SLOVENSKI STANDARD
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Advanced technical ceramics - Methods of test for ceramic powders - Part 7: Determination of the absolute density

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Hochleistungskeramik - Prüfverfahren für keramische Pulver - Teil 7: Bestimmung der absoluten Dichte

Céramiques techniques avancées - Méthodes d'essai pour les poudres céramiques - Partie 7: Détermination de la masse volumique absolue

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CEN

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

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Contents

Foreword

This European Standard has been prepared by the Technical Committee CEN/TC184 'Advanced Technical Ceramics',of which the Secretariat is held by BSI.

The method given is closely based on ISO 901 : 1976 'Aluminium oxide primarily used for the production of aluminium - Determination of absolute density - Pyknometer method'.

EN 725 consists of 11 Parts

Part 1 :	Determination of impurities in alumina
Part 2 :	Determination of impurities in barium titanate (ENV)
Part 3 :	Determination of oxygen content of non-oxides by thermal extraction with a carrier gas
Part 4 :	Determination of oxygen content of aluminium nitride by XRF (ENV)
Part 5 :	Determination particle size distribution
Part 6 :	Determination of the specific surface area
Part 7 :	Determination of absolute density
Part 8 :	Determination of tapped density
Part 9 :	Determination of untamped density
Part 10 :	Determination of compaction properties
Part 11 :	Determination of the densification on natural sintering (ENV)

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of an identical text or by endorsement, at the latest by May 1996, and conflicting national standards shall be withdrawn at the latest by May 1996.

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 and the United Kingdom.

1 Scope

This standard specifies a method for the determination of the absolute density, by liquid pyknometry, of powders for use as raw materials in the preparation of advanced technical ceramics.

2 Normative references

This European Standard incorporates by dated or undated references, 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.

ISO 802 : 1976 Aluminium oxide primarily used for the production of aluminium - Preparation and storage of test samples

3 Principle

The absolute density of the powder is determined by a pyknometer method, after degasification.

4 Reagents

4.1 Reagents (see 4.2 to 4.4) of recognized analytical grade and distilled (or de-ionized) water shall be used.

4.2 Xylene, of density 0,860 to 0,865 g/ml, distilling between 138 °C and 144 °C.

4.3 Ethanol, 95 % or 96 % solution (V/V), density approximately 0,81 g/ml.

4.4 Diethyl ether, density approximately 0,715 g/ml.

5 Apparatus

5.1 Pyknometer (see figure 1), consisting of

a) Flask (A), of capacity approximately 25 ml fitted with a side arm with a ground glass cover (F).

b) Thermometer (B), covering the range 15 °C to 25 °C, graduated in intervals of 0,1 °C, which can be fitted to the ground socket (G) of the flask.

5.2 Degassing and filling apparatus (see figure 2), consisting of a tap funnel (C) of capacity about 50 ml, fitted with a side arm, with a tap (D) for connecting to the vacuum pump (see 5.4). This can be fitted to the pyknometer flask (A) by means of a conical ground glass joint (E), into the socket (G).

5.3 Water bath, capable of being controlled to ± 0,1 K at temperatures between 15 °C and 25 °C.

5.4 Vacuum pump, capable of giving a vacuum below 1,33 kPa (10 mm Hg).

5.5 Vacuum gauge, such as a mercury manometer.

5.6 Balance, weighing to the nearest 0,0001 g.

6 Procedure

6.1 General

6.1.1 Weigh the pyknometer with the thermometer and the side tube cover in position, to the nearest 0,000 1 g.

6.1.2 When the pyknometer contains liquid, stabilize its temperature and record the test temperature to an accuracy of ± 0,1 K.

6.2 Determination

6.2.1 Clean the pyknometer (see 5.1), including its accessories, taking all necessary precautions. Thoroughly rinse, first with tap water, then with distilled water and ethanol (see 4.3) and finally with diethyl ether (see 4.4).

Thoroughly dry the apparatus and weigh (m_o).

6.2.2 Fill the pyknometer (see 5.1) with distilled water and connect via the degassing apparatus (see 5.2) to the vacuum pump (see 5.4), the vacuum gauge (see 5.5) being inserted to control the vacuum. Close the side tube of the pyknometer with its cover (F) and slowly open tap (D) and apply the vacuum for approximately 15 min. Occasionally tap the walls of the pyknometer to facilitate the release of any air bubbles. Restore atmospheric pressure in the pyknometer, disconnect it from the degassing apparatus (see 5.2) and put the thermometer (B) in position.

Stabilize the temperature of the pyknometer in the water bath (see 5.3) and record the temperature to an accuracy of ± 0.1 K. Completely fill the side tube with water, using a length of narrow glass tubing. Remove the pyknometer from the water bath, cool slightly under running cold water and close the side tube with its ground glass cover (F). Carefully dry the pyknometer and weigh.

The volume, in ml, of the pyknometer, is given by the formula

$$V = \frac{m_1 - m_o}{\rho} \quad \ldots (1)$$

where

m_o is the mass, in g, of the dry pyknometer;

m_1 is the mass, in g, of the pyknometer filled with degassed distilled water;

ρ is the absolute density of water, in g/ml, at the test temperature (see table 1)

Table 1 : Absolute density of water as a function of temperature between 15 °C and 25 °C

Temperature (°C)	Density (g/cm^3)
15	0,999099
16	0,998943
17	0,998774
18	0,998595
19	0,998405
20	0,998203
21	0,997992
22	0,997770
23	0,997538
24	0,997296
25	0,997044

6.2.3 Weigh the pyknometer (see 5.1) as in 6.2.1 and fill with xylene (see 4.2). Carry out the procedure specified in 6.2.2, i.e. degas, stabilize the temperature and record the test temperature to an accuracy of ± 0.1 K.

The mass m_x, in g, of xylene equivalent to the volume V of the pyknometer is given by the formula

$$m_x = m_2 - m_o \quad \ldots (2)$$

where

m_o is the mass, in g, of the dry pyknometer;

m_2 is the mass, in g, of the degassed pyknometer full of xylene.

The absolute density ρ_x, in g/ml, of xylene is given by the formula

$$\rho_x = \frac{m_x}{V} \quad \ldots (3)$$

where

m_x is the mass, in g, of xylene equivalent to the volume V of the pyknometer;

V is the volume, in ml, of the pyknometer.

6.2.4 Transfer to the flask of the pyknometer (see 5.1), about 10 g of the test sample, dried at a temperature less than the decomposition temperature of the ceramic powder (see 3.3 of ISO 802 : 1976) and weigh. The mass m_e in g, of the test portion is given by the formula

$$m_e = m_3 - m_o \quad \ldots (4)$$

where

m_o is the mass, in g, of the dry pyknometer;

m_3 is the mass, in g, of the pyknometer and test portion.

NOTE : The amount given is suitable for powders with absolute densities from 2,5 g/cm^3 to 4,0 g/cm^3; outside this range, take a proportionately different amount.