SLOVENSKI STANDARD
SIST ENV 725-2:2000

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Advanced technical ceramics - Methods of test for ceramic powders - Part 2: Determination of impurities in barium titanate

Advanced technical ceramics - Methods of test for ceramic powders - Part 2: Determination of impurities in barium titanate

Hochleistungskeramik - Prüfverfahren für keramische Pulver - Teil 2: Bestimmung von Verunreinigungen in Bariumtitanat

Céramiques techniques avancées - Méthodes d'essai pour poudres céramiques - Partie 2: Détermination des impuretés dans le titane de baryum

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English version

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CEN

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

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Contents list

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 impurities in barium titanate powders, using inductively coupled plasma atomic emission (ICP-AES).

The method is applicable only to stoichiometric barium titanate. The maximum concentrations measured for each impurity are as follows:

- Sr: 4 000 ppm
- Ca: 500 ppm
- K : 200 ppm
- Na, Mg, Al, Fe, Nb: 100ppm

The minimum concentration or detection limits are from ppm to 5 ppm.

2 Normative references

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

EN 45001	General criteria for the operation of testing laboratories.
ISO 13527	Chemical analysis of ferrous materials - Guidelines on the use of inductively coupled plasma atomic emission spectrometry.[1]

3 Principle

Calcined barium titanate powder is dissolved in a mixture of hydrochloric acid and hydrogen peroxide, and the impurities in the solution are determined by ICP-AES analysis, using an addition method of synthetic matrix matched solutions for the calibration.

4 Reagents

Spectroscopic grade reagents shall be used, including:

4.1 Quartz distilled water or water of equivalent purity;
4.2 Hydrochloric acid, density = 1.18 g/cm^3 at 20°C;
4.3 Hydrogen peroxide, 110 volume;
4.4 Barium chloride solution;
4.5 Titanium chloride, $TiCl_4$, in solution.

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[1] In course of preparation.

5 Apparatus

5.1 Spectrometer for inductively coupled plasma atomic emission, with a quartz torch hand pneumatic cross-flow nebuliser. See ISO 13527.
5.2 Muffle furnace for operation at 900 °C ± 50 °C.
5.3 Platinum crucible and lid.
5.4 Balance to weigh to ± 0,001 g.
5.5 PTFE beakers, 100 ml, with paraffin film.
5.6 Polyethylene pipettes, micro-pipettes and volumetric flasks.
5.7 Magnetic stirrer with a small bar.
5.8 Desiccator.

6 Preparation of sample solution

6.1 Sample calcination

Weigh about 1,2 g of the sample in the platinum crucible. Calcine the sample for two hours in the muffle furnace at 900 °C ± 50 °C. Remove it from the hot furnace and allow it to cool in the desiccator.

6.2 Test sample

Weigh 1,000 g of the calcined powder from 6.1, to the nearest 0,001 g, and place this test sample in a 100 ml PTFE beaker.

6.3 Dissolution of the test sample

Place the magnetic bar (5.7) in the beaker containing the test sample and carefully add 25 ml of water (4.1), 25 ml of hydrochloric acid (4.2) and 8 ml of hydrogen peroxide (4.3). Cover the beaker with paraffin film. Heat the beaker at 60 °C, with magnetic stirring, until either the sample is dissolved or the amount of residue is constant.

NOTE : A 'double boiler' is recommended for heating.

If there is residue, remove this by sedimentation and repeat the dissolution procedure.

Transfer the cool solution to a 100 ml volumetric flask and make up to volume with the distilled water (4.1). Use the solution for the analysis (clause 8) within 48 h.

7 Calibration

7.1 Samples with unknown impurity levels

Samples with unknown impurity levels shall be calibrated by an addition method. Prepare the final test solutions by diluting the sample solution (6.3) so that:

a) the final test solutions with and without added amounts of standard solutions (4.4 and 4.5) are in the concentration range given in table 1.

b) the amount of standard solution added is similar to the amount of impurities in the final test solution.

7.2 Samples with known impurity levels

Samples with known impurity levels shall be calibrated by a synthetic method. This method uses solutions which are matrix-matched with $BaCl_2$, $TiCl_4$, HCl and H_2O_2. The concentration of matrix components in these solutions shall be such that the concentration of the impurities is within the range given in table 1.

Table 1 : Concentration range for impurities

Element	Concentration range $(10^{-6}\ g\ ML^{-1})$
Na	0 - 10
K	0 - 5
Mg	0 - 0,3
Ca	0 - 3
Sr	0 - 5
Al	0 - 10
Fe	0 - 1
Nb	0 - 15

8 Procedure

Adjust the ICP spectrometer (see 5.1) to provide a stable plasma, a high signal-to-noise ratio and a separation of spectral overlaps for the impurities being determined. Follow the manufacturer's instructions for igniting the plasma and wear UV eye protectors as soon as the plasma can be seen.

Use the wavelengths given in table 2 for determination of each impurity.