pm 0,1)$  °C 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 pycnometer causing an amount of liquid to come out of the capillary.

Dry the top of the capillary and remove the pycnometer from the water bath. Quickly cool the pycnometer in cold (running) water, to prevent liquid expanding out of the capillary due to warm handling. Carefully dry the outside and weigh the pycnometer 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 equation:

$$\rho_f = \frac{m_1 - m_0}{V - \frac{m_2 - m_1}{\rho_l}}$$

where:

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

$m_1$  is the mass of the pycnometer with the filler test portion, in grams;

$m_2$  is the mass of the pycnometer with the filler test portion, topped up with liquid (see 5.1), in grams;

$V$  is the volume of the pycnometer, in millilitres (see annex A);

$\rho_l$  is the density of liquid at 25 °C, in megagrams per cubic metre (see annex B);

$\rho_f$  is the particle density of the filler at 25 °C, in megagrams per cubic metre.

Calculate the particle density of the filler as the mean of the three determinations, and round off to the nearest 0,01 Mg/m<sup>3</sup>.

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

## 10 Test report

### 10.1 Required data

The test report shall include the following information:

- the particle density  $\rho_f$  of the filler;
- reference to this European Standard;
- the brand name or type/source of the filler;
- name and location of the sample source;
- the liquid used for the determination and its density  $\rho_l$  (see annex B);
- the date of the determination.

### 10.2 Optional data

The test report can include the following information:

- a description of the material;
- a description of the sampling procedure;
- the weighing data and the densities from the three individual determinations.

## Annex A (normative)

### Calibration of the pyknometer

#### A.1 General

The pyknometer (see 6.2) shall be calibrated by determining its volume.

#### A.2 Procedure

**A.2.1** Clean the pyknometer by thoroughly rinsing it several times with acetone (see 5.3) and dry it.

**A.2.2** Weigh the pyknometer together with the stopper ( $m_0$ ). Carry out both weighings with an accuracy of 0,0001 g.

**A.2.3** Fill the pyknometer with demineralized water (see 5.2).

**A.2.4** Place the pyknometer (without stopper) in the water bath at  $(25,0 \pm 0,1)^\circ\text{C}$ , so that the top protrudes between 2 mm and 3 mm above the water level in the bath.

**A.2.5** After 60 min, place the stopper on the pyknometer, causing an amount of water to come out of the capillary.

**A.2.6** Dry the top of the capillary and remove the pyknometer from the water bath.

**A.2.7** Quickly cool the pyknometer in cold (running) water, to prevent demineralized water expanding out of the capillary due to warm handling.

**A.2.8** Carefully dry the outside and weigh the pyknometer filled with water ( $m_3$ ).

**A.2.9** Calculate the volume of the pyknometer, in millilitres, in accordance with the following equation:

$$V = \frac{m_3 - m_0}{\rho_w}$$

where:

- $V$  is the volume of the pyknometer, in millilitres;
- $m_0$  is the mass of the empty pyknometer with stopper, in grams;
- $m_3$  is the mass of the pyknometer, filled with water, in grams;
- $\rho_w$  is the density of water at  $25^\circ\text{C}$ , in megagrams per cubic metre ( $= 0,99707$ ).

Report the result to the nearest 0,01 ml.

## Annex B (normative)

### Procedure for the determination of the density of the liquid used to determine the particle density of the filler

#### B.1 General

The pyknometer (see 6.2) shall be calibrated by determining its volume.

#### B.2 Procedure

**B.2.1** Clean the pyknometer by thoroughly rinsing it several times with acetone (see 5.3) and dry it.

**B.2.2** Weigh the pyknometer together with the stopper ( $m_0$ ). Carry out both weighings with an accuracy of 0,0001 g.

**B.2.3** Fill the pyknometer with a suitable liquid (see 5.1).

**B.2.4** Place the pyknometer (without stopper) in the liquid bath at  $(25,0 \pm 0,1)^\circ\text{C}$ , so that the top protrudes between 2 mm and 3 mm above the liquid level in the bath.

**B.2.5** After 60 min, place the stopper on the pyknometer, causing an amount of liquid to come out of the capillary.

**B.2.6** Dry the top of the capillary and remove the pyknometer from the liquid bath.

**B.2.7** Quickly cool the pyknometer in cold (running) water, to prevent liquid expanding out of the capillary due to warm handling.

**B.2.8** Carefully dry the outside and weigh the pyknometer filled with liquid ( $m_4$ ).

**B.2.9** Calculate the density of the liquid, in megagrams per cubic metre, in accordance with the following equation:

$$\rho_l = \frac{m_4 - m_0}{V} \text{ Mg/m}^3$$

where:

- $\rho_l$  is the density of liquid at  $25^\circ\text{C}$ , in megagrams per cubic metre;
- $m_0$  is the mass of the empty pyknometer with stopper, in grams;
- $m_4$  is the mass of the pyknometer, filled with liquid, in grams;
- $V$  is the volume of the pyknometer, in millilitres.

Report the result to the nearest 0,001 Mg/m<sup>3</sup>.



**Annex C (informative)****Precision**

Repeatability  $r$  and reproducibility  $R$  have been determined by a proficiency scheme of testing, started in 1978, involving Dutch laboratories working in co-operation with the Netherlands State Road Laboratory, as follows:

Repeatability ( $r$ ) = 0,06 Mg/m<sup>3</sup>

Reproducibility ( $R$ ) = 0,07 Mg/m<sup>3</sup>

NOTE The precision of the test can be affected by the type of filler and the choice of liquid.

**Annex D (informative)****Bibliography**

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