SLOVENSKI STANDARD
SIST EN 725-2:2009

01-januar-2009

Nadomeġļ a:
SIST ENV 725-2:2000

Sodobna tehniļ na keramika - Preskusne metode za keramiļ ne praġke - 2. del: Ugotavljanje neļ istoļ v barijevem titanatu

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 Bariumtitanatpulvern

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

Ta slovenski standard je istoveten z: EN 725-2:2007

ICS:

81.060.30	Sodobna keramika	Advanced ceramics

SIST EN 725-2:2009 en,fr,de

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 725-2

November 2007

ICS 81.060.30

Supersedes ENV 725-2:1994

English Version

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

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Ref. No. EN 725-2:2007: E

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Foreword

This document (EN 725-2:2007) 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of an identical text or by endorsement, at the latest by May 2008, and conflicting national standards shall be withdrawn at the latest by May 2008.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes ENV 725-2:1994.

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Part 6: *Determination of the specific surface area* [withdrawn]

Part 7: *Determination of the absolute density* [withdrawn]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

Part 6 and part 7 of this series were superseded in 2005 by EN ISO 18757 and EN ISO 18753.

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 the 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 optical emission spectroscopy (ICP-OES).

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

— Sr 4 mg/g (4 000 ppm)

— Ca 500 µg/g (500 ppm)

— K 200 µg/g (200 ppm)

— Na, Mg, Al, Fe, Nb 100 µg/g (100 ppm)

The minimum concentration or detection limits are from 1 µg/g to 5 µg/g (1 ppm to 5 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/IEC 17025, *General requirements for the competence of testing and calibration laboratories (ISO/IEC 17025:2005)*

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-OES analysis, using an addition method of synthetic matrix matched solutions for the calibration.

4 Reagents

Spectroscopic grades of the following reagents shall be used.

4.1 Quartz distilled water or water of equivalent purity.

4.2 Hydrochloric acid, density = 1,18 g/cm³.

4.3 Hydrogen peroxide, 110 V/V.

4.4 Barium chloride solution, 99,999 % purity.

4.5 Titanium chloride, $TiCl_4$, in solution, 99,999 % purity.

4.6 Reagents for calibration

A commercial solution, or solution obtained by dissolution of a pure chemical compound, of concentration 1g/l shall be used for each of the following: strontium; calcium; potassium; sodium; magnesium; aluminium; iron; and niobium.

5 Apparatus

5.1 Spectrometer for inductively coupled plasma atomic emission, with a quartz torch hand pneumatic cross-flow nebulizer.

5.2 Muffle furnace, for operation at 900 °C ± 50 °C.

5.3 Platinum or platinum/gold 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 100 ml volumetric flasks.

5.7 Hot plate, with magnetic stirrer.

5.8 Dessicator.

5.9 Double boiler.

6 Preparation of sample solution

6.1 Sample calcination

Weigh about 1,2 g to an accuracy of ± 0,001 g of the sample into a platinum crucible. Calcine the sample for 2 h 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 ± 0,001 g of the calcined powder from 6.1 and place this test sample into 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 to 60 °C ± 10 °C and maintain at this temperature while 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 filtration and repeat the dissolution procedure.

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

7 Calibration

7.1 Standard addition method

For non-routine samples with unknown impurity levels, standard addition, as follows, can be used to determine the concentration of impurities. Prepare the final test solutions by diluting the sample solution (6.3) so that:

a) final test solutions with added amounts of standard solutions (4.6) are in the concentration range given in Table 1;

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

7.2 Calibration method

Routine samples with known impurity levels shall be calibrated by a synthetic calibration 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 the scope.

Table 1 — Concentration range for impurities

Element	Concentration range (10^{-6} gml^{-1})
Na	0 to 10
K	0 to 5
Mg	0 to 0,3
Ca	0 to 3
Sr	0 to 5
A1	0 to 10
Fe	0 to 1
Nb	0 to 15

8 Procedure

8.1 ICP spectrometer operation

Adjust the ICP spectrometer (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.

NOTE Use of UV eye protectors is necessary.

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

Table 2 — Wavelengths for each impurity

Element	Wavelength (nm)
Na	589,59
K	766,47
Mg	279,55
Ca	422,67 or 393,37
Sr	346,44 or 407,77
Al	396,15
Fe	259,94
Nb	309,42 or 316,34

8.2 Standard addition method

Aspirate the blank solution and then the samples in order of increasing signal. Extrapolate the signal back to give the concentration of impurity in ppm, after the blank value has been subtracted.

8.3 Calibration method

Aspirate the blank solution and then the calibration solutions in order of increasing signal. Aspirate the equivalent hydrochloric acid solution (4.2) between each of these solutions and record the readings when stable responses are reached. Take three measurements and calculate the mean.

Prepare calibration graphs for each element by plotting the signal values against quantities of impurity in the test sample, expressed as ppm. Use these calibration graphs to convert the signal values, after the blank value has been subtracted, of the test solution into concentration of impurities in the test sample (see 6.2).

Aspirate the test solutions and read the intensity values from the graphs. Express the results in ppm of the test sample.

9 Test report

The test report shall be in accordance with the reporting provisions of EN ISO/IEC 17025 and shall include at least the following information:

a) name and address of the testing establishment;

b) date of the test;

c) on each page, a unique report identification and page number;

d) customer name and address;

e) reference to this standard, i.e. determined in accordance with EN 725-2:2007;

f) authorizing signature;

g) any deviation from the method described, with appropriate validation, i.e. demonstrated to be acceptable to the parties involved;