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

EESTI STANDARDIMIS- JA AKREDITEERIMISKESKUS
ESTONIAN CENTRE FOR STANDARDISATION AND ACCREDITATION

EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

See Eesti standard EVS-EN 15991:2025 sisaldab Euroopa standardi EN 15991:2025 ingliskeelset teksti.	This Estonian standard EVS-EN 15991:2025 consists of the English text of the European standard EN 15991:2025.
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 and Accreditation.
Euroopa standardimisorganisatsioonid on teinud Euroopa standardi rahvuslikele liikmetele kättesaadavaks 24.09.2025.	Date of Availability of the European standard is 24.09.2025.
Standard on kättesaadav Eesti Standardimis-ja Akrediteerimiskeskusest.	The standard is available from the Estonian Centre for Standardisation and Accreditation.

Tagasisidet standardi sisu kohta on võimalik edastada, kasutades EVS-i veebilehel asuvat tagasiside vormi või saates e-kirja meiliaadressile standardiosakond@evs.ee.

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EUROPEAN STANDARD

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English Version

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

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

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

This European Standard was approved by CEN on 27 July 2025.

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

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Contents

Page

European foreword

This document (EN 15991:2025) 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 March 2026, and conflicting national standards shall be withdrawn at the latest by March 2026.

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

This document supersedes EN 15991:2015.

EN 15991:2025 includes the following significant technical changes with respect to EN 15991:2015:

— Clause 2 and Clause 3 have been added, noting that they neither add any normative references nor terms and definitions to the document;

— Clause 4 adds significantly more detail about the analysis and process;

— Clause 5 provides additional information on the spectrometry methodology;

— Annex B, Table B.1 provides additional information on the limits of determination.

Any feedback and questions on this document should be directed to the users’ national standards body. A complete listing of these bodies can be found on the CEN website.

According to the CEN-CENELEC Internal Regulations, the national standards organisations 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, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Türkiye and the United Kingdom.

1 Scope

This document specifies a method for the determination of the mass fractions of the elements Al, Ca, Cr, Cu, Fe, Mg, Ni, Ti, V and Zr in powdered and granular silicon carbide.

Dependent on element, emission lines, plasma conditions and sample mass, this test method is applicable for mass fractions 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 and electrothermal vaporization (ETV-ICP-OES) 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 sample mass or by choosing emission lines with different sensitivity.

After adequate verification, this document 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, boron carbide (B_4C), silicon nitride (Si_3N_4), boron nitride (BN) and several metal oxides as well as with the determination of P and S in some of these materials.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Principle

For the determination of impurities in silicon carbide, ICP-OES is a suited method. The classic application of ICP-OES is based on the nebulization of sample solutions. For silicon carbide, sample digestion by wet-chemical methods is required to obtain these sample solutions, for example by melt-fusion or acid/pressure-decomposition. These sample digestion procedures are time-consuming, require the use of hazardous chemicals, deteriorate the detection limits due to the dilution of the sample and the possibility of introduction of impurities as well as analyte losses represents a source of systematic errors.

With ETV-ICP-OES, the impurities are measured directly from the powdered silicon carbide sample, thus avoiding sample digestion and the associated disadvantages. Compared to wet-chemical ICP-OES methods, ETV-ICP-OES requires more effort for method development and is therefore particularly suitable when many samples of one matrix are to be measured with high sample throughput and with high detection sensitivity.

In ETV-ICP-OES, sample introduction by nebulization of liquids is replaced by the electrothermal vaporization of solid samples at high temperatures in the graphite tube furnace of the ETV-system.