



SLOVENSKI STANDARD

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**Metode za preskušanje gostih oblikovanih ognjevzdržnih izdelkov - 18. del:
Ugotavljanje prostorninske mase podrobljenih materialov z metodo z vodo v
vakuumu**

Methods of test for dense shaped refractory products - Part 18: Determination of bulk density of granular materials by the water method with vacuum

Prüfverfahren für dichte geformte feuerfeste Erzeugnisse - Teil 18: Bestimmung der Rohdichte an körnigem Gut nach dem Wasserverdrängungsverfahren unter Vakuum

Méthodes d'essai pour produits réfractaires façonnés denses - Partie 18: Détermination de la masse volumique apparente des matériaux en grains par la méthode à l'eau sous vide

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81.080 Ognjevzdržni materiali Refractories

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EUROPEAN STANDARD
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English version

**Methods of test for dense shaped refractory products - Part 18:
Determination of bulk density of granular materials by the water
method with vacuum**

Méthodes d'essai pour produits réfractaires façonnés
denses - Partie 18: Détermination de la masse volumique
apparente des matériaux en grains par la méthode à l'eau
sous vide

Prüfverfahren für dichte geformte feuerfeste Erzeugnisse -
Teil 18: Bestimmung der Rohdichte an körnigem Gut nach
dem Wasserverdrängungsverfahren unter Vakuum

This European Standard was approved by CEN on 5 July 2002.

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. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

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



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Foreword

This document (EN 993-18:2002) has been prepared by Technical Committee CEN/TC 187 "Refractory products and materials", 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 February 2003, and conflicting national standards shall be withdrawn at the latest by February 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, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

EN 993 'Methods of test for dense shaped refractory products' consists of 20 Parts:

- Part 1: *Determination of bulk density, apparent porosity and true porosity.*
- Part 2: *Determination of true density.*
- Part 3: *Test methods for carbon-containing refractories.*
- Part 4: *Determination of permeability to gases.*
- Part 5: *Determination of cold crushing strength.*
- Part 6: *Determination of modulus of rupture at ambient temperature.*
- Part 7: *Determination of modulus of rupture at elevated temperatures.*
- Part 8: *Determination of refractoriness-under-load.*
- Part 9: *Determination of creep in compression.*
- Part 10: *Determination of permanent change in dimensions on heating.*
- Part 11: *Determination of resistance to thermal shock (ENV).*
- Part 12: *Determination of pyrometric cone equivalent.*
- Part 13: *Specification for pyrometric cones.*
- Part 14: *Determination of thermal conductivity (hot wire, cross-array).*
- Part 15: *Determination of thermal conductivity (hot wire, parallel).*
- Part 16: *Determination of resistance to acids.*
- Part 17: *Determination of bulk density of granular material (mercury method).*
- Part 18: *Determination of bulk density of granular material (water method).*
- Part 19: *Determination of resistance to thermal expansion.*
- Part 20: *Determination of resistance to abrasion at ambient temperature.*

1 Scope

This European Standard specifies a method based on water absorption with vacuum for the determination of the bulk density of granular refractory materials (grain bulk density) having a grain size greater than 2 mm.

NOTE 1 This method is proposed as an alternative to the method based on mercury displacement with vacuum described in EN 993-17 which is to be used as the reference method because of its reproducibility. However, mercury is known to be a hazardous substance and the method described in this European Standard is recommended for all routine purposes. Nevertheless, depending on the nature of the material tested, the two methods may give different results.

NOTE 2 For materials that are sensitive to contact with water, a suitable organic liquid should be used. The immersion liquid should not fractionate at a pressure above the absolute pressure attained in the test. For example, distilled paraffin can be used for hydratable materials.

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2 Normative references

Not applicable.

3 Terms and definitions

For the purposes of this European Standard, the following terms and definitions apply.

3.1

bulk density of a granular material (grain bulk density)

the ratio of the mass of a quantity of the material to the total volume of its grains, including the volume of any closed pores within the grains

3.2

closed pores

pores that are not penetrated under the conditions of this test when grains are immersed in water

4 Principle

The following are determined by weighing: the mass of a dry quantity of the granular material, then its apparent mass when immersed in water with which it has been impregnated under vacuum, and then its mass in air while still soaked with water.

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5 Apparatus

5.1 Drying oven, capable of being controlled to $110^{\circ}\text{C} \pm 5^{\circ}\text{C}$.

NOTE A fan-assisted oven with ventilation would assist in attaining an even temperature distribution and efficient drying of the test pieces.

5.2 Balance, with an accuracy of $\pm 0,1$ g and a scale graduation of 0,01 g that can be arranged so that the test samples can be suspended in water.

5.3 Small crucibles (or beakers), to contain the granular test samples.

5.4 Beakers, of a suitable size to contain the sample crucibles during soaking (see 7.2) and when determining the apparent immersed mass.

5.5 Evacuating equipment, capable of reducing the absolute pressure to a value not greater than 2 500 Pa and a means of measuring the pressure used (see Figure 2).

5.6 Cotton cloth.

5.7 Distilled or de-ionized water.

5.8 Desiccator.

6 Test samples

6.1 Preparation of test samples

The material to be tested shall consist of fractions or groups of fractions of grains. However, laboratory samples shall be produced from these fractions by sieving and comminution to obtain a grain size between 2 mm and 5,6 mm.

NOTE Any dust or loose particles adhering to the grains should be removed before testing by washing or, with materials sensitive to moisture or humidity, by air blowing.

6.2 Number of test samples

Take at least three test samples from the laboratory sample (see 6.1) and carry out one determination of bulk density on each test sample.

6.3 Mass of test samples

The mass of test samples depends on the grain size and the homogeneity of the material being tested. A mass of approximately 200 g is recommended.

NOTE In the case of homogeneous materials, the mass of the test samples can be reduced to approximately 100 g.

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7 Method

7.1 Determination of the mass of dry test sample (m_1)

Place the test sample into a dry, pre-weighed crucible (see 5.3) and dry the test sample at $110\text{ °C} \pm 5\text{ °C}$ to constant mass, i.e. until two successive weighings made, before and after at least 2 h in the oven, do not differ by more than 0,1 %.

Before each weighing, place the crucible containing the test sample in a desiccator until it has cooled to ambient temperature. Weigh each crucible to the nearest 0,01 g scale reading. Subtract the mass of the empty crucible to obtain the mass of the dry test sample (m_1).

7.2 Impregnation of the test sample

Carry out a check test to ensure that the apparatus will hold a vacuum. Place the cooled and dried crucible into a large beaker contained in an air-tight vessel (see Figure 2). After sealing the vessel, evacuate it until a pressure of not more than 2 500 Pa is attained. Maintain this vacuum for at least 15 min. In order to ensure that all the air has been removed from the open pores, isolate or disconnect the vessel from the vacuum pump and check that pressure does not rise through any de-gassing of the test sample. Re-connect the vessel to the vacuum pump and progressively introduce the water (see 5.7) so that after 3 min, the test sample is covered by about 20 mm. Maintain this reduced pressure for 30 min, then switch off the pump and open the vessel. The test sample or test samples shall remain covered with the water throughout the impregnation and until removed for subsequent weighing (see 7.3).

7.3 Determination of the apparent mass of the immersed test sample (m_5) and the mass of the impregnated test sample (m_3)

Remove the crucible from the air-tight vessel, ensuring that the test sample remains covered with water. Suspend the crucible by a thin thread from the load-pan suspension point of the balance (see 5.2) and weigh it to the nearest 0,01 g scale reading (m_2) while completely immersed in a quantity of water contained in a beaker standing on the bridge, if used (see Figure 1).

Take the crucible out of the water and transfer the test sample, without loss, on to a flat cotton cloth (see 5.6), which has been saturated with water and wrung out by hand. Blot the grains until the wet sheen disappears. Immediately weigh the test sample, in air, to the nearest 0,01 g scale reading. This gives the mass of the impregnated test sample (m_3).

Suspend the empty crucible from the load pan suspension point of the balance and weigh it to the nearest 0,01 g scale reading (m_4). The apparent mass of the immersed test sample is given by:

$$m_5 = m_2 - m_4 \quad (1)$$

where

m_2 = mass of suspended crucible containing test sample;

m_4 = mass of empty suspended crucible.

8 Calculation of results

8.1 Calculation of the volume of the test sample (V_R)

Providing that determinations specified in section 7.3 have been carried out at constant temperature (thus with a constant water density), the volume V_R of the sample, in cubic centimetres, is given by the equation:

$$V_R = \frac{m_3 - m_5}{\rho} \quad (2)$$

where

- m_5 is the apparent mass of the immersed sample, in grams;
- m_3 is the mass of the impregnated sample, in grams;
- ρ is the true density of water at test temperature in grams per cubic centimetre.

If an immersion liquid other than water has been used, determine its true density at the test temperature.

NOTE If water is used, the accuracy of the test is such that its density between 15 °C and 30 °C can be assumed to be 1,0 g/cm³.

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8.2 Calculation of the bulk density of the test sample (ρ_R)

The bulk density ρ_R , in grams per cubic centimetre, is given by the equation:

$$\rho_R = \frac{m_I}{V_R} \quad (3)$$

where

- m_I is the mass, in grams, of the sample (see 7.1);
- V_R is the volume, in cubic centimetres, of the sample (see 8.1).

The result shall be reported to three significant figures.