

INTERNATIONAL STANDARD

ISO
5017

First edition
1988-06-15



INTERNATIONAL ORGANIZATION FOR STANDARDIZATION
ORGANISATION INTERNATIONALE DE NORMALISATION
МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Dense shaped refractory products — Determination of bulk density, apparent porosity and true porosity

*Produits réfractaires façonnés denses — Détermination de la masse volumique apparente,
de la porosité ouverte et de la porosité totale*

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ISO 5017:1988

<https://standards.iteh.ai/catalog/standards/sist/81dc8ce3-e467-4443-86af-276e6a9dfc28/iso-5017-1988>

Reference number
ISO 5017:1988 (E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 5017 was prepared by Technical Committee ISO/TC 33, *Refractories*.

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Dense shaped refractory products — Determination of bulk density, apparent porosity and true porosity

1 Scope

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

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 758 : 1976, *Liquid chemical products for industrial use — Determination of density at 20 °C*

ISO 5016 : 1986, *Shaped insulating refractory products — Determination of bulk density and true porosity*

ISO 5018 : 1983, *Refractory materials — Determination of true density*

ISO 5022 : 1979, *Shaped refractory products — Sampling and acceptance testing*

3 Definitions

For the purpose of this International Standard, the following definitions apply.

3.1 bulk density, ρ_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.¹⁾

3.3 true density, ρ : The ratio of the mass of the material of a porous body to its true volume.

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 π_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 π_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 π_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 ISO 5018), its bulk density, apparent porosity and true porosity are determined by calculation.

1) The roughness of the surface limits the accuracy of definition of the bulk volume and, in consequence, of the bulk density. Also, the notion 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.

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.

5 Apparatus and materials

5.1 Drying oven, capable of being controlled at 110 ± 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 not greater than 25 mbar, and a **means of measuring the pressure used**.

5.5 Thermometer.

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.

For these materials, the bulk density and true porosity may alternatively be determined by the method specified for shaped insulating refractory products in ISO 5016, preferably carrying out the test on standard bricks.

5.7 Desiccator.

6 Number and shape of test pieces

6.1 The number of items (bricks) to be tested shall be in accordance with ISO 5022 or with another standard sampling plan agreed between the interested parties.

6.2 The number of test pieces to be tested per item (brick) shall be agreed between the parties concerned; it shall be stated in the test report. If several bricks are tested, the same number of test pieces shall be taken from each brick so as to facilitate statistical evaluation.

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 cm³, and not more than 200 cm³. The ratio of the longest to the shortest dimension of a test piece shall not exceed 2 : 1.

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

Dry the test piece at 110 ± 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 %.

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

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 25 mbar 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 use a manometer to 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.

7.3 Determination of apparent mass of immersed test piece

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.

7.4 Determination of mass of soaked test piece

Remove the test piece from the liquid and, without waiting, sponge it quickly and carefully with a damp sponge or cloth¹⁾ to remove droplets and the surface film of liquid but without drawing liquid out of any of the pores.

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 ρ of the liquid used in the operation at the temperature of the test. (For water, see table 1.)

Refer also to ISO 758.

1) 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.

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

Temperature °C	Density ρ , g/cm ³
15	0,999
16	0,999
17	0,999
18	0,999
19	0,998
20	0,998
21	0,998
22	0,998
23	0,998
24	0,997
25	0,997
26	0,997
27	0,997
28	0,996
29	0,996
30	0,996

8 Expression of results

8.1 The bulk density ρ_b , expressed in grams per cubic centimetre, is given by the equation

$$\rho_b = \frac{m_1}{m_3 - m_2} \times \rho_{liq} \quad \dots (1)$$

8.2 The apparent porosity π_a , expressed as a percentage by volume, is given by the equation

$$\pi_a = \frac{m_3 - m_1}{m_3 - m_2} \times 100 \quad \dots (2)$$

8.3 The true porosity π_t , expressed as a percentage by volume, is given by the equation

$$\pi_t = \frac{\rho - \rho_b}{\rho} \times 100 \quad \dots (3)$$

8.4 The closed porosity π_f , expressed as a percentage by volume, is given by the equation

$$\pi_f = \pi_t - \pi_a \quad \dots (4)$$

8.5 In the equations in 8.1 to 8.4:

m_1 is the mass, in grams, of the dry test piece;

m_2 is the apparent mass, in grams, of the immersed test piece;

m_3 is the mass, in grams, of the soaked test piece;

ρ_{liq} is the density, in grams per cubic centimetre, of the immersion liquid;

ρ is the true density, in grams per cubic centimetre, of the product, determined in accordance with ISO 5018.

The bulk density shall be expressed in grams per cubic centimetre or in kilograms per cubic metre [by multiplying the result from equation (1) by 10³]. The value shall be given to three significant digits.

The values of porosity shall be given to the nearest 0,1 % (V/V).

9 Test report

The test report shall include the following information :

- a) the name of the testing establishment;
- b) the date of the test;
- c) a reference to this International Standard, i.e. "determined in accordance with ISO 5017"
- d) the description of the test material (manufacturer, type, batch number, etc.);
- e) the number of test pieces per item (brick);
- f) the number of items tested;
- g) the pressure to which the vacuum chamber was reduced;
- h) the immersion liquid used;
- i) the individual values and the mean value of the bulk density, apparent porosity and true porosity for each item (brick).

NOTE — The individual values are used for determining the mean. The mean is used for further statistical work, for example in accordance with ISO 5022.

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UDC 666.76-128 : [531.755 + 539.217.1]

Descriptors : refractory materials, shaped refractories, tests, physical tests, determination, density (mass/volume), bulk density, porosity.

Price based on 3 pages
