SLOVENSKI STANDARD

SIST ENV 955-4:1998

01-april-1998

Kemijska analiza ognjevzdržnih izdelkov - 4. del: Izdelki, ki vsebujejo kremen in/ali glinico (analiza s plamensko absorpcijsko spektrometrijo (FAAS) in atomsko emisijsko spektrografijo z indukcijsko sklopitveno plazmo (ICP)

Chemical analysis of refractory products - Part 4: Products containing silica and/or alumina (Analysis by Flame Atomic Absorption Spectrometry (FAAS) and Inductively Coupled Plasma Atomic Emission Spectrography (ICP)

Chemische Analyse von feuerfesten Erzeugnissen - Teil 4: Silicium(IV)-oxid und/oder Aluminiumoxid enthaltende Erzeugnisse (Analyse mittels Flammen Atomabsorptionsspektrometrie (FAAS) und Atomabsorptionsspektrographie mit induktiv gekoppeltem Plasma (ICP))

Analyse chimique des produits réfractaires - Partie 4: Produits contenant de la silice et/ou de l'alumine (Analyse par spectrométrie d'absorption atomique dans la flamme (SAAF) et par spectrographie d'émission a plasma inductif (ICP))

Ta slovenski standard je istoveten z: **ENV 955-4:1997**

ICS:

81.080

SIST ENV 955-4:1998 **en**

EUROPEAN PRESTANDARD

PRÉNORME EUROPÉENNE

EUROPÄISCHE VORNORM

ENV 955-4

May 1997

ICS 81.080

Descriptors: refractory materials, silicon dioxide, aluminium oxide, chemical analysis, determination of content, components, atomic absorption spectrophotometry, emission spectrophotometry

English version

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CEN

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

Central Secretariat: rue de Stassart,36 B-1050 Brussels

Ref. No. ENV 955-4:1997 E

Contents

Foreword

This European Prestandard has been prepared by Technical Committee CEN/TC 187 "Refractory products and materials", the secretariat of which is held by BSI.

EN 955 'Chemical analysis of refractories' consists of four Parts:

Part 1 : Magnesites and dolomites
Part 2 : Products containing silica and/or alumina(wet method)
Part 3 : Chrome bearing products (wet method)
Part 4 : Products containing silica and/or alumina (analysis by FAAS and ICP)(ENV)
Part 5 : Analysis by XRF fused cast bead method

CEN/TC 187 approved this European pre-standard by resolution No 4 during its eighth meeting held in Paris, 96-02-23.

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

1 Scope

This European Prestandard applies to refractory materials in the alumino-silicate family. It is intended for the determination of the following constituents within the following range :

SiO_2		40 to 93 %
Al_2O_3		1 to 60 %
Fe_2O_3		0 to 10 %
TiO_2	CaO	0,01 to 5 %
MgO	K_2O	
Na_2O		

NOTE 1 : Though Fe_2O_3 contents higher than 5% are quite unusual in refractory materials, the field of application of this standard extends to 10% Fe_2O_3.

NOTE 2 : Using FAAS, K and Na are determined in emission mode.

NOTE 3 : For contents in Al_2O_3 and SiO_2 greater than 10%, it is recommended to use ICP, XRF or gravimetric methods.

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.

ISO 565	Test sieves - Woven metal wire cloth, perforated plate and electroformed sheets - nominal sizes of aperture.
ISO 1042	Laboratory glass-ware - One-mark volumetric flask.
ISO 13527	Chemical analysis of ferrous materials - Guidelines on the use of inductively coupled plasma atomic emission spectroscopy.
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.

NOTE : As an alternative to test sieves, a disposable nylon bolting cloth can be used.

3 Principle

- Decomposition of the sample by fusion;
- Dissolution of the product of the fusion in diluted acid;
- Addition of lanthanum chloride, only in the case of analysis by FAAS;
- Dilution of the solution to a defined volume and nebulization into a flame for FAAS, or into a plasma, for ICP;
- For each element, comparison of the emitted intensity for a determined analytical peak with those emitted by a range of reference solutions of known concentrations.

4 Sampling

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

5 Reagents

All reagents, and substances, shall be pure, of analytical grade. Distilled, or de-ionised water, shall be used throughout the whole procedure.

5.1 Reagents for preparation

5.1.1 Lithium tetraborate $Li_2B_4O_7$. = flux 1.
5.1.2 Anhydrous sodium carbonate Na_2CO_3 (12 parts) + boric acid H_3BO_3 (2 parts) = flux 2.
5.1.3 Hydrochloric acid d = 1,19 (1 + 1, volume).
5.1.4 Solution for dilution D : 4 g of lithium tetraborate + 40 ml of hydrochloric acid (1 + 1)/l solution
5.1.5 Hydrated lanthanum chloride $LaCl_3 6H_2O$, 200 g/l solution.
5.1.6 Potassium chloride KCl 2 % in water.
5.1.7 Anhydrous potassium carbonate : K_2CO_3
5.1.8 Internal standard : to be defined in the test report.

5.2 Reagents for calibration

All reagents must be of analytical grade.

Silica	SiO_2 99,99% ignited at 1200 °C ± 5 °C
Aluminium	Al
Iron	Fe
Titanium	Ti
Calcium carbonate	$CaCO_3$ dried to 220°C ± 20°C
Magnesium	Mg or MgO
Potassium carbonate	K_2CO_3 dried to 220°C ± 20°C
Sodium carbonate	Na_2CO_3 dried to 220°C ± 20°C

6 Apparatus

The spectrometers used for FAAS- and ICP-analysis should comply with the requirements laid down by E CSC/C19 and ISO 13527, respectively.

7 Equipment

7.1 Drying oven, capable of being controlled at 110°C ± 10°C.
7.2 Grinder, with a tungsten or boron carbide mill.
7.3 Test sieve, complying with the requirements of ISO 565 or the alternative nylon bolting cloth.
7.4 Desiccator.
7.5 Non-wettable platinum alloy crucibles (e.g. approximately 35 g, diameter 45 mm, height 40 mm).
7.6 Balance, to read to 0,1 mg.
7.7 Furnace, capable of being heated up to 1250 °C.
7.8 Beakers, 500 ml, 250 ml preferably PTFE.
7.9 Magnetic stirrer, with PTFE-protected stirring bar.
7.10 Tongs, to hold crucibles.

NOTE : Platinum tipped tongs should be used if the tongs are in contact with the interior of the crucible.

7.11 Volumetric flasks, borosilicate glass, 1000, 500, 250 and 100 ml, grade B or better, complying with the requirements of ISO 1042.
7.12 Beakers, borosilicate glass, 600 ml.

8 Sample preparation

Dry the wet sample at 110 °C ± 10°C, in the drying oven (7.1) for 4 h at least, prior to grinding. Grind it in the mill (7.2) in such a manner to obtain a sample the grain size of which is less than 63 μm (7.3). Dry it again at 110 °C ± 10°C for 1 hour, at least, and cool it to room temperature in a desiccator (7.4).

9 Procedure

9.1 Fusion

9.1.1 Fusion with lithium tetraborate (flux 1)

In a platinum alloy crucible (7.5) previously ignited and cooled down in the desiccator (7.4), weigh a test portion of 0,2 g ± 0,0005 g. Add 2 g ± 0,001 g of flux 1 (5.1.1). Mix carefully. Put the crucible and its content into a furnace heated at 1150 °C ± 50 °C (7.7). Allow it to react until it decomposes (normally 25 min is sufficient).

Pour the molten fusion into a beaker (7.8) filled with approximately 350 ml water containing 20 ml HCl (5.1.3), the whole being continuously stirred on the magnetic stirrer (7.9).

Immerse the crucible in the mixture to ensure full dissolution of the fused residue. Stir until dissolved. Remove the crucible and rinse it twice or thrice with water. Collect the rinsing water in the beaker.

9.1.2 Fusion with sodium carbonate and boric acid (flux 2)

Weigh 0,5g ± 0,005 g of sample, dried at 110°C ± 10°C and cooled in a dessicator, into the platinum alloy crucible (7.5).
Add 1,4 g of flux 2, mix well. Put crucible into a muffle furnace at 1200°C ± 50°C for 25 min. Remove crucible and allow to cool. Add 1,0 g of anhydrous potassium carbonate (5.1.7), no mixing is required. Put the crucible back in the furnace for a further 5 min. Remove and cool. Place the crucible and lid into a 250 ml beaker (7.8) containing 100 ml of hydrochloric acid (5.1.3), cover with a watch glass, heat with continuous agitation until dissolution is complete, do not boil.

9.2 Preparation

9.2.1 FAAS-analysis (fusion of the sample with flux 1).

Transfer the solution into a 500 ml volumetric flask (7.11). Add 20 ml of lanthanum chloride (5.1.5) Fill to the mark with water and mix.

9.2.2 FAAS analysis (fusion of the sample with flux 2)

Transfer the dissolved sample to a 250 ml volumetric flask and rinse crucible, lid, beaker well with distilled water. Make up to the mark and mix. Take a 10 ml aliquot and transfer to a 100 ml volumetric flask, dilute to the mark and mix.