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EUROPEAN STANDARD

EN 993-1

NORME EUROPÉENNE

EUROPÄISCHE NORM

February 1995

ICS 81.080

Descriptors: Refractory materials, shaped refractories, dense shaped refractory products, determination, bulk density, porosity

English version

**Methods of test for dense shaped refractory products - Part 1: Determination of bulk density, apparent porosity and true porosity**

Méthodes d'essai pour produits réfractaires façonnés denses - Partie 1: Détermination de la masse volumique apparente, de la porosité ouverte et de la porosité totale

Prüfverfahren für dichte geformte feuerfeste Erzeugnisse - Teil 1: Bestimmung der Rohdichte, offenen Porosität und Gesamtporosität

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**CEN**

European Committee for Standardization  
Comité Européen de Normalisation  
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

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Ref. No. EN 993-1:1995 E

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## Foreword

This European Standard has been prepared by the Technical Committee CEN/TC 187 "Refractory products and materials", the secretariat of which is held by BSI.

It is closely based on the corresponding International Standard, ISO 5017 "Dense shaped refractory products - Determination of bulk density, apparent porosity and true porosity", published by the International Organization for Standardization (ISO).

Reproducibility and repeatability data are not available, but may be given in a subsequent edition.

EN 993 'Methods of test for dense shaped refractory products' consists of 18 Parts:

- Part 1 : Determination of bulk density and 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 rupture, ambient temperatures
- Part 7 : Determination of modulus rupture, 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)

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 August 1995, and conflicting national standards shall be withdrawn at the latest by August 1995.

According to the CEN/CENELEC Internal Regulations, the following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, United Kingdom.

## 1 Scope

This Part of EN 993 specifies a method for the determination of the bulk density, apparent porosity and true porosity of dense shaped refractory products.

NOTE: For shaped insulating refractory products, the bulk density and true porosity are determined in accordance with EN 1094-4.

## 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 993-2 : Methods of test for dense shaped refractory products  
Part 2 : Determination of true density.

ISO 758 : Liquid chemical products for industrial use - Determination of density at 20 °C.

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## 3 Definitions

For the purpose of this Part of EN 993, the following definitions apply.

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**3.1 bulk density ( $\rho_b$ ):** The ratio of the mass of the dry material of a porous body to its bulk volume, expressed in grams per cubic centimetre or in kilograms per cubic metre.

**3.2 bulk volume ( $V_b$ ):** The sum of the volumes of the solid material, the open pores and the closed pores in a porous body.

NOTE : The roughness of the surface limits the accuracy of definition of the bulk volume and, in consequence, of the bulk density. Also, the concept of bulk density becomes less precise when the volume of the sample diminishes below certain limits or when its texture (size of pores and grains) is too coarse.

**3.3 true density ( $\rho$ ):** The ratio of the mass of the material of a porous body to its true volume, expressed in grams per cubic centimetre or in kilograms per cubic metre.

**3.4 true volume:** The volume of the solid material in a porous body.

**3.5 open pores:** Those pores that are penetrated by the immersion liquid in the test described.

NOTE : These pores are, in principle, all those that are connected with the atmosphere, either directly or via one another. Here also the roughness of the surface imposes a limit to the accuracy of the definition of the volume of the open pores.

**3.6 closed pores:** Those pores that are not penetrated by the immersion liquid in the test described.

**3.7 apparent porosity ( $\pi_a$ ):** The ratio of the total volume of the open pores in a porous body to its bulk volume, expressed as a percentage of the bulk volume.

**3.8 closed porosity ( $\pi_c$ ):** The ratio of the total volume of the closed pores in a porous body to its bulk volume, expressed as a percentage of the bulk volume.

**3.9 true porosity ( $\pi_t$ ):** The ratio of the total volume of the open and closed pores to the bulk volume of the material, expressed as a percentage.

NOTE : Consequently, the true porosity is the sum of the apparent porosity and the closed porosity.

**3.10 dense shaped refractory product:** A product having a true porosity of less than 45 % (V/V).

#### 4 Principle

**4.1** The following are determined by weighing: the mass of a dry test piece, then its apparent mass when immersed in a liquid with which it has been impregnated under vacuum, and then its mass in air while still soaked with the liquid.

From these values and from the true density of the material, determined by the method specified in EN 993-2, its bulk density, apparent porosity and true porosity are determined by calculation.

**4.2** The precision of the results does not require any correction to be made for the fact that weighings are carried out in air, not in a vacuum.

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#### 5 Apparatus and materials

**5.1** Drying oven, capable of being controlled at  $(110 \pm 5)$  °C.

**5.2** Balance, with an accuracy of  $\pm 0,01$  g.

**5.3** Bridge, to be placed over the load-bearing scale pan of the balance (see 7.3), if a two-pan balance is used.

**5.4** Evacuating equipment, capable of reducing the pressure to a value no greater than 2500 Pa and a means of measuring the pressure used.

**5.5** Thermometer, accurate to  $\pm 1$  °C.

**5.6** Immersion liquid: For materials that do not react with water, the immersion liquid may be cold distilled water. For materials that are sensitive to contact with water, a suitable organic liquid shall be used.

NOTE : For example, distilled paraffin may be used for hydratable materials.

#### 5.7 Desiccator

## 6 Number and shape of test pieces

- 6.1** The number of items (for example, bricks, shapes, nozzles) to be tested shall be determined by agreement between the interested parties.
- 6.2** The number of test pieces to be tested per item shall be agreed between the parties; it shall be stated in the test report. If the test pieces are cut out of bricks or blocks, the same number shall be cut from each one, in order to facilitate statistical analysis.
- 6.3** Test pieces shall be cut in the form of prisms or cylinders. The bulk volume of a test piece shall be not less than 50 cm<sup>3</sup>, and shall be not more than 200 cm<sup>3</sup>. The ratio of the longest to the shortest dimension of a test piece shall not exceed 2 : 1.

NOTE : Where it is not possible to obtain the given size and volume from the item, test pieces of other dimensions and volume may be used by agreements between parties, and are to be reported.

- 6.4** Any test piece showing cracks shall be eliminated, since these might falsify the determination of the bulk volume.

## 7 Procedure

### 7.1 Determination of mass of dry test piece ( $m_1$ )

Dry the test piece at  $(110 \pm 5)$  °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 %.

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Before each weighing, place the test piece in a desiccator until it has cooled to room temperature. Weigh each test piece to the nearest 0,01 g. The mass determined is the mass of the dry test piece ( $m_1$ ).

### 7.2 Soaking of test piece

Carry out a check test to ensure that the apparatus will hold a vacuum. Place the cooled and dried test piece in an air-tight vessel. After sealing the vessel, evacuate it until a pressure of not more than 2500 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 piece. Re-connect the vessel to the vacuum pump and progressively introduce the immersion liquid so that, after 3 min, the test piece is covered by about 20 mm of liquid. Maintain this reduced pressure for 30 min, then switch off the pump and open the vessel. Wait a further 30 min to ensure that the liquid penetrates into all the open pores.

NOTE : Certain fine porosity materials such as refractories containing carbon and some clay products may require longer periods of evaporation.

### 7.3 Determination of apparent mass of immersed test piece ( $m_2$ )

Suspend the test piece by a thin thread from the load-pan suspension point of a hydrostatic balance and weigh it while completely immersed in a quantity of the immersion liquid, contained in a beaker standing on the bridge, if used. In this way, the apparent mass of the immersed test piece



is obtained ( $m_2$ ). The weighing shall be made to the nearest 0,01 g. Determine the temperature of the immersion liquid to an accuracy of  $\pm 1$  °C.

#### 7.4 Determination of mass of soaked test piece ( $m_3$ )

Remove the test piece from the liquid and immediately sponge it quickly and carefully with a damp sponge or cloth to remove droplets and the surface film of liquid but without drawing liquid out of any of the pores.

NOTE: Consistent results have been obtained by keeping for this purpose alone a linen cloth which, having been washed two or three times when new to remove the dressing, is immersed in the immersion liquid and lightly wrung out by hand before each use.

Immediately weigh the test piece in air to the nearest 0,01 g. Take care to ensure that evaporation of the immersion liquid does not lead to any appreciable loss in mass during the weighing operation. In this way, the mass of the soaked test piece is obtained ( $m_3$ ).

#### 7.5 Determination of density of immersion liquid

Determine the density  $\rho(\text{liq})$  of the liquid used in the operation at the temperature of the test. (For water, see table 1.) Refer also to ISO 758.

Table 1: Density of water as a function of temperature between 15 °C and 30 °C.

Temperature (° C)	Density (g/cm <sup>3</sup> )
15	0,9991
16	0,9989
17	0,9988
18	0,9986
19	0,9984
20	0,9982
21	0,9980
22	0,9978
23	0,9975
24	0,9973
25	0,9970
26	0,9968
27	0,9965
28	0,9962
29	0,9959
30	0,9956