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ISO 8840

Second edition
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Refractory materials — Determination of bulk density of granular materials (grain density)

Matériaux réfractaires — Détermination de la masse volumique apparente des matériaux en grains (masse volumique des grains)

Reference number
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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee 33, *Refractories.*

This second edition cancels and replaces the first edition (ISO 8840:1987), which has been technically revised.

The main changes compared to the previous edition are as follows:

— a new method according to ISO 5017 has been added. It is identical to the method given in EN 993-18 with a suggested improvement to avoid the most influencing factor of the operator.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Refractory materials — Determination of bulk density of granular materials (grain density)

1 Scope

This document specifies three methods for the determination of the bulk density of granular refractory materials (grain density) having a grain size larger than 2 mm:

— Method 1: mercury method with vacuum;

— Method 2: arrested water absorption method;

— Method 3: vacuum method with spin dryer option according to ISO 5017.

Method 1 is intended as the reference method.

NOTE Depending on the nature of the material tested, the three methods can give different results. Any statement of the value of a bulk density can therefore be accompanied by an indication of the method used or to be used in case of dispute.

The same method can be used for the determination of the volume of the sample, for selecting and preparing the sample, for calculating the bulk density and for presenting the test report.

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.

ISO 383, *Laboratory glassware — Interchangeable conical ground joints*

ISO 385-1, *Laboratory glassware — Burettes — Part 1: General requirements*

ISO 5017, *Dense shaped refractory products — Determination of bulk density, apparent porosity and true porosity*

ISO 8656, *Refractory products — Raw materials and unshaped products — Sampling*

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

— IEC Electropedia: available at https://www.electropedia.org/

3.1
grain density
bulk density of granular material
ratio of the mass of a quantity of the material to the total volume of its grains, including the volume of any *closed pores* (3.2) within the grains

3.2
closed pore
pore that is not penetrated by a liquid in which the grains are immersed

4 Principle

Measurement of the volume of a given mass of a granular material by displacement of a liquid.

5 Sampling

Sampling shall be carried out in accordance with ISO 8656 or with another standard sampling scheme agreed between the interested parties.

6 Preparation, number and test size of samples

6.1 Preparation of samples

The material to be tested shall consist of fractions or groups of fractions with grain sizes above 2 mm. Laboratory samples shall be produced by sieving, after any preliminary comminution of the material above 5,6 mm grain size. Test results can be affected by the comminution technique and the equipment used.

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

To improve reproducibility, it is recommended to reduce the distribution of grains from between 2 mm and 5,6 mm to between 3 mm and 4 mm.

6.2 Number of samples

Take at least three test samples from the laboratory sample 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. Recommended sizes are shown in Table 1.

Table 1 — Mass of test samples

Grain fraction mm	Method	Size of test samples g	
		Good homogeneity	Poor homogeneity
2,0 to 5,6	1	100	200
	2	50	50
3,0 to 4,0	3	200	200

7 Determination of mass of test sample

Dry the test sample to constant mass in the drying oven maintained at 110 ± 5 °C and allow it to cool to ambient temperature in the desiccator. Weigh the test sample to the nearest 0,1 g and a scale graduation of 0,01 g.

8 Determination of volume of test sample — Method 1: Mercury method with vacuum

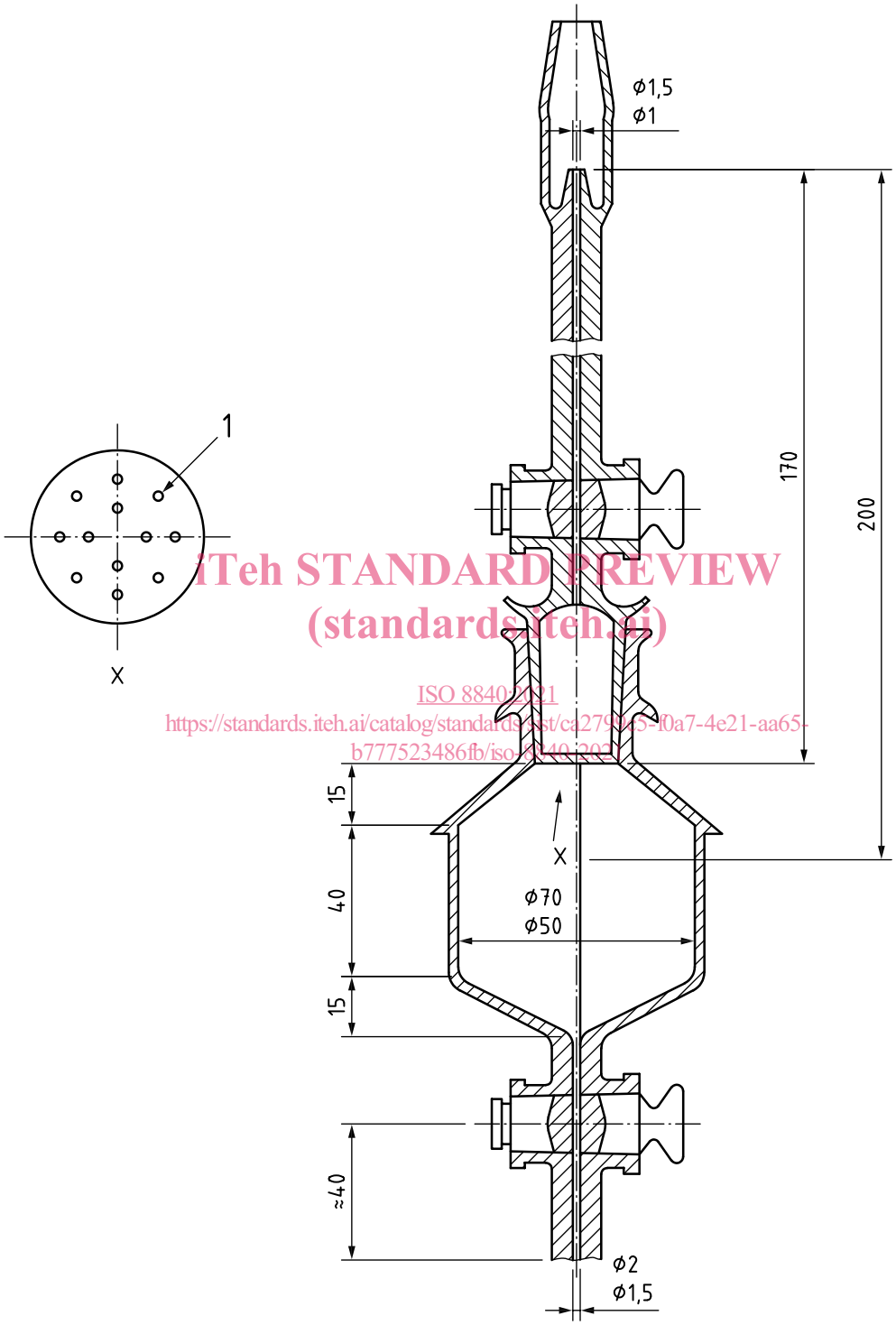
8.1 Principle

Determination of the volume of the test sample by the mercury displacement method with a vacuum below 3 000 Pa residual pressure, preferably with a residual pressure of 133 Pa.

Method 1 is preferred as a reference method because of its reproducibility and simplicity in use. However, mercury is known to be a hazardous substance, and therefore method 2 (see Clause 9) is recommended for all routine purposes.

8.2 Apparatus

8.2.1 Vacuum pycnometer, a vessel as shown in Figure 1 (incorporating conical ground glass joints in accordance with ISO 383).



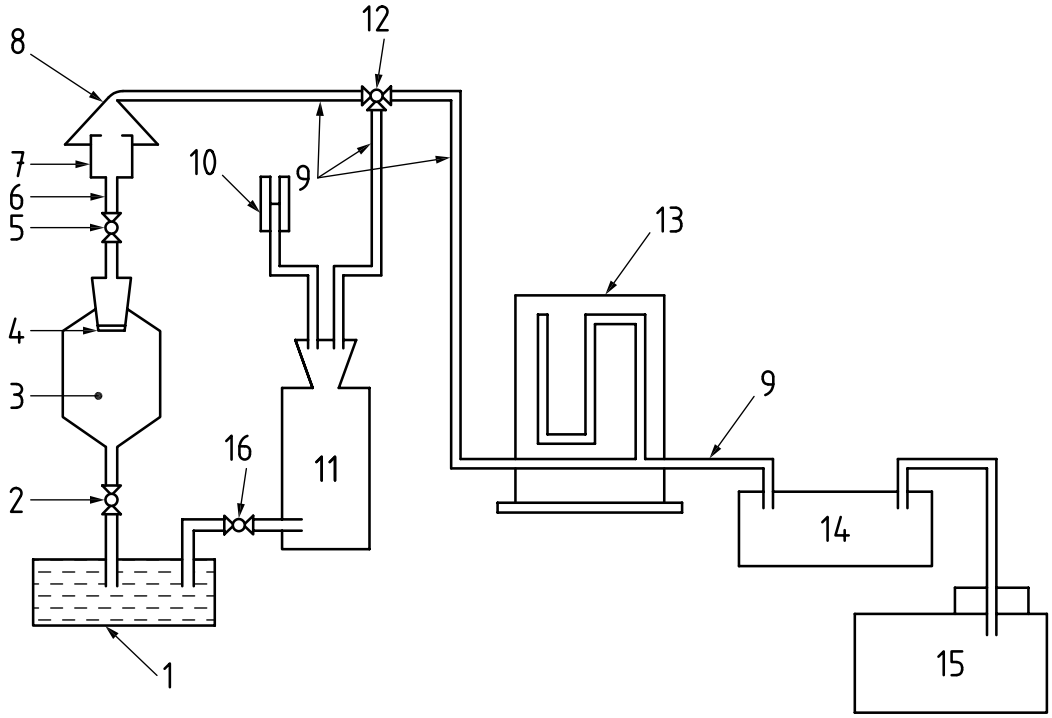
Key

X view of X (enlarged)

1 holes, Ø ≤ 1 mm

Figure 1 — Vacuum pycnometer

8.2.2 Test arrangement, as shown in Figure 2.



Key

1 dish for mercury
2 stopcock 2
3 pycnometer lower part
4 glass seal with holes (diameter ≤ 1 mm)
5 stopcock 5
6 pycnometer upper part
7 capillary and overflow tube
8 pycnometer vacuum connection
9 vacuum tubing
10 mercury extraction bush
11 mercury reservoir
12 three-way tap
13 vacuum manometer
14 Woulfe's bottle
15 vacuum pump
16 stopcock

Figure 2 — Schematic representation of the test arrangement

8.2.3 Balance, with an accuracy of ±0,1 g and a scale graduation of 0,01 g.

8.3 Determination of mass of empty vacuum pycnometer

Clean and dry the empty vacuum pycnometer (8.2.1) and weigh it to the nearest 0,1 g.

NOTE The weighing is unnecessary if all the determinations are carried out at the same temperature.

8.4 Determination of mass of pycnometer filled with mercury

Evacuate the vacuum pycnometer and fill it with mercury by suction until mercury emerges from the capillary tube (see Figure 2). Close the stopcocks 2 and 5 of the pycnometer in that order and disconnect the apparatus from the vacuum pump. Pour off the surplus mercury that has come out of the capillary tube and remove the mercury remaining in the suction tube, up to stopcock 2, with a steel wire. Weigh the pycnometer filled with mercury to the nearest 0,1 g.

8.5 Determination of mass of pycnometer containing test sample and filled with mercury

Transfer the dried and weighed test sample (see Clause 7), without loss, into the pycnometer and fill the pycnometer, under vacuum, with mercury as specified in 8.4. This gives an average pressure on the grains of about 26 500 Pa. Weigh the pycnometer and contents to the nearest 0,1 g. Thereupon, under vacuum, remove the mercury from the vessel. Determine the amount of mercury still remaining in the test sample by weighing the sample after the mercury adhering to it has been removed and by finding the difference from the original mass of the test sample. If the mass of mercury remaining in the test sample is over 5 % of the original mass of the sample, state the amount, expressed as a percentage by mass, in the test report.

8.6 Calculation of volume of test sample

If the weighings in accordance with 8.4 and 8.5 were made at a constant temperature (and therefore with a constant mercury density), the volume V_R of the test sample is given, in millilitres, by Formula (1):

$$V_R = \frac{m_G + m_p - m_T}{\rho} \tag{1}$$

If the weighings in accordance with 8.4 and 8.5 were made at different temperatures (and thus with different mercury densities), the volume V_R of the test sample is given, in millilitres, by Formula (2):

$$V_R = \frac{m_G - m_L}{\rho_1} - \frac{m_T - m_L - m_p}{\rho_2} \tag{2}$$

where

m_G is the mass, in grams, of the pycnometer filled with mercury only;

m_T is the mass, in grams, of the evacuated pycnometer filled with test sample and mercury;

m_L is the mass, in grams, of the empty pycnometer;

m_p is the mass, in grams, of the test sample;

ρ is the temperature corrected density, in grams per cubic centimetre, of mercury (Table 2) if calibration and measurement were carried out at the same temperature;

ρ_1 is the density, in grams per cubic centimetre, of mercury when determining the filled mass of the pycnometer containing mercury;

ρ_2 is the density, in grams per cubic centimetre, of mercury when determining the filled mass of the pycnometer containing both test sample and mercury.

Table 2 — Density of mercury as a function of temperature

Temperature °C	Density g/cm³	Temperature °C	Density g/cm³
15	13,559	23	13,539
16	13,556	24	13,536
17	13,554	25	13,534
18	13,551	26	13,532
19	13,549	27	13,529
20	13,546	28	13,527
21	13,544	29	13,524
22	13,541	30	13,522

9 Determination of volume of test sample — Method 2: Arrested water absorption method

NOTE For materials that react with water, method 1 or 3 with a suitable organic liquid is used.

9.1 Apparatus

9.1.1 Beaker, of capacity 150 ml.

9.1.2 Funnel, with an upper diameter of approximately 100 mm.

9.1.3 Calibrated burette, 100 ml, graduated in 0,2 ml, in accordance with the requirements of ISO 385-1.

9.1.4 Flat-weave cotton cloth

9.1.5 Burette magnifier

9.1.6 Balance, with an accuracy of ± 0,1 g and a scale graduation of 0,01 g

9.2 Determination of volume of test sample

Transfer the dried and weighed test sample (see Clauses 6 and 7) to the beaker (9.1.1) and add water at ambient temperature until the sample is covered. Free the burette from grease and wash it out immediately before each use. Fill it with water to a level between the 20 and the 25 ml marks and allow it to drain for 1 min, then take the initial reading, by estimation, to one-tenth of a division (0,05 ml) with the aid of the magnifier. Then attach the funnel (9.1.2) to the burette (9.1.3) by means of a piece of plastic tubing.

Saturate the cotton cloth (9.1.4) with water and wring it out by hand as dry as possible before each determination. Fold the towel to form a pad with four to six thicknesses of cloth.

When the test sample has been soaking for at least 2 min, place a cover glass over the beaker to retain the sample and pour the water off as completely as possible. Transfer the test sample to the towel and blot it with the towel until the wet sheen has disappeared on the grains. Pour the test sample through the funnel into the burette, folding the towel to facilitate this operation. Read the final level, by estimation, to 0,05 ml, with the aid of the magnifier (9.1.5).

The volume of the test sample is the difference between the final and initial readings.