SLOVENSKI STANDARD
SIST EN 725-1:2000

01-december-2000

Advanced technical ceramics - Methods of test for ceramic powders - Part 1: Determination of impurities in alumina

Advanced technical ceramics - Methods of test for ceramic powders - Part 1: Determination of impurities in alumina

Hochleistungskeramik - Prüfverfahren für keramische Pulver - Teil 1: Bestimmung von Verunreinigungen in Aluminiumoxidpulver

Céramiques techniques avancées - Méthodes d'essai pour poudres céramiques - Partie 1: Détermination des impuretés dans l'alumine

Ta slovenski standard je istoveten z: **EN 725-1:1997**

ICS:

81.060.30	Sodobna keramika	Advanced ceramics

SIST EN 725-1:2000 **en**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 725-1

March 1997

ICS 81.060.99

Descriptors: ceramics, powdery materials, impurities, aluminium oxide, chemical analysis, determination of content, sodium oxides, potassium oxides, iron oxides, magnesium oxides, calcium oxides, silicon oxides, atomic absorption spectrophotometry

English version

Advanced technical ceramics - Methods of test for ceramic powders - Part 1: Determination of impurities in alumina

Céramiques techniques avancées - Méthodes d'essai pour poudres céramiques - Partie 1: Détermination des impuretés dans l'alumine

Hochleistungskeramik - Prüfverfahren für keramische Pulver - Teil 1: Bestimmung von Verunreinigungen in Aluminiumoxidpulver

This European Standard was approved by CEN on 1997-02-24. 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 725-1:1997 E

Contents

Foreword

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

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 September 1997, and conflicting national standards shall be withdrawn at the latest by September 1997.

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
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 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 reactivity on sintering (ENV)

According to the CEN/CENELEC Internal Regulations, the national standards organizations of 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 Part of EN 725 specifies methods for the determination of elements of sodium, potassium, iron, silicon, calcium and magnesium present as impurities in alumina using atomic absorption (AAS) or inductively coupled plasma (ICP) instruments. For each element present as impurities, the methods are applicable to the following ranges, calculated as oxides:

Sodium oxide	20 ppm to 6000 ppm
Potassium oxide	20 ppm to 100 ppm
Ferric oxide	20 ppm to 300 ppm
Silica	50 ppm to 2000 ppm
Calcium oxide	20 ppm to 700 ppm
Magnesium oxide	5 ppm to 1000 ppm

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and in the publications 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.

ECSC/CI 9	Chemical analysis of ferrous materials - Operational guidelines for the application of flame atomic absorption spectrometry in standard methods for the chemical analysis of iron and steel.
ISO 3696	Water for analytical laboratory use - Specification and test methods.
ISO 5725	Precision of test methods - Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.
ISO/DIS 13527	Chemical analysis of ferrous materials - Guidelines on the use of inductively coupled plasma atomic emission spectroscopy

3 Principle

The test sample is decomposed by using either a fusion method or an acid dissolution method. The acid dissolution method cannot be used for the determination of silicon. The solution is transferred to a volumetric flask and diluted to a known volume, and the elements are determined by AAS or ICP (see clause 1).

4 Reagents

4.1 General

During the analysis, use only reagents and calibration solutions of at least 99,99 % purity and only distilled water or water of equivalent purity (see ISO 3696).

4.2 Reagents for fusion

4.2.1 Lithium carbonate - Li_2CO_3

4.2.2 Potassium carbonate K_2CO_3

4.2.3 Boric acid H_3BO_3

4.2.4 Sulphuric acid H_2SO_4 - (ρ_{20} = 1,84 g/ml)

4.2.5 Lithium metaborate $LiBO_2$

4.2.6 Nitric acid HNO_3 - (ρ_{20} = 1,33 g/ml)

4.2.7 Phosphoric acid H_3PO_4 - (ρ_{20} = 1,78g/ml)

4.3 Reagents for acid dissolution

4.3.1 Sulphuric acid-phosphoric acid mixture (A)

Pour 500 ml of phosphoric acid (ρ_{20} = 1,78 g/ml) into 500 ml of sulphuric acid (ρ_{20} = 1,84 g/ml)

4.3.2 Sulphuric acid-phosphoric acid mixture (B)

Pour 700 ml of phosphoric acid (ρ_{20} = 1,78 g/ml) into 300 ml of sulphuric acid (ρ_{20} = 1,84 g/ml).

4.4 Reagents for calibration

4.4.1 Pure alumina, of very low and known impurity levels.

4.4.2 Sodium - commercial solution or solution obtained by dissolution of pure chemical compound, concentration 1 g/l.

4.4.3 Potassium - commercial solution or solution obtained by dissolution of pure chemical compound, concentration 1 g/l.

4.4.4 Iron (ferric) - commercial solution or solution obtained by dissolution of pure chemical compound, concentration 1 g/l.

4.4.5 Silicon - commercial solution or solution obtained by dissolution of pure chemical compound, concentration 1 g/l.

4.4.6 Calcium - commercial solution or solution obtained by dissolution of pure chemical compound, concentration 1 g/l.

4.4.7 Magnesium - commercial solution or solution obtained by dissolution of pure chemical compound, concentration 1 g/l.

5 Apparatus

5.1 Platinum crucible with a capacity of at least 50 ml

5.2 Gold-platinum crucible with a capacity of at least 50 ml

5.3 Vitreous carbon crucible with a capacity of at least 50 ml with lid and heating device

5.4 Muffle furnace, suitable for operation in the range of 1000 °C to 1200 °C

5.5 Magnetic stirrer, with heating

5.6 Atomic absorption spectrometer and/or inductively coupled plasma spectrometer, in accordance with ECSC/CI 9 or ISO 13527 respectively

5.7 Laboratory glassware

6 Test sample

Use samples of approximately :

- 2 g for decomposition by fusion;
- 1 g for decomposition by acid dissolution.

Weigh them to 0,0005 g.

7 Decomposition of the test sample

7.1 General

Dissolve either by a fusion method (see 7.2 to 7.4) or an acid dissolution method (see 7.5 and 7.6)

7.2 Fusion 1

In a platinum crucible (see 5.1) weigh 1,5 g Li_2CO_3 (see 4.2.1) 5 g K_2CO_3 (see 4.2.2) and 2,5 g H_3BO_3 (see 4.3.3). Add the test sample of approximately 2 g (see clause 6) and mix intimately using a platinum spatula.

Place the crucible and contents into the muffle furnace (see 5.4), maintained at 1050 °C ± 50 °C, for 30 min. Remove the crucible from the furnace and swirl the contents on the sides of the crucible, then allow to cool to room temperature.

Dissolve the fused product in a 400 ml beaker which contains 100 ml of water and 10 ml of sulphuric acid (see 4.2.4). Place the beaker, covered with a watch glass, on a hot plate and heat to boiling. Maintain at boiling point to obtain a complete dissolution. Remove the beaker from the hot plate. Allow to cool.

Transfer quantitatively the solution into a 200 ml volumetric flask. This procedure allows for a concentration of alumina up to 8 g/l but if needed, a dilution to a higher volume is possible.

Allow to cool to room temperature and make up to the mark.

7.3 Fusion 2

In a platinum crucible (see 5.1) weigh 4 g of $LiBO_2$ (see 4.2.5) and 1 g of test sample. Mix intimately using a platinum spatula.

Place the crucible and contents into the muffle furnace (see 5.4) maintained at 1150 °C ± 50 °C for 30 min (after the first 15 min, swirl the contents of the crucible for a few seconds). Remove the crucible from the furnace and dip its base in water at ambient temperature (this procedure allows easy removal of the bead from the crucible). To prevent sticking of melt in the crucible, either use a new crucible or, with an old one, immerse it in the solution. Place the bead into a 400 ml beaker which contains 80 ml of water and 20 ml of nitric acid (see 4.2.6).