
**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)*

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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.

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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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

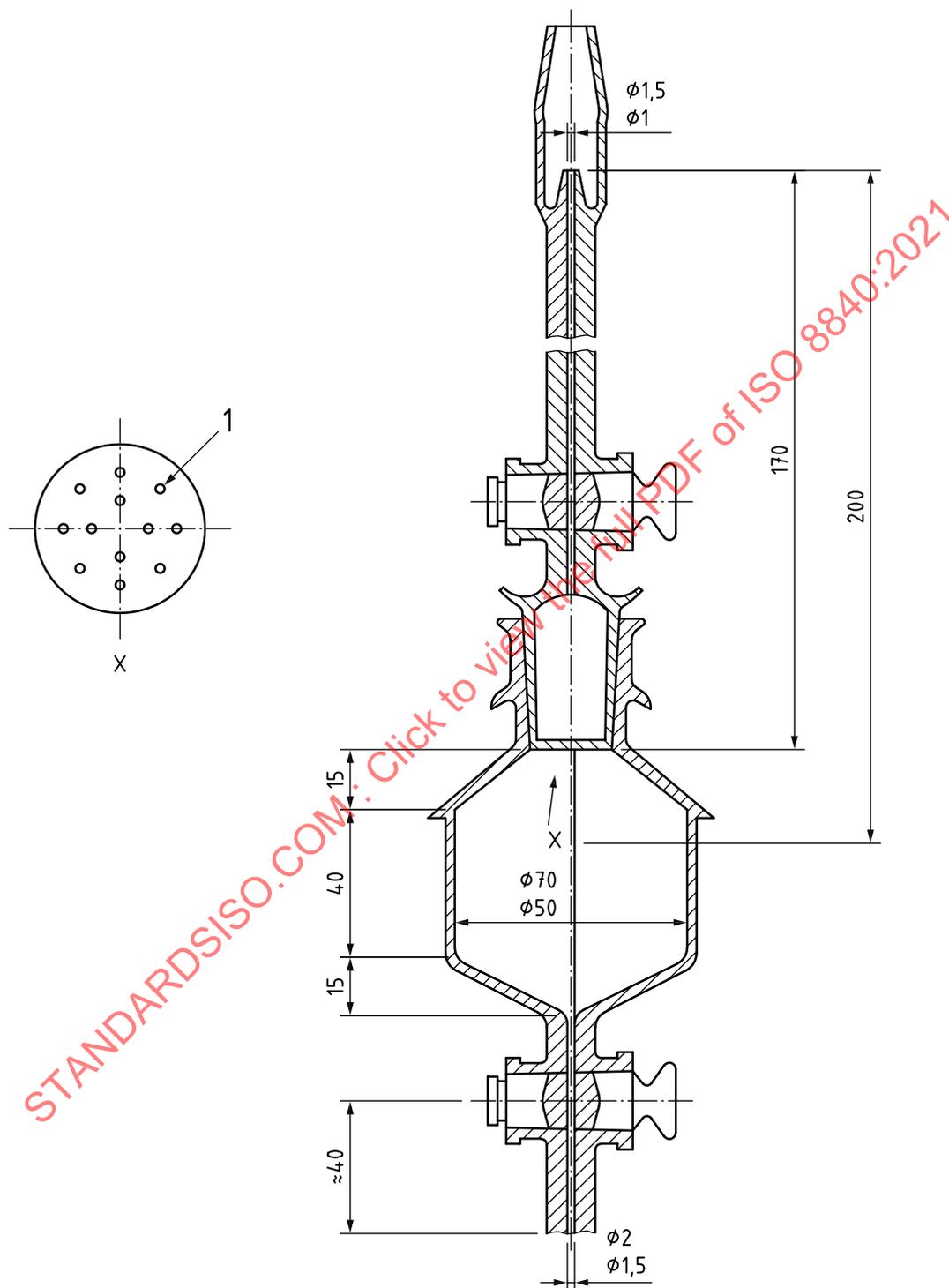
Determination of the volume of the test sample by the mercury displacement method with a vacuum below 3 000 Pa residual pressure, preferably with a residual pressure of 133 Pa.

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.

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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).



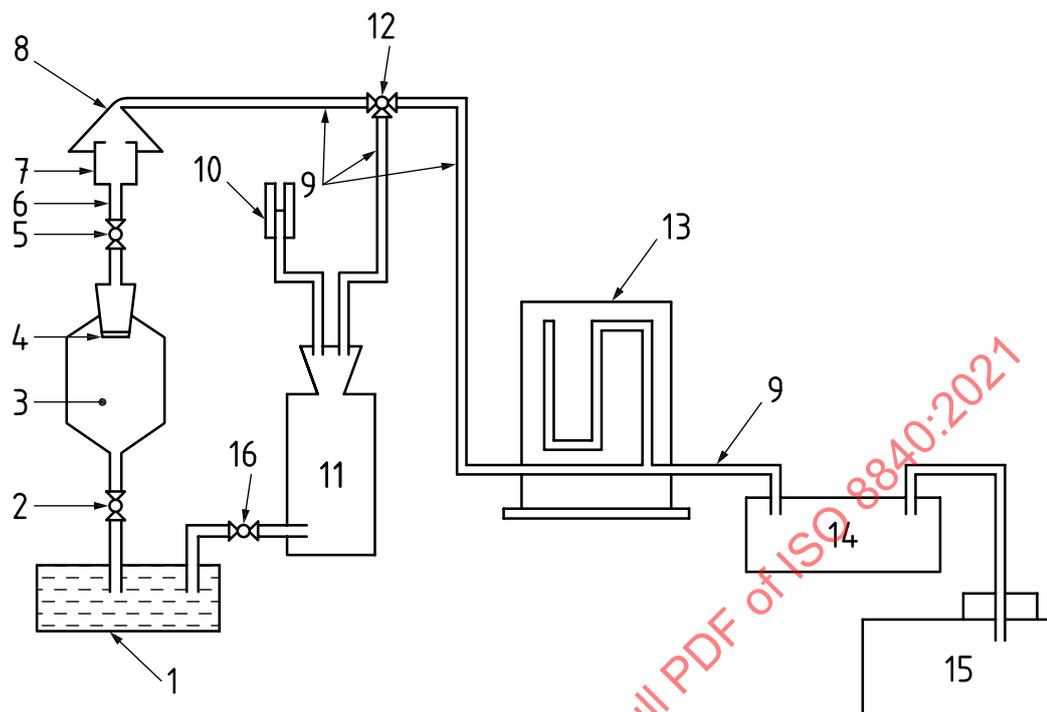
Key

X view of X (enlarged)

1 holes, $\phi \leq 1$ 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 $\pm 0,1$ 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 \(1\)](#):

$$V_R = \frac{m_G + m_p - m_T}{\rho} \quad (1)$$

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 \(2\)](#):

$$V_R = \frac{m_G - m_L}{\rho_1} - \frac{m_T - m_L - m_p}{\rho_2} \quad (2)$$

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/cm ³	Temperature °C	Density g/cm ³
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 $\pm 0,1$ 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.

9.3 Calculation of results

The bulk density ρ_R is given, in grams per cubic centimetre, by [Formula \(3\)](#):

$$\rho_R = \frac{m}{V_R} \tag{3}$$

where

m is the mass, in grams, of the dried test sample (see [Clause 7](#));

V_R is the volume, in millilitres, of the test sample (see [Clause 8](#) or [9.2](#)).

10 Determination of bulk density of test sample — Method 3: Vacuum method with spin dryer option

10.1 Principle

Vacuum method with spin dryer option shall be in accordance with ISO 5017.

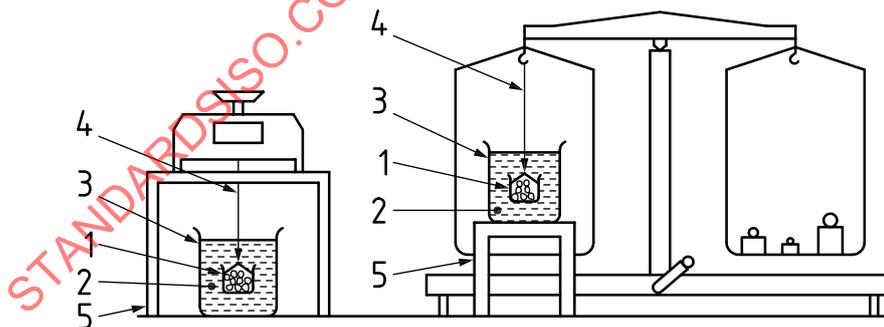
The bulk density is determined by weighing: the mass of a dry quantity of the granular material, then its apparent mass when immersed in a liquid with which it has been impregnated under vacuum, and then its mass in air while still soaked with liquid.

10.2 Apparatus and materials

10.2.1 Drying oven, capable of being controlled at $110\text{ °C} \pm 10\text{ °C}$.

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

10.2.2 Balance, with an accuracy of $\pm 0,1\text{ g}$ and a scale graduation of $0,01\text{ g}$, that can be arranged so that test pieces can be suspended in the immersion liquid (see [Figure 3](#)).



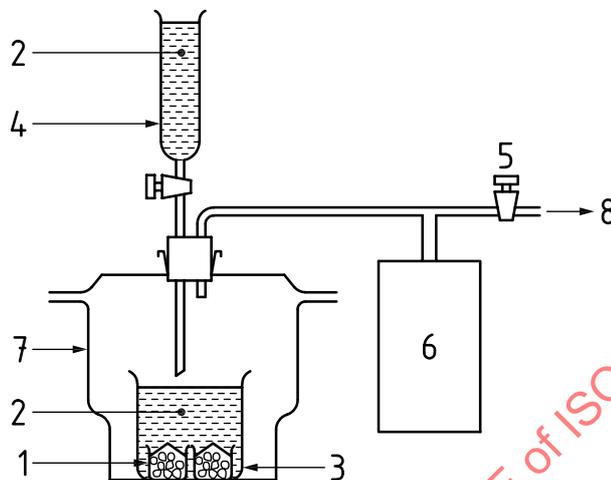
Key

- 1 crucible containing test sample
- 2 liquid
- 3 beaker
- 4 suspension thread
- 5 bridge

Figure 3 — Arrangement for the determination of apparent mass of immersed test sample using single- and double-pan balance

10.2.3 Beakers, of a suitable size for containing the samples during soaking (see 10.3.2) and when determining the apparent immersed mass (see 10.3.3).

10.2.4 Evacuating equipment, capable of reducing the absolute pressure to a value no greater than 2 500 Pa and a means of measuring the pressure used (see Figure 4).



Key

- | | | | |
|---|---------------------------------|---|--|
| 1 | crucible containing test sample | 5 | pump isolation valve |
| 2 | liquid | 6 | pressure measuring device (e.g. digital manometer) |
| 3 | beaker | 7 | desiccator |
| 4 | tap funnel | 8 | to vacuum pump |

Figure 4 — Example of a vacuum system for impregnating test samples

10.2.5 Thermometer, accurate to ± 1 °C.

10.2.6 Immersion liquid. For materials that do not react with water, the immersion liquid shall be cold distilled water or deionized water. For materials that are sensitive to contact with water, a suitable organic liquid shall be used. The immersion liquid shall not fractionate at a pressure above the absolute pressure attained in the test. The liquid shall be exchanged when staining or haze appears.

NOTE Distilled paraffin is normally a suitable immersion liquid for hydratable materials.

10.2.7 Desiccator.

10.2.8 Spin dryer, capable for one or more samples with a circumferential speed of 14 ± 1 m/s and a timer (see Figure 5).

10.3 Procedure

10.3.1 Determination of the mass of dry test piece (m_1)

Dry the test piece 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 (10.2.1) do not differ by more than 0,1 %.

Before each weighing, place the crucible containing the test sample in a desiccator (10.2.7) until it has cooled to room 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).

10.3.2 Soaking of the test sample

Verify that the evacuating equipment (10.2.4) is capable to achieve a vacuum of less than 2 500 Pa. Place the cooled and dried crucible containing the test sample into a large beaker containing an air-tight vessel (see Figure 4). 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 (10.2.4) and check that the pressure does not rise through any de-gassing of the test sample. Re-connect the vessel to the vacuum pump and progressively introduce the immersion liquid (10.2.6) 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. The test sample or test samples shall remain covered by the immersion liquid throughout the impregnation and until removed for subsequent weighing (see 10.3.3).

10.3.3 Determination of the apparent mass of the immersed test sample (m_5) and the mass of the soaked test sample (m_3)

Remove the crucible from the air-tight vessel, ensuring that the test sample remains covered with liquid. Suspend the crucible by a thin thread from the load-pan suspension point of a balance (see 10.2.2) and weigh it to the nearest 0,01 g scale reading (m_2) while completely immersed in a quantity of liquid contained in a beaker standing on the bridge, if used.

Take the crucible out of the liquid and transfer the test sample, without loss, on to a flat cotton cloth, which has been saturated with liquid 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 (m_5) is given by Formula (4):

$$m_5 = m_2 - m_4 \quad (4)$$

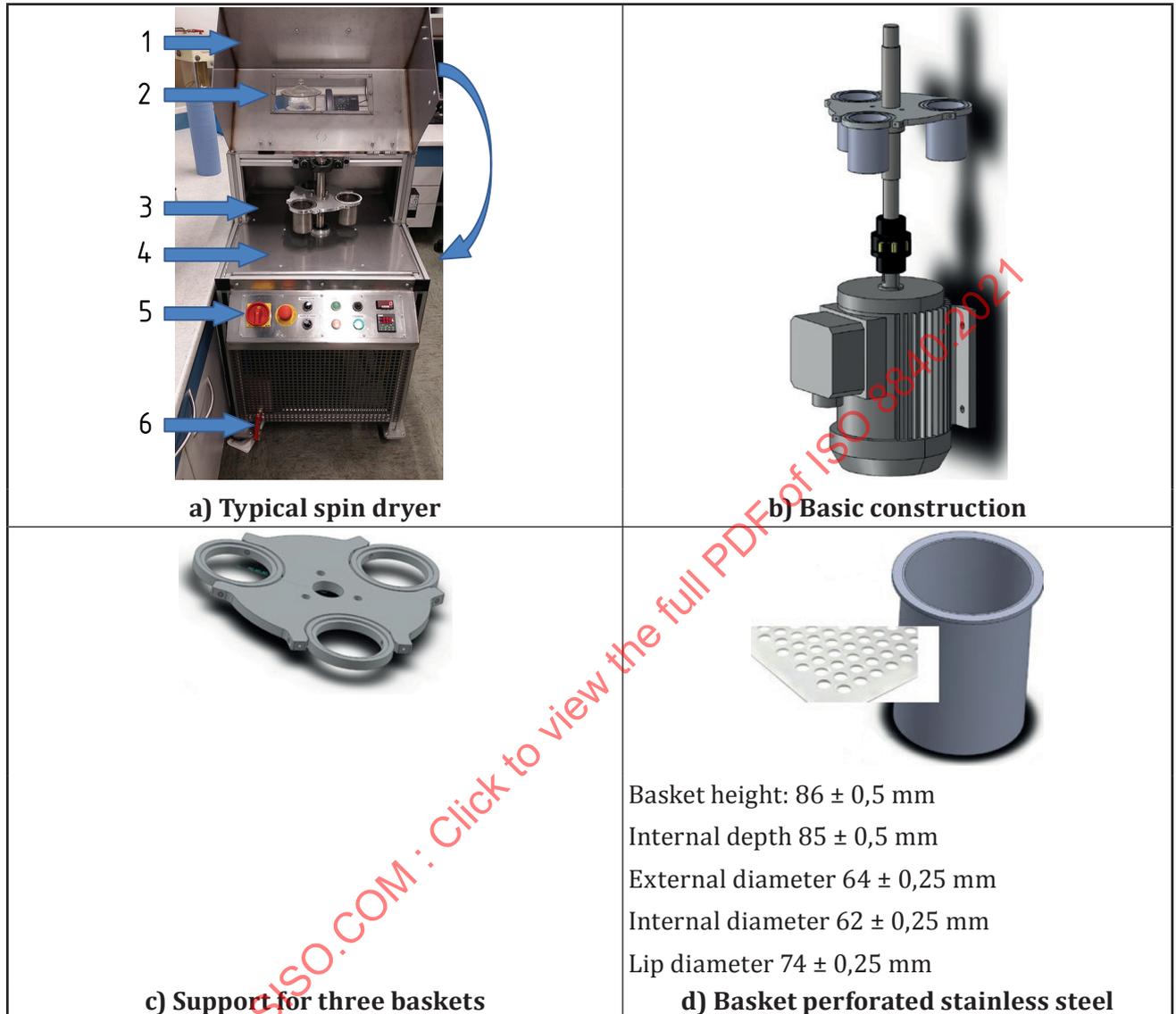
where

m_2 is the mass of suspended crucible containing test sample;

m_4 is the mass of empty suspended crucible.

As alternative method to remove adhered liquid from the grain surface a spin dryer (10.2.8 and Figure 5) can be used.

Mount the crucibles in the spin dryer and let it rotate with a circumferential speed of (14 ± 1) m/s for (210 ± 1) s.



Key

- 1 opening/closing flap
- 2 window to follow operation
- 3 three baskets for samples (200 g)
- 4 stainless steel
- 5 all control elements clearly arranged on front panel
- 6 drain cock

Figure 5 — Example of a spin dryer

10.4 Calculation of results

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

Providing that determinations specified in [10.3.3](#) have been carried out at constant temperature (thus with a constant liquid density) the volume V_R of the sample, in cubic centimetres, is given by the [Formula \(5\)](#):

$$V_R = \frac{m_3 - m_5}{\rho} \quad (5)$$

where

m_5 is the apparent mass of the immersed sample, in grams;

m_3 is the mass of the immersed sample, in grams;

ρ is the true density of the liquid at test temperature in grams per cubic centimetre.

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.

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

The bulk density ρ_R in grams per cubic centimetre, is given by [Formula \(6\)](#):

$$\rho_R = \frac{m_1}{V_R} \quad (6)$$

where

m_1 is the mass of the sample, in grams (see [10.3.1](#));

V_R is the volume of the sample, in cubic centimetres (see [10.4.1](#)).

The result shall be the mean value of three test samples, be given to 3 significant digits, or to 4 significant digits for interlaboratory studies.

10.5 Precision and bias

10.5.1 Interlaboratory data

An interlaboratory study was conducted in 2020 in which 10 different sample types (magnesia, fused magnesia, dolomite, bauxite, chamotte, corundum, tabular alumina and chrome ore) was split and tested in eight laboratories. One to three operators in each laboratory tested the material three times each.

10.5.2 Precision

Precision and relative precision data at the 95 % confidence level according to calculation of ISO 5725-2 are given in [Table 3](#).