SLOVENSKI STANDARD
SIST EN 725-3:2000

01-december-2000

Advanced technical ceramics - Methods of test for ceramic powders - Part 3: Determination of the oxygen content of non-oxides by thermal extraction with a carrier gas

Advanced technical ceramics - Methods of test for ceramic powders - Part 3: Determination of the oxygen content of non-oxides by thermal extraction with a carrier gas

Hochleistungskeramik - Prüfverfahren für keramische Pulver - Teil 3: Bestimmung des Sauerstoffgehaltes in Nichtoxid-Pulvern mittels Trägergasheißextraktion

Céramiques techniques avancées - Méthodes d'essai pour poudres céramiques - Partie 3: Détermination de la teneur en oxygene de poudres non-oxydes par extraction a chaud sous gaz porteur

Ta slovenski standard je istoveten z: **EN 725-3:1994**

ICS:

81.060.30	Sodobna keramika	Advanced ceramics

SIST EN 725-3:2000 **en**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 725-3

January 1994

UDC 666.3-121:620.1:546.21

Descriptors: Ceramics, powdery materials, chemical analysis, determination of content, oxygen, extraction methods, reduction methods, carbon monoxide

English version

Advanced technical ceramics - Methods of test for ceramic powders - Part 3: Determination of the oxygen content of non-oxides by thermal extraction with a carrier gas

Céramiques techniques avancées - Méthodes d'essai pour poudres céramiques - Partie 3: Détermination de la teneur en oxygène de poudres non-oxydes par extraction à chaud sous gaz porteur

Hochleistungskeramik - Prüfverfahren für keramische Pulver - Teil 3: Bestimmung des Sauerstoffgehaltes in Nichtoxid-Pulvern mittels Trägergasheißextraktion

This European Standard was approved by CEN on 1994-01-10. CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

The European Standards exist in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

CEN

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

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

Ref. No. EN 725-3:1994 E

Contents list

	Page
Foreword	3
1 Scope	4
2 Principle	4
3 Apparatus	4
4 Sample preparation	5
5 Procedure	5
6 Blank determination	6
7 Calibration	6
8 Test report	6
Annex A (Informative) Guidance in the selection of test conditions	7

Foreword

This European Standard has been prepared by Technical Committee CEN/TC 184 'Advanced technical ceramics', the Secretariat of which 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)

This document was submitted to the formal vote and approved as European Standard.

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 July 1994, and conflicting national standards shall be withdrawn at the latest by July 1994.

In accordance with the CEN/CENELEC Internal Regulations, 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 United Kingdom.

1 Scope

This part of EN 725 describes a method for the determination of the oxygen content of non-oxide powders used for advanced technical ceramics, using an inert carrier gas thermal extraction method. The method is suitable for oxygen contents of less than 3 %.

NOTE : The limits of determination are given by the manufacturers of the apparatus.

2 Principle

A test piece is heated in a graphite crucible at a high temperature in a flow of an inert carrier gas. Oxygen in the sample is converted to oxides of carbon, which are extracted and transformed to consist entirely of either carbon monoxide or carbon dioxide. This volume is then determined by a suitable method of gas analysis.

NOTE : Guidance on the selection of test conditions is given in annex A.

3 Apparatus

3.1 Graphite crucible, which is used as a carbon source. The crucible is capable of being electrically heated by two electrodes, or by induction.

3.2 Tin capsule, (optional) to contain the ceramic powder sample.

3.3 Nickel wire basket, (optional) for use as a fluxing agent with certain powders such as aluminium nitride.

3.4 Gas analysis apparatus, based on one of the following techniques:

a) volumetric analysis, for measurement of carbon monoxide gas

b) chromatography, for carbon monoxide

c) thermal conductivity, for carbon monoxide and carbon dioxide

d) coulometric analysis, for carbon dioxide

e) infrared absorption, for carbon dioxide

3.5 Furnace, normally capable of reaching at least 2 500 °C operating in an inert gas atmosphere (nitrogen, helium or argon).

NOTE : Apparatus which does not achieve 2 500 °C may be used for lower test temperatures, but 2 500 °C will needed to give the correct result for certain types of material.

4 Sample preparation

Take a sample of the dry powder, the quantity taken being based on the detection limit of the apparatus used for gas analysis and on the expected oxygen content of the powder.

NOTE : For example, for infrared absorption (see 3.4 e)), samples of 100 mg to 300 mg are required.

Use a scoop to handle the powder and place the sample either directly in the crucible or in the tin capsule. If the capsule is used, squeeze it to expel air and then fold over the open end several times, weighing again after squeezing.

5 Procedure

5.1 Heat the empty graphite crucible inside the furnace to a temperature greater than the temperature for analysis. Record the flow of gas used in the furnace.

NOTE 1 : This is to prevent additional degassing of the crucible during the test run, which would produce errors.

NOTE 2 : A recommendation for gas flow will normally be given by the manufacturer of the instrument.

Cool to ambient temperature.

5.2 Place the tin capsule and sample in the crucible. Select a temperature which is high enough to dissociate the oxides to be determined (see annex A). Use the nickel wire basket (see 3.3) if the preliminary tests suggest that it is necessary.

Heat the furnace to the test temperature and record the temperature and the time at temperature. Collect the gas evolved and measure the amount, using one of the analytical techniques listed in 3.4.

5.3 Repeat the procedure given in clause 4 and subclause 5.2 at least four times.

6 Blank determination

Carry out a blank determination for the apparatus whenever the gas supply or crucible batch is changed. Use the procedure given in clause 5 and record the amount of gas evolved. Use this blank value to calculate the results for the sample analysis.

7 Calibration

Calibration shall be carried out preferably by using a reference material of certified oxygen content to calibrate the furnace and gas analysis equipment, using the procedure given in clause 5.

An alternative method is to use a sample of pure carbon monoxide or carbon dioxide gas to calibrate the gas analysis equipment, with a minimum of three results being recorded.

8 Test report

The test report shall include the following information:

a) the name of the test establishment;
b) the date of the test, a unique identification of the report, the name and address of the customer, a signatory of the report;
c) a reference to this European standard, i.e. Determined in accordance with EN 725-3;
d) a description of the test material (manufacturer, type, batch or code number);
e) the type of analytical equipment used (see 3.4);
f) the method of calibration (see clause 7);
g) the purity of the carrier gas and gas used for calibration;
h) the mass of the samples and reference to optional use of the tin capsule (see 3.2);
i) the test temperature;
j) the individual values of oxygen content, mean value and standard deviation;
k) Comments about the test or test results.