SLOVENSKI STANDARD

SIST ENV 14226:2007

01-januar-2007

Sodobna tehniļ na keramika - Preskusne metode za keramiļ ne praġke - Doloļ evanje kalcija, magnezija, ġeleza in aluminija v silicijevem nitridu z uporabo plamenske atomske absorpcijske spektrometrije (FAAS) ali atomske emisijske spektrometrije z induktivno sklopljeno plazmo (ICP - AES)

Advanced technical ceramics - Test methods for ceramic powders - Determination of calcium, magnesium, iron and aluminium in silicon nitride by using flame atomic absorption spectroscopy (FAAS) or inductively coupled plasma atomic emission spectroscopy (ICP-AES)

Hochleistungskeramik - Prüfverfahren für keramische Pulver - Bestimmung von Calcium, Magnesium, Eisen und Aluminium in Siliciumnitrid mittels Flammen-Atomabsorptionsspektroskopie (FAAS) oder Atomemissionsspektroskopie mit induktiv gekoppeltem Plasma (ICP-AES)

Céramiques techniques avancées - Méthodes d'essai pour poudres céramiques - Détermination du calcium, magnésium, fer, et aluminium dans le nitrure par spectrométrie d'absorption atomique dans la flamme (FAAS) ou par spectrométrie d'émission atomique avec plasma a couplage inductif (ICP-AES)

Ta slovenski standard je istoveten z: **ENV 14226:2002**

ICS:

81.060.30	Sodobna keramika	Advanced ceramics

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EUROPEAN PRESTANDARD

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English version

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This European Prestandard (ENV) was approved by CEN on 23 December 2001 as a prospective standard for provisional application.

The period of validity of this ENV is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the ENV can be converted into a European Standard.

CEN members are required to announce the existence of this ENV in the same way as for an EN and to make the ENV available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the ENV) until the final decision about the possible conversion of the ENV into an EN is reached.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

This document ENV 14226:2002 has been prepared by Technical Committee CEN/TC 184 “Advanced technical ceramics”, the secretariat of which is held by BSI.

This Prestandard includes a Bibliography.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this European Prestandard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This European Prestandard specifies methods for the determination of calcium, magnesium, iron, and aluminium, using flame atomic absorption spectroscopy (FAAS), or inductively coupled plasma atomic emission spectroscopy (ICP-AES).

The methods are applicable to the concentration ranges given in clause 3.

2 Normative references

This European Prestandard incorporates, by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Prestandard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

ISO 3696, *Water for analytical laboratory use - Specification and test methods.*

3 Concentration ranges

The method is applicable to concentration ranges for each element as follows:

Calcium	5×10^{-6} to 500×10^{-6} (*w*/*w*);
Magnesium	5×10^{-6} to 200×10^{-6} (*w*/*w*);
Iron	5×10^{-6} to 250×10^{-6} (*w*/*w*);
Aluminium	5×10^{-6} to 1000×10^{-6} (*w*/*w*).

4 Principle

The test sample is decomposed using either acid pressure digestion, or acid pressure microwave digestion.

The resulting solution is diluted to a defined volume and nebulization into a flame for FAAS, or into a plasma, for ICP-AES (see clause 1).

For each element, comparison is made of the recorded absorbance or emitted intensity for a determined analytical peak with those of a range of solutions of known concentrations.

5 Sampling

Bulk sampling is not within the scope of this European Prestandard and the method starts with a laboratory sized sample.

6 Reagents and materials

6.1 General

All reagents, and substances, shall be of at least analytical grade. Distilled or de-ionised water, conforming to ISO 3696, shall be used throughout the whole procedure.

6.2 Reagents for preparation

6.2.1 Hydrofluoric acid HF ρ_{20} about 1,16 g ml^{-1}.

6.2.2 Nitric acid HNO_3 ρ_{20} about 1,4 g ml^{-1}.

6.3.Reagents for calibration

6.3.1 All reagents shall be of ultra high purity.

6.3.2 Silicon, commercial solution with very low and known impurity levels, concentration 10 gl^{-1}.

6.3.3 Calcium, commercial solution or solution obtained by the dissolution of a pure chemical product, concentration 0,1 gl^{-1}.

6.3.4 Magnesium, commercial solution or solution obtained by the dissolution of a pure chemical product, concentration 0,1 gl^{-1}.

6.3.5 Iron, commercial solution or solution obtained by the dissolution of a pure chemical product, concentration 0,1 gl^{-1}.

6.3.6 Aluminium, commercial solution or solution obtained by the dissolution of a pure chemical product, concentration 0,1 gl^{-1}.

7 Apparatus

7.1 **Spectrometer** used for FAAS and ICP-AES analysis should comply with the requirements laid down in ECSC/C 9. The ICP-AES shall be equipped with a nebulization system and torch resistant to hydrofluoric acid.

7.2 **Drying oven**, capable of being controlled at (110 ± 10) ^{o}C.

7.3 **Desiccator** containing effective desiccant.

7.4 **Balance**, to read to 0,1 mg.

7.5 **Sample jar or bag**, with a seal.

7.6 **50 ml volumetric flasks**, resistant to hydrofluoric acid (e.g. polypropylene, PTFE).

7.7 **Vessels**, resistant to hydrofluoric acid, of at least 30 ml capacity, and suitable for operation at (170 ± 15) ^{o}C for 15 h, for use with (7.8).

7.8 **Acid pressure digestion apparatus**.

7.9 **Vessels**, resistant to hydrofluoric acid, for use with (7.10).

7.10 **Microwave digestion system**.

8 Sample preparation

Dry approximately 10g of the sample at (110 ± 10) ^{o}C, in the drying oven (7.2) for at least 1 h. Remove and cool it to room temperature in a desiccator (7.3). When cool store the sample in a jar or bag that is airtight (7.5).

9 Procedure

9.1 General

9.1.1 Dissolve the test sample by acid pressure digestion (9.2) or by acid pressure microwave digestion (9.3)

9.1.2 Run a blank in parallel with the chosen procedure, using identical reagents, conditions, and dilutions throughout.

9.2 Acid pressure digestion

Weigh (0,100 ± 0,000 5) g of test sample (8) into the PTFE-lined vessel (7.6).

Carefully add 3 ml of HF (6.2.1) and 1 ml of HNO_3 (6.2.2).

Install the cap and close the pressure vessel according to the manufacturers instructions.

Place the pressure vessel into the heating block and digest for 15 h at (170 ± 15) ^{o}C.
Allow to cool.

After cooling, open the pressure vessel according to the manufacturer's instructions.

Check that the test portion has been completely digested.

If not, repeat the digestion.

Transfer the dissolved sample to a 50 ml volumetric flask (7.6) and rinse the pressure vessel, and lid with distilled or de-ionised water.

Make up to the mark with distilled or de-ionised water and mix well.

9.3 Acid pressure microwave digestion

Examples of procedures:

9.3.1 Weigh 0,1g ± 0,000 5 g of test sample (8) into the vessel (7.9).

Add 5ml of HF (6.2.1) and 1 ml of HNO_3 (6.2.2) and seal according to the manufacturer's instructions.

Put the capped vessel into the microwave.

Apply the following stages:

- Stage 1: Power level 60% for 2 min;
- Stage 2: Power level 30% for 15 min;
- Stage 3: Power level 0% for 5 min;
- Stage 4: Power level 20% for 20 min.

Allow the vessel to cool in the microwave for about 20 min.

Repeat the heating cycle once more.

Inspect to see if the test sample has completely digested.

When cool, open the vessel according to the manufacturer's instructions.

Transfer the dissolved sample into a 50 ml polypropylene volumetric flask (7.6), and rinse the pressure vessel, and lid with distilled or de-ionised water.

Mix, and make up to the mark with distilled or de-ionised water.

9.3.2 Weigh 0,1 g ± 0,000 5 g of test sample (8) into the pressure vessel (7.7)

Add, carefully, 10 ml of HF (6.2.1) and 2 ml of HNO3 (6.2.2), and seal the vessel according to the manufacturers instructions.

Place the capped vessel into the microwave.

Apply the following stages:

- Stage 1: Temperature 50°C for 2 min;
- Stage 2: Temperature 150°C for 6 min;
- Stage 3: Temperature 260°C for 18 min;
- Stage 4: Temperature 0°C for 10 min.

Cool and open the vessel according to the manufacturers instructions.

Inspect the solution.

If the sample has not completely dissolved repeat stages 1 to 4 again.

Transfer the solution into a 50 ml volumetric flask (7.6), washing the pressure vessel and lid with distilled or de-ionised water.

Mix and make up to the mark with distilled or de-ionised water.

10 Calibration solutions

A calibration graph is obtained using calibration solutions prepared to span the range of concentrations of elements to be determined in the test solution. The calibration solutions are prepared using the same acids that were used to digest the test sample to ensure accurate matrix matching.

The following procedure is given as an example:

Prepare the calibration solutions by adding the volumes of solutions indicated in Table 1 to a series of 50 ml volumetric flasks (7.6). When additions are complete, mix and make the volume of each flask up to the mark with distilled water or de-ionised water.