

**Testing of ceramic and basic materials - Direct determination of mass fractions of impurities in powders and granules of silicon carbide by inductively coupled plasma optical emission spectrometry (ICP OES) with electrothermal vaporisation (ETV)**

## EESTI STANDARDI EESSÕNA

## NATIONAL FOREWORD

Käesolev Eesti standard EVS-EN 15991:2011 sisaldab Euroopa standardi EN 15991:2011 ingliskeelset teksti.

Standard on kinnitatud Eesti Standardikeskuse 28.02.2011 käskkirjaga ja jõustub sellekohase teate avaldamisel EVS Teatajas.

Euroopa standardimisorganisatsioonide poolt rahvuslikele liikmetele Euroopa standardi teksti kättesaadavaks tegemise kuupäev on 26.01.2011.

Standard on kättesaadav Eesti standardiorganisatsioonist.

This Estonian standard EVS-EN 15991:2011 consists of the English text of the European standard EN 15991:2011.

This standard is ratified with the order of Estonian Centre for Standardisation dated 28.02.2011 and is endorsed with the notification published in the official bulletin of the Estonian national standardisation organisation.

Date of Availability of the European standard text 26.01.2011.

The standard is available from Estonian standardisation organisation.

ICS 81.060.10

### Standardite reprodutseerimis- ja levitamiseõigus kuulub Eesti Standardikeskusele

Andmete paljundamine, taastekitamine, kopeerimine, salvestamine elektroonilisse süsteemi või edastamine ükskõik millises vormis või millisel teel on keelatud ilma Eesti Standardikeskuse poolt antud kirjaliku loata.

Kui Teil on küsimusi standardite autorikaitse kohta, palun võtke ühendust Eesti Standardikeskusega:  
Aru 10 Tallinn 10317 Eesti; [www.evs.ee](http://www.evs.ee); Telefon: 605 5050; E-post: [info@evs.ee](mailto:info@evs.ee)

### Right to reproduce and distribute belongs to the Estonian Centre for Standardisation

No part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying, without permission in writing from Estonian Centre for Standardisation.

If you have any questions about standards copyright, please contact Estonian Centre for Standardisation:  
Aru str 10 Tallinn 10317 Estonia; [www.evs.ee](http://www.evs.ee); Phone: 605 5050; E-mail: [info@evs.ee](mailto:info@evs.ee)

ICS 81.060.10

English Version

Testing of ceramic and basic materials - Direct determination of mass fractions of impurities in powders and granules of silicon carbide by inductively coupled plasma optical emission spectrometry (ICP OES) with electrothermal vaporisation (ETV)

Essais sur matériaux céramiques et basiques - Détermination directe des fractions massiques d'impuretés dans les poudres et les granulés de carbure de silicium par spectroscopie d'émission optique à plasma induit par haute fréquence (ICP OES) avec vaporisation électrothermique (ETV)

Prüfung keramischer Roh- und Werkstoffe - Direkte Bestimmung der Massenanteile von Spurenverunreinigungen in pulver- und körnigem Siliciumcarbid mittels optischer Emissionsspektrometrie mit induktiv gekoppeltem Plasma (ICP OES) und elektrothermischer Verdampfung (ETV)

This European Standard was approved by CEN on 10 December 2010.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

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-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, 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.



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

Management Centre: Avenue Marnix 17, B-1000 Brussels

**Contents**

Page

|  |    |
|--|----|
| Foreword.....  | 3  |
| 1 Scope .....  | 4  |
| 2 Principle .....  | 4  |
| 3 Spectrometry .....   | 4  |
| 4 Apparatus .....  | 6  |
| 5 Reagents and auxiliary material.....   | 7  |
| 6 Sampling and sample preparation .....  | 7  |
| 7 Calibration .....  | 7  |
| 8 Procedure .....  | 8  |
| 9 Wavelength and working range.....  | 9  |
| 10 Calculation of the results and evaluation .....   | 9  |
| 11 Reporting of results.....   | 9  |
| 12 Precision.....  | 10 |
| 13 Test report .....   | 10 |
| Annex A (informative) Results of interlaboratory study.....  | 11 |
| Annex B (informative) Wavelength and working range.....  | 16 |
| Annex C (informative) Possible interferences and their elimination .....   | 18 |
| Annex D (informative) Information regarding the evaluation of the uncertainty of the mean value .....  | 21 |
| Annex E (informative) Commercial certified reference materials .....   | 22 |
| Annex F (informative) Information regarding the validation of an analytical method based on liquid standards at the example of SiC and graphite..... | 23 |
| Bibliography.....  | 26 |

## Foreword

This document (EN 15991:2011) has been prepared by Technical Committee CEN/TC 187 “Refractory products and materials”, 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 July 2011, and conflicting national standards shall be withdrawn at the latest by July 2011.

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.

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, Croatia, 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.

This document is a preview generated by EVS

## 1 Scope

This European Standard defines a method for the determination of the trace element concentrations of Al, Ca, Cr, Cu, Fe, Mg, Ni, Ti, V and Zr in powdered and granular silicon carbide.

Dependent on element, wavelength, plasma conditions and weight, this test method is applicable for mass contents of the above trace contaminations from about 0,1 mg/kg to about 1 000 mg/kg, after evaluation also from 0,001 mg/kg to about 5 000 mg/kg.

NOTE 1 Generally for optical emission spectrometry using inductively coupled plasma (ICP OES) and electrothermal vaporisation (ETV) there is a linear working range of up to four orders of magnitude. This range can be expanded for the respective elements by variation of the weight or by choosing lines with different sensitivity.

After adequate verification, the standard is also applicable to further metallic elements (excepting Rb and Cs) and some non-metallic contaminations (like P and S) and other allied non-metallic powdered or granular materials like carbides, nitrides, graphite, soot, coke, coal, and some other oxidic materials (see [1], [4], [5], [6], [7], [8], [9] and [10]).

NOTE 2 There is positive experience with materials like for example graphite, B<sub>4</sub>C, Si<sub>3</sub>N<sub>4</sub>, BN and several metal oxides as well as with the determination of P and S in some of these materials.

## 2 Principle

The sample material, crushed if necessary, is evaporated in an argon- carrier-gas stream in a graphite boat in the graphite tube furnace of the ETV unit. The evaporation products containing the element traces are transported as a dry aerosol into the plasma of the ICP-torch and there excited for the emission of optical radiation. In a simultaneous emission spectrometer in, for example Paschen-Runge- or Echelle-configuration, the optical radiation is dispersed. The intensities of suited spectral lines or background positions are registered with applicable detectors like photomultipliers (PMT), charge coupled devices (CCD), charge injection devices (CID), and serial coupled devices (SCD). By comparison of the intensities of the element-specific spectral lines of the sample with calibration samples of known composition, the mass fractions of the sample elements are determined.

## 3 Spectrometry

Optical emission spectrometry is based on the generation of line spectra of excited atoms or ions, where each spectral line is associated with an element and the line intensities are proportional to the mass fractions of the elements in the analysed sample.

In contrary to the wet chemical analysis from dilution in ICP OES the classical sample digestion is replaced by electrothermal vaporisation at high temperatures in a graphite furnace.

By a suitable design of the furnace (see Figures 1 and 2) and a suited gas regime in the transition area graphite tube / transport tube (see Figure 1), it is ensured that the sample vapour is carried over into a form that is to transport effectively (see [5], [6], [7], [8], [10]). Carbide forming elements, for example titanium, zirconium, that are not or incompletely evaporating need a suited modifier, like a halogenation agent, preferably dichlorodifluoromethane, to be converted into a form that is easy to transport (see [1], [3], [5] and [10].)

The dry aerosol is introduced into the ICP plasma by the injector tube and there excited for the emission of light (see Figures 1 to 3).