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Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of absolute density of ceramic powders by pyknometer

Céramiques techniques — Détermination de la masse volumique absolue des poudres céramiques à l'aide d'un pycnomètre

Reference number
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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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Foreword

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ISO 18753 was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of absolute density of ceramic powders by pyknometer

1 Scope

This International Standard specifies a method for determining the particle density of fine ceramic powders using liquid pyknometry.

2 Normative references

The following referenced documents are indispensable for the application 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 758, *Liquid chemical products for industrial use — Determination of density at 20 °C*

ISO 3507, *Laboratory glassware — Pyknometers*

ISO 6353-2, *Reagents for chemical analysis — Part 2: Specifications — First series*

ISO 6353-3, *Reagents for chemical analysis — Part 3: Specifications — Second series*

ISO 8213, *Chemical products for industrial use — Sampling techniques — Solid chemical products in the form of particles varying from powders to coarse lumps*

3 Terms and definitions

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

3.1
particle density
density of an individual particle of powder

NOTE When an enclosed space occurs inside the particle, the space is considered to be part of the individual particle.

3.2
pyknometry
method of measuring particle density using a pyknometer

4 Preparation of measurement

4.1 Sampling

A representative sample for analysis shall be taken in accordance with ISO 8213.

4.2 Drying of sample

The selected sample is thoroughly dried in an air bath at approximately 110 °C, and then cooled in a desiccator to room temperature. In the case of powders requiring a long drying time, the sample is thoroughly spread, and stirred lightly once or twice midway through the drying process.

NOTE 1 When the material is not stable at the heating temperature, the sample should be dried under reduced pressure instead of heating.

NOTE 2 If necessary, it is recommended to record the mass loss as a function of time to ensure that the sample has reached equilibrium.

4.3 Preparation of immersion liquid

The immersion liquid used for pyknometry shall not be reactive and shall not dissolve the sample. A liquid with good wetting properties and a low evaporation rate under vacuum conditions shall be selected.

The standard liquid for immersion shall be xylene, as specified in ISO 6353-3. When this liquid is not suitable, due to reaction or dissolution of the powders, distilled water, ethanol (e.g. 95 % purity by volume fraction) as specified in ISO 6353-2, or 1-butanol as specified in ISO 6353-3 may be used.

The absolute density of the immersion liquid at the measured temperature shall be determined using pyknometry, in accordance with ISO 758. Ethanol purity is not important. Content of ethanol is given as an example.

5 Apparatus

5.1 Pyknometer, Gay-Lussac type made of glass, of capacity 25 ml or 50 ml (see Figure 1), as specified in ISO 3507, or other suitable type of pyknometer.

5.2 Vacuum container, having an integrated pyknometer with a structure wherein internal conditions of the pyknometer can be observed. The pyknometer shall be connected to a vacuum pump, which is used to create the vacuum conditions.

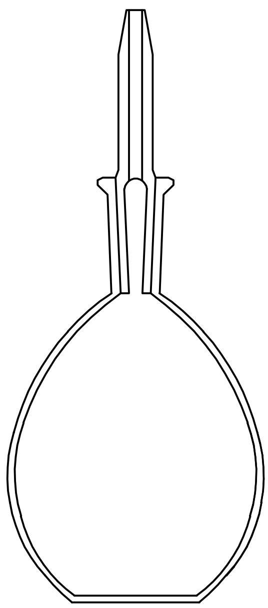

Figure 1 — Example of Gay-Lussac type pyknometer

5.3 Vacuum pump, capable of obtaining a vacuum of 666,5 Pa (5 mmHg) or better.

5.4 Vacuum gauge, capable of measuring from 0 kPa to 26,66 kPa (0 mmHg to 200 mmHg).

5.5 Balance, with a reciprocal sensitivity of 0,1 mg.

5.6 Thermometer, capable of reading to $\pm$ 0,1 °C.

6 Procedure

Measurements shall be carried out according to the following procedure. All masses shall be measured to the nearest 0,1 mg.

a) Clean the pyknometer (5.1), thoroughly dry it, then measure the mass, m_{p1}.

b) Transfer the powder sample to the pyknometer, filling the pyknometer to approximately one-third of its capacity, and measure the mass, m_{p2}.

c) Gently fill the pyknometer with immersion liquid until the powder sample is covered. It is permitted to slightly exceed this level. When the immersion liquid is dispensed, care should be taken not to scatter the sample.

d) Install the pyknometer containing the powder sample, immersed in liquid, in a vacuum container (5.2), reduce pressure to 13,33 kPa (100 mmHg) or less, and implement the degassing procedure. Allow a reduction of pressure to a level at which the immersion liquid cannot boil, and carry out the degassing until bubbles cannot be generated from the sample.

e) Remove the pyknometer from the vacuum container and allow it to stand until the temperature of the liquid falls to room temperature. After thermal equilibrium is reached, record the temperature. The temperature of the sample/container shall be recorded and thermal equilibrium shall be reached before the mass is recorded.

f) Add the specific amount of immersion liquid to the pyknometer, and measure the mass, m_{p3}.

g) Remove the sample and the immersion liquid from the pyknometer. After washing and drying the pyknometer, fill with fresh immersion liquid to the specified level, and measure the mass, m_{p4}.

h) Determine the absolute density of the immersion liquid at the measured temperature using pyknometry, in accordance with ISO 758, and determine the absolute density of the immersion liquid to four places of decimals. Where distilled water is used as the immersion liquid, the absolute density for a given temperature, listed in Table A.1, shall be used. The permissible temperature difference for measurements taken in steps f) to h) shall be within $\pm$ 1 °C.

7 Calculation

Calculate particle density ρ by means of Equation (1) by using the values obtained from Clause 6. Four significant digits shall be obtained.

$$\rho = \frac{(m_{p2} - m_{p1})}{(m_{p4} - m_{p1}) - (m_{p3} - m_{p2})}\,\rho_L \qquad (1)$$

where

ρ is the particle density in grams per cubic centimetre;

m_{p2} is the mass of the sample and the measuring container in grams;

m_{p1} is the mass of the measuring container in grams;

m_{p4} is the mass of the specific amount of immersion liquid and the measuring container in grams;

m_{p3} is the mass of the specific amount of sample, the immersion liquid and the measuring container in grams;

ρ_L is the density of the immersion liquid at measuring temperature in grams per cubic centimetre.

8 Tests in duplicate

Carry out tests in duplicate on representative analysis samples. If the difference between the original and duplicate results is greater than 0,03 g/cm^3, repeat the procedure.

9 Test report

The test report shall contain the following information:

a) date of the test;

b) name of the testing establishment;

c) reference to this International Standard, i.e. ISO 18753;

d) description of the test material (manufacturer, type, batch or code number);

e) description of the sample pre-treatments (evacuation time and evacuator, heating temperature and period);

f) pyknometer (type, capacity);

g) description of the immersion liquid used and temperature of measurements;

h) absolute density of the sample;

i) comments concerning the test or test results.

Annex A
(normative)

The reference data for absolute density of distilled water

Table A.1 indicates the absolute density of distilled water as a function of temperature between 15 °C and 30 °C [1].

Table A.1 — Absolute density of distilled water as a function of temperature between 15 °C and 30 °C

Temperature °C	**Density** g/cm^3
15	0,999 1
16	0,999 0
17	0,998 8
18	0,998 6
19	0,998 4
20	0,998 2
21	0,998 0
22	0,997 8
23	0,997 6
24	0,997 3
25	0,997 1
26	0,996 8
27	0,996 5
28	0,996 3
29	0,996 0
30	0,995 7

Bibliography

[1] LANDOLT-BÖRNSTEIN, *Zahlenwerte und Funktionen aus Physik, Chemie, Astronomie, Geophysik und Techni*k, 6th Edition, **II**, Part 1, p. 36 (1971); **IV**, Part 1, p. 101 (1955), Springer-Verlag

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