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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

See Eesti standard EVS-EN 15991:2015 sisaldab Euroopa standardi EN 15991:2015 ingliskeelset teksti.	This Estonian standard EVS-EN 15991:2015 consists of the English text of the European standard EN 15991:2015.
Standard on jõustunud sellekohase teate avaldamisega EVS Teatajas	This standard has been endorsed with a notification published in the official bulletin of the Estonian Centre for Standardisation.
Euroopa standardimisorganisatsioonid on teinud Euroopa standardi rahvuslikele liikmetele kättesaadavaks 25.11.2015.	Date of Availability of the European standard is 25.11.2015.
Standard on kättesaadav Eesti Standardikeskusest.	The standard is available from the Estonian Centre for Standardisation.

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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 granules 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 kornförmigem  
Siliciumcarbid mittels optischer  
Emissionsspektroskopie mit induktiv gekoppeltem  
Plasma (ICP OES) und elektrothermischer  
Verdampfung (ETV)

This European Standard was approved by CEN on 3 October 2015.

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.

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

**CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels**

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## European foreword

This document (EN 15991:2015) 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 May 2016 and conflicting national standards shall be withdrawn at the latest by May 2016.

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 EN 15991:2011.

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## 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 vaporization (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.

Contrary to the wet chemical analysis from dilution in ICP OES the classical sample digestion is replaced by electrothermal vaporization 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 incompletely or not evaporating need a suitable reaction gas (halogenating agent) to be converted into a form that is easy to transport (see [1], [3], [5] and [10].) Dichlorodifluoromethane (CCl<sub>2</sub>F<sub>2</sub>) shall be used as halogenating agent. Compared to other halogen containing carbon compounds CCl<sub>2</sub>F<sub>2</sub> provides optimum analyte release and transport efficiency. CCl<sub>2</sub>F<sub>2</sub> is required for simultaneous determination of the elements listed in Clause 1. The results of the interlaboratory study (see Annex A) were obtained using CCl<sub>2</sub>F<sub>2</sub> as reaction gas.

The dry aerosol is introduced into the ICP plasma by the injector tube and there excited for the emission of light (see Figure 1, Figure 2 and Figure 3).