SLOVENSKI STANDARD

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Sodobna tehniļna keramika - Preskusne metode za keramiļne praġke - 1. del: Ugotavljanje neļistoļ v aluminijevem oksidu

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

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

Céramiques techniques avancées - Méthodes d'essai des poudres céramiques - Partie 1 : Dosage 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 11 August 2007.

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This European Standard exists 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 CEN Management Centre has the same status as the official versions.

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Foreword

This document (EN 725-1:2007) 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of an identical text or by endorsement, at the latest by March 2008, and conflicting national standards shall be withdrawn at the latest by March 2008.

This document supersedes EN 725-1:1997.

EN 725 *Advanced technical ceramics — Methods of test for ceramic powders* was prepared in parts as follows:

Part 1: *Determination of impurities in alumina*

Part 2: *Determination of impurities in barium titanate*

Part 3: *Determination of the oxygen content of non-oxides by thermal extraction with a carrier gas*

Part 4: *Determination of oxygen content in aluminium nitride by XRF analysis*

Part 5 *Determination of particle size distribution*

Part 6: *Determination of the specific surface area* [withdrawn]

Part 7: *Determination of the absolute density* [withdrawn]

Part 8: *Determination of tapped bulk density*

Part 9: *Determination of un-tapped bulk density*

Part 10: *Determination of compaction properties*

Part 11: *Determination of densification on natural sintering*

Part 12: *Chemical analysis of zirconia*

Parts 6 and 7 of the series were superseded in 2005 by EN ISO 18757 and EN ISO 18753 respectively.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

1 Scope

This Part of EN 725 specifies one fusion and one acid dissolution method for the determination of elements of sodium, potassium, iron, silicon, calcium and magnesium present as impurities in alumina using atomic absorption spectroscopy (AAS) or inductively coupled plasma (ICP) spectroscopy. 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

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN ISO 3696, *Water for analytical laboratory use - Specification and test methods (ISO 3696:1987)*

EN ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories (ISO/IEC 17025:2005)*

3 Principle

A test sample is decomposed by using either a fusion method or an acid dissolution method.

NOTE 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.

4 Reagents

4.1 General

During the analysis, use only reagents and calibration solutions of at least 99,99 % purity and water conforming to EN ISO 3696, Grade 3, or better.

4.2 Reagents for fusion

4.2.1 Lithium metaborate - $LiBO_2$

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

4.3 Sulphuric acid-phosphoric acid mixture for acid dissolution

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, 99,99% purity.

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

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

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

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

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

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

5 Apparatus

5.1 Platinum or platinum-gold crucible with a capacity of at least 50 ml

5.2 Muffle furnace, suitable for operation in the range of (1 000 ± 50) °C to (1 200 ± 50) °C

5.3 Hot plate with magnetic stirrer

5.4 Atomic absorption spectrometer and/or inductively coupled plasma spectrometer

5.5 Laboratory glassware

5.6 Platinum spatula

6 Test sample

Use samples of approximately:

— 1 g for decomposition by fusion;

— 1 g for decomposition by acid dissolution.

Weigh them to ± 0,0001 g.

7 Decomposition of the test sample

7.1 General

Dissolve either by a fusion method (see 7.2) or an acid dissolution method (see 7.3)

7.2 Fusion

Weigh 4 g of $LiBO_2$ (4.2.1) and 1 g of test sample into a platinum or platinum-gold crucible (5.1). Mix intimately using a platinum spatula, put on lid.

Place the crucible and contents into the muffle furnace (5.2) maintained at 1150 °C ± 50 °C for 30 min (after the first 15 min, swirl the contents of the crucible for a few seconds and put back in the muffle). Remove the crucible from the furnace, remove lid and rinse with distilled water, pouring the residue into a 400 ml beaker containing 80 ml of water and 20 ml of nitric acid (4.2.2). 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. Pour the bead, carefully, into the 400 ml beaker that contains 80 ml of water and 20 ml of nitric acid (4.2.2).

Place the beaker, covered with a watch glass on a hot plate (5.3) with magnetic stirring and maintain the agitation at approximately 80 °C ± 10 °C until complete dissolution. Remove the beaker from the stirrer and allow it to cool down. Transfer the solution quantitatively into a 200 ml volumetric flask. Allow it to cool down to room temperature and make up to the mark.

7.3 Acid dissolution

Weigh the test sample (see Clause 6) into a platinum or gold-platinum crucible (5.1). Add carefully 12 ml of sulphuric acid-phosphoric acid mixture (4.3) and cover with a lid. Put the crucible with the lid on to the hot plate (5.3) and maintain it at boiling for 12 min. Remove the crucible from the heating device and allow it to cool down.

Transfer the content quantitatively into a 100 ml volumetric flask which contains 30 ml of water. Rinse the crucible and the lid with distilled water into the flask and after cooling, make up to the mark with water.

8 Calibration graph

8.1 General

The optimum calibration graph is obtained using calibration solutions whose concentrations are compatible both with the analytical method (AAS or ICP) and with the impurity concentrations in the sample, and matrix matched.

The following procedure is given as an example.

8.2 Fusion

Prepare five decompositions of pure alumina (4.4.1) in accordance with 7.2. Transfer into five 200 ml volumetric flasks and dilute to 150 ml with water.

Add the quantities of solutions indicated in Table 1. Make up to the mark with water.

Table 1 — Quantities of solutions for fusion

Elements	1	2	3	4	5
Na	0 µl	1 µl	2 µl	4 µl	6 µl
K	0 µl	100 µl	200 µl	300 µl	400 µl
Ca	0 µl	250 µl	500 µl	750 µl	1000 µl
Fe	0 µl	100 µl	200 µl	300 µl	400 µl
Si	0 µl	200 µl	400 µl	800 µl	1600 µl
Mg	0 µl	250 µl	500 µl	750 µl	1000 µl

8.3 Acid dissolution

Prepare 5 dissolutions of pure alumina (4.4.1) in accordance with 7.3. Transfer into five 100 ml volumetric flasks and dilute to 50 ml with water. Add the quantities indicated in Table 2. Make up to the mark with water.

Table 2 — Quantities of solutions for acid dissolution

Elements	1	2	3	4	5
Na	0 µl	500 µl	1000 µl	2000 µl	3000 µl
K	0 µl	50 µl	100 µl	150 µl	200 µl
Ca	0 µl	125 µl	250 µl	375 µl	500 µl
Fe	0 µl	50 µl	100 µl	150 µl	200 µl
Mg	0 µl	125 µl	250 µl	375 µl	1000 µl

8.4 Drawing the calibration curve

8.4.1 Blank test

Prepare a blank test in accordance with 8.2 or 8.3 using the same quantities of all reagents as for dissolution of the test sample, but using pure alumina (4.4.1) in place of the test sample.

8.4.2 Drawing the calibration curve

Draw a graph of the AAS or ICP intensities recorded using the calibration solutions prepared against the impurity concentrations.

9 Adjustment of the apparatus

9.1 Atomic absorption spectrometer

Follow the manufacturer's instructions for igniting and extinguishing the nitrous oxide-acetylene flame to avoid explosion, and ensure the safety screen is in place.

Set the wavelengths for the elements to be analysed (see Table 3) and adjust the apparatus so as to obtain maximum absorbance. Fit the correct burner and, in accordance with the manufacturer's instructions, light the