



**SLOVENSKI STANDARD**  
**SIST EN 725-6:2000**  
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**Advanced technical ceramics - Methods of test for ceramic powders - Part 6:  
Determination of the specific surface area**

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Hochleistungskeramik - Prüfverfahren für keramische Pulver - Teil 6: Bestimmung der  
spezifischen Oberfläche

Céramiques techniques avancées - Méthodes d'essai pour les poudres céramiques -  
Partie 6: Détermination de la surface spécifique

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ceramic powders - Part 6: Determination of the  
specific surface area**

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<b>Contents</b>	<b>Page</b>
Foreword	3
1 Scope	4
2 Principle	4
3 Apparatus	4
4 Procedure	6
5 Expression of results	9
6 Test report	15

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## Foreword

This European Standard has been prepared by CEN/TC 184 "Advanced technical ceramics", of which the secretariat is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical standard or by endorsement, at the latest by July 1996, and conflicting national standards shall be withdrawn at the latest by July 1996.

The method given is closely based on ISO 8008:1987 "Aluminium oxide primarily used for the production of aluminium - Determination of specific surface area by nitrogen absorption (single point method)", published by the International Organization for Standardization (ISO).

EN 725 'Advanced technical ceramics - Methods of test for ceramic powders' consists of eleven 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
- Part 4 : Determination of oxygen content of non-oxides by XRF analysis (ENV)
- Part 5 : Determination of particle size distribution
- Part 6 : Determination of specific surface area
- Part 7 : Determination of absolute density
- Part 8 : Determination of tapped density
- Part 9 : Determination of untamped bulk density
- Part 10 : Determination of compaction properties
- Part 11 : Determination of the densification on natural sintering (ENV)

In accordance with the Common CEN/CENELEC Rules, 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, UK.

## 1 Scope

This Part of EN 725 specifies a method for the determination of the specific surface area of powders used for technical ceramics, by a nitrogen adsorption, single-point method.

The method is limited to the measurement of surface areas of over 1 m<sup>2</sup>/g.

## 2 Principle

The method is based on the property of the solids to adsorb gas molecules at their surface.

In the range from 0,05 to 0,3 times the saturation vapour pressure of the measuring gas, the multilayer absorption begins. The formation of the first monolayer of adsorbed molecules can be detected from the behaviour of the adsorption isotherm in this range; this is the only process relevant to the present method.

Nitrogen is introduced at ambient temperature and at atmospheric pressure into two bottles of equal volume, one of which contains the test portion while the other is empty. The connected bottles are immersed in a refrigerant bath of liquid nitrogen. Since some nitrogen is adsorbed by the sample, a differential pressure occurs between the two bottles and is measured by means of a differential manometer. From this difference, the number of molecules adsorbed on the surface is calculated.

This number is multiplied by the known area occupied by a single adsorbed molecule so that the total surface area is obtained. The area occupied by an adsorbed nitrogen molecule is taken as  $16,2 \times 10^{-20}$  m<sup>2</sup>.

## 3 Apparatus

### 3.1 Adsorption apparatus (see figure 1)

The apparatus consists of a reference bottle (7) and a sample adsorption bottle (8) which are moved on to the two connecting pieces, with sealing rings in between to make the joint gas-tight. At each connecting piece, there is a valve (1 and 2), by which the bottles can be connected to the atmosphere. The measuring gas is admitted to the bottle through the capillaries inside the connecting pieces.

The bottles, made of shock-resisting glass, have a volume of about 100 cm<sup>3</sup>. The difference between the volume of the two bottles shall not exceed 0,1 %. The necks of the bottles consist of calibrated glass tubes with an internal diameter of 9 mm  $\pm$  0,02 mm. Each tube has an upper and lower mark.

In this way, several bottles can be used as either sample or reference bottles without compensating the volumes for each combination of bottles. A differential manometer containing dibutyl phthalate is arranged between the two bottles. The legs of the differential manometer are connected to the two inlet capillaries of the adsorption bottles. By means of the valve 4, the two bottles, i.e. the two inlet capillaries, can be separated from or connected to each other. By means of the valve 5, the liquid in the two legs of the differential manometer can be separated or connected. The legs of the differential manometer consist of calibrated glass tubes with an internal diameter of  $5 \text{ mm} \pm 0,02 \text{ mm}$ . This ensures that the change in volume during the adsorption measurement can be calculated with sufficient accuracy. The inlet capillary of the sample bottle, being the shorter of the two, is connected to a compensating volume (10), which is adjusted during preparation of the apparatus.

The measuring gas is admitted to the apparatus via the valve 3. If the valves 1, 2 and 4 are open, the measuring gas passed through both bottles. If the valves 1 and 4 are closed, the reference bottle is shut off and only the sample bottle is purged with the measuring gas.

During measurement, only a part of the gas volume, which is downstream of the valve 3 and upstream of the valves 1 and 2, is cooled by liquid nitrogen to the measuring temperature. The gas volume remaining at room temperature shall be limited to 10 % of the total volume at maximum. For this reason, the connections to the adsorption bottles are capillaries, which occupy most of the necks of the bottles in order to keep the portion remaining at room temperature as small as possible.

The adsorption bottle has calibrated volume of  $100 \text{ cm}^3$ . To facilitate weighing of the sample as well as the cleaning of the adsorption bottle, two-piece bottles are applied. The two pieces are connected by ground joints and fixed by hooks and springs.

**3.2 Thermostat**, for heating the adsorption bottles in the desorption procedure (see 4.3 and figure 2).

**3.3 Device** for degassing under vacuum (see figure 3).

**3.4 Cooling bath**, containing boiling nitrogen.

**3.5 Water bath**, capable of being controlled at  $22 \text{ }^\circ\text{C} \pm 3 \text{ }^\circ\text{C}$  or at approximately  $40 \text{ }^\circ\text{C}$ , as required.

## 4 Procedure

### 4.1 Test portion

Choose the mass of sample to be used depending on the supposed specific surface area.

Recommended values are given in table 1.

**Table 1 : Recommended masses of sample**

Supposed specific surface area	Mass of sample
$\text{m}^2 \text{g}^{-1}$	g
1	5
5	3
10	2
50	from 0,7 to 1,0
100	from 0,2 to 0,3
NOTE : If the surface area is completely unknown, preliminary tests are necessary to find the best test portion.	

### 4.2 Preparation of apparatus

#### 4.2.1 Checking of new apparatus

##### 4.2.1.1 General

New apparatus shall be checked to ensure that the compensating volume (10) is adjusted correctly and that the apparatus is gas-tight.

##### 4.2.1.2 Checking the equalization of volumes

The valve 4 shall be gas-tight when the equalization of the volumes is checked.

Fit the empty adsorption bottles to the connecting pieces; the upper mark of the bottle neck shall coincide with the lower sealing ring. Purge the apparatus with nitrogen by opening all the valves and allowing the gas to flow at approximately 10 l/h. During purging, the bottles are immersed in the water bath (see 3.5), up to the lower mark on the neck, in order to bring them to the set temperature of  $22 \text{ }^\circ\text{C} \pm 3 \text{ }^\circ\text{C}$ . When the equalization of the temperature can be assumed (after at least 5 min), shut off the apparatus from the atmosphere and shut off the two bottles from each other, by closing the valves in the sequence 1, 2, 3 and 4.



On shutting the valve 4, a small pressure difference can appear.

If this pressure difference changes within the next 2 min, a complete temperature equalization has not been reached in the adsorption bottles. In this case, open the valves again in the sequence 4, 3, 2 and 1, and purge the apparatus further with nitrogen. After a few minutes, repeat this check. If temperature equalization is achieved, close valve 5.

Remove the bottles from the water bath, dry any drops of water adhering to them, and immerse the bottles up to the lower mark in the cooling bath (see 3.4). When the bottles have attained the temperature of the boiling nitrogen (with empty bottles after about 1 min), open the valve 5 slowly. If the volumes of the sample and reference sides have been fully equalized, no pressure difference occurs. In this case, close the valve 5 again and open the valve 4. Then remove the cooling bath and replace it by the water bath (see 3.5), maintained at approximately 40 °C, to thaw out the bottles. Remove the warm water bath as soon as the bottles have reached ambient temperature again. After 2 min, reopen the valves 3, 2 and 1 in that sequence, and purge the apparatus with nitrogen. During the cooling and heating of the bottles, great pressure differences may occur momentarily so that the measuring liquid in the manometer may be transferred into other parts of the apparatus. For this reason, do not open the valve 5 before temperature equalization.

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If the pressure difference should occur during the preparation of the apparatus, change the compensating volume (10) in such a way that the volumes on both sides of the differential manometer are equal. To check the equalization of the volumes, repeat the test described above.

#### 4.2.1.3 Checking of tightness

If, after the slow opening of valve 5, steadily increasing or very great pressure differences occur (more than 400 mm of liquid column), during the equalization of volume at the temperature of boiling nitrogen, then the apparatus will have a leakage to the atmosphere (for instance, caused by the valves 1, 2, 3). Leakage of valve 4 cannot be recognized by a pressure difference and the following special check shall be carried out. Immerse the bottles in the cooling bath (see 3.4), close the valves 2, 3 and 4, and open the valves 1 and 5. By means of valve 1, create a pressure difference of approximately 300 mm of liquid column. Close the valve 1.

Within the next 10 min, the liquid column of the differential manometer shall not sink more than 1 mm.

#### 4.2.2 Checking of functioning

When the apparatus is put into operation, and later from time to time, check its functioning by measuring a solid with a known specific surface area. Measure the specific surface area of the test substance by the same method.

#### 4.2.3 Maintenance

Replace the measuring liquid of the differential manometer when it is dirty or at least every year. Change the used drying agent in the drying tower.

#### 4.3 Degassing of samples

Weigh the adsorption bottle and record its mass. Dry the test sample and place it in the adsorption bottle in such a way that no powder adheres to the neck of the bottle. Weigh the bottle again.

Evacuate the bottle and sample using a pressure of about 0,01 bar. Purge intermittently with pure nitrogen. The time necessary will vary for each type of powder and is reported (see clause 6). It is essential that the sample is completely degassed before the adsorption measurement. Weigh the bottle again after degassing. Then heat the bottle in the thermostat (see 3.2). The temperature used will also vary for each type of powder and shall be reported (see clause 6).

NOTE : For example, alumina powders require approximately 30 min at 200 °C.

#### 4.4 Adsorption measurement

Open the valves 2 and 3, pass nitrogen through the apparatus and connect the adsorption bottle filled with the pretreated sample and nitrogen to the connecting piece. Then open the valves 1, 4 and 5, and warm the bottles in the water bath (3.5), maintained at approximately 22 °C.

When the equilibrium pressure is attained, close the valves 1, 2, 3 and 4 and check the sample bottle and the reference bottle for temperature equalization. If a pressure difference appears, reopen the valves in the sequence 4, 3, 2 and 1 and continue purging with nitrogen. When temperature equalization is achieved, close the valve 5 and shut off the nitrogen flow. Immerse the bottles in the cooling bath (see 3.4) down to the lower mark, and open the valve 5 very slowly. If the existing pressure difference has become constant, read off this difference to an accuracy of 0,5 mm.

NOTE : The time to reach a constant pressure difference can be quite long for some materials, for example those having micropores or a low thermal conductivity.

Report the time interval from immersion of the bottles until reading of the pressure difference.

After having finished the measurement, close the valve 5 and open the valve 4. Replace the cooling bath (see 3.4) by the water bath (see 3.5), maintained at approximately 40 °C. After a few minutes, start the nitrogen flow and open the valves 3, 2, 1 and 5 in that sequence. When the connecting pieces are at ambient temperature, close the valves 1, 4 and 5 and change the sample bottle to carry out a new measurement.

## 5 Expression of results

### 5.1 Equations

The specific surface area  $S_m$  is given by the following equation:

$$S_m = 1,05 \frac{N_L \sigma_{N_2} (P_o - p)}{m R p_o} \left[ \frac{V \Delta p}{T_k} + \frac{V_z \Delta p}{T_z} + \frac{2V_x p}{T_z} + \frac{V_x \Delta p}{T_z} \right] + 1,05 \frac{N_L \sigma_{N_2} (P_o - p) V_p}{m R p_o} \left[ \frac{p}{T_k} - \frac{P_B}{T_z} \right] \quad \dots(1)$$

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where the symbols are as defined in table 3.

In this equation, the single-point calculation of the BET equation has been assumed and the correcting factor of 1,05 which represents the density of the liquid in the differential manometer introduced. For practical purposes,  $\sigma_{N_2}$ ,  $V/V_z T_k p_o$  have been taken as constants. In addition, the expression  $V_z \Delta p / T_z$  is small compared with the other terms of the sum in the first square brackets and has been neglected.

Incorporating the numerical values and units given in table 3, the following numerical equation is obtained with  $T_z = 295$  K for calculating the mass-related surface area:

$$S_m = 1,1736 \times 10^3 (1,05 - p) \frac{\Delta h}{m} (1,3693 \times 10^{-4} + 6,655 \times 10^{-5} p) + 1,1736 \times 10^3 (1,05 - p) \frac{V_p}{m} \left( \frac{p}{77,6} - \frac{P_B}{295} \right) \quad \dots(2)$$