



# SLOVENSKI STANDARD

## SIST ENV 725-4:2000

01-december-2000

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### Advanced technical ceramics - Methods of test for ceramic powders - Part 4: Determination of oxygen content in aluminium nitride by XRF analysis

Advanced technical ceramics - Methods of test for ceramic powders - Part 4:  
Determination of oxygen content in aluminium nitride by XRF analysis

Hochleistungskeramik - Prüfverfahren für keramische Pulver - Teil 4: Bestimmung des  
Sauerstoffgehaltes in Aluminiumnitrid-Pulvern mittels Röntgenfluoreszenzanalyse

Céramiques techniques avancées - Méthodes d'essai pour poudres céramiques - Partie  
4: Détermination de la teneur en oxygène du nitrure d'aluminium par spectrométrie de  
fluorescence X

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EUROPEAN PRESTANDARD

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English version

**Advanced technical ceramics - Methods of test for  
ceramic powders - Part 4: Determination of  
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**CEN**

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

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

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## Foreword

This European prestandard has been prepared by Technical Committee CEN/TC 184 'Advanced technical ceramics', of which the secretariat is held by BSI.

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 in aluminium nitride by XRF analysis (ENV)
- Part 5 : Determination of particle size distribution
- Part 6 : Determination of the specific surface area
- Part 7 : Determination of absolute density
- Part 8 : Determination of tapped bulk density
- Part 9 : Determination of untamped bulk density
- Part 10 : Determination of compaction properties
- Part 11 : Determination of densification on natural sintering (ENV)

CEN/TC 184 approved this European prestandard by resolution 1 during its seventh meeting held in Berlin, 1992-12-08/09.

In accordance with the CEN/CENELEC Internal Regulations, the following countries are bound to announce this European prestandard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

## 1 Scope

This part of EN 725 describes a method for the determination of oxygen contents of 2 % or less in aluminium nitride powder, by XRF analysis.

## 2 Normative references

This European prestandard incorporates by dated or undated reference provisions from other publications. These normative references are cited at 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 prestandard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

- EN 725-5      Advanced technical ceramics - Methods of test for ceramic powders - Part 5: Determination of particle size distribution<sup>1)</sup>
- EN 725-6      Advanced technical ceramics - Methods of test for ceramic powders - Part 6: Determination of specific surface area<sup>1)</sup>
- EN 955-5      Chemical analysis of refractory products - Part 5: XRF method using fused cast beads<sup>1)</sup>
- EN 45001      General criteria for the operation of testing laboratories

## 3 Principle

The sample is compacted to obtain a pellet, and the net intensity of the oxygen  $K_{\alpha}$  characteristic ( $I_0 K_{\alpha}$ ) of the pellet is measured by XRF spectrometer. The net intensity is the background intensity subtracted from the peak intensity at the peak position. The oxygen content in the sample is determined from this net intensity and by reference to previously defined calibration graphs; the calibration graph expresses the direct relation between net intensity and oxygen concentration. Calibration standards are prepared from different mixtures of fine aluminium nitride powder and a fine alumina powder.

## 4 Apparatus and experimental conditions

### 4.1 Apparatus

Sequential X-ray fluorescence spectrometer, equipped with a multilayer pseudo crystal and a gas flow proportional counter.

NOTE : For further details, refer to EN 955-5.

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1) In preparation.

#### 4.2 Experimental conditions

The experimental conditions depend on the apparatus, with an example of good practice being as follows:

- a) Mode: vacuum
- b) Excitation: tube voltage : 30 kV  
tube current : 95 mA
- c) Spectrometer : crystal : multilayer pseudo crystal  
2 d = 55 Å
- d) Peak : wavelength : 23,62 Å  
energy : 0,525 keV  
angular position :  $51^\circ 2 \theta$  (to be adjusted)
- e) Detection : detector type : gas flow proportional counter  
Ar-CH<sub>4</sub> or He-CO<sub>2</sub>
- pulse height selection : the discriminate levels  
(V<sub>min</sub>, V<sub>max</sub>) should be set so as:
- $$V_{\min}, V_{\max} = V \pm 1,3 W$$

where

W is the width at half height of the pulse amplitude distribution peaking at V (this allows more than 99,7 % of the pulse amplitude distribution to pass through the window).

- f) Background : Background intensity at the peak position is obtained using a linear regression between two background measurements before and after the fluorescence peak.

The recommended positions ( $2\theta$  min,  $2\theta$  max) are :

$$2\theta \text{ min, max} = 2\theta \pm 4 W_p$$

where  $2\theta_p$  is the peak position

$W_p$  is the peak width at half height of the peak

- g) Counting time: 100 s for both peak and background measurements;  
total counting time 300 s.

## 5 Sample preparation

### 5.1 Granulometry

Powders shall be fine enough to obtain a good homogeneous pellet without a binder. The particle size distribution (see EN 725-5) shall be between 10  $\mu\text{m}$  and 2  $\mu\text{m}$  with an equivalent diameter corresponding to 50 % of the grain size.

NOTE : Grinding the sample may increase the oxygen content of the powder.

### 5.2 Test piece preparation

5.2.1 Where comparatively large (10 g) samples are available, pour the powder into a die, 40 mm in diameter, and compact it using a hydraulic press. Apply a pressure of 80 MPa for 20 s. The powder may be placed in an aluminium cup before pressing if required.

5.2.2 For smaller samples (2 g), press the powder on to a backing plate made of cellulose or boric acid powder. Apply a pressure of 80 MPa for 20 s. The aluminium cup may again be used if required.

5.2.3 Prepare two samples for each determination.

NOTE : In most quantitative analysis, sample thickness is considered as infinite. If critical thickness is the thickness that would yield 99,9 % of the maximum intensity, calculation shows that critical thickness increased from 2,9  $\mu\text{m}$  to 3,35  $\mu\text{m}$  when oxygen concentration in AIN increases from 0,5 % to 10 %.

## 6 Calibration

### 6.1 Preparation of standards

Prepare calibration standards from mixtures of aluminium nitride powder in accordance with 5.1 and alumina powder with a specific surface area of about 6  $\text{m}^2/\text{g}$  (see EN 725-6) and an equivalent diameter corresponding to 50 % of the distribution, normally 1  $\mu\text{m}$  or less. Mix the powders for 10 min in an agate mortar and pestle, and vibrate the mixture for 1 min.

### 6.2 Calibration curves

Establish curves which record the oxygen ( $K_{\alpha}$ ) net intensity with the oxygen mass fraction in the different calibration mixtures.

As aluminium nitride powders used in calibration mixtures are not oxygen free, an addition method shall be used in order to determine the total oxygen concentration. In this method, a known amount of oxygen,  $\Delta[\text{O}]$ , is added to the sample. Fluorescence intensities  $I_1$  and  $I_2$ , before and after addition, are used to extrapolate to the unknown concentration. If linearity is assumed:



$$\frac{[O]}{\Delta [O]} = \frac{I_1}{I_2 - I_1}$$

The addition method is limited to fairly low concentrations, usually less than 1 %. Thereby, the use of an aluminium nitride powder with oxygen concentration  $\leq 1$  % is recommended for calibration purposes.

The shape of the calibration curve depends on the oxygen concentration range, but within a range of 0 % to 5 %, a linear regression is likely to be convenient.

### 6.3 Recalibration

Recalibration is needed in order to check long term instrumental drift. An aluminium nitride sintered pellet with a flat polished surface can be used to shift the calibration curve. The oxygen content of this reference standard should not be lower than 3 % in order to obtain a good signal intensity level.

Another solution, which is more time consuming, consists in the determination of a new calibration curve.

### 6.4 Sensitivity - limit of detection

EXAMPLE:

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Sensitivity (S) : slope of the linear part of the calibration curve  
expected sensitivity  $S = 80$  cps/% (counts per second per %).

Limit of detection for a confidence level of  $3\sigma$  ( $LD_{3\sigma}$ ).

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$$LD_{3\sigma} = \frac{3\sqrt{n/t}}{S}$$

where

n is the background intensity at the peak position = 40 cps  
t is the measuring time (100 s)  
 $LD_{3\sigma}$  is the oxygen = 240 ppm

### 7 Repeatability

This procedure has been applied to nine pellets of the same fine aluminium nitride powder, with the analytical results given in table 1.