



NSAI
Standards

Irish Standard
I.S. EN 15991:2015

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)

I.S. EN 15991:2015

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I.S. xxx: Irish Standard — national specification based on the consensus of an expert panel and subject to public consultation.

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NSAI
1 Swift Square,
Northwood, Santry
Dublin 9

T +353 1 807 3800
F +353 1 807 3838
E standards@nsai.ie
W NSAI.ie

Sales:
T +353 1 857 6730
F +353 1 857 6729
W standards.ie

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National Foreword

I.S. EN 15991:2015 is the adopted Irish version of the European Document EN 15991:2015, 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)

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EUROPEAN STANDARD

EN 15991

NORME EUROPÉENNE

EUROPÄISCHE NORM

November 2015

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Supersedes EN 15991:2011

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 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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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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EN 15991:2015 (E)

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, B₄C, Si₃N₄, 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 (CCl₂F₂) shall be used as halogenating agent. Compared to other halogen containing carbon compounds CCl₂F₂ provides optimum analyte release and transport efficiency. CCl₂F₂ is required for simultaneous determination of the elements listed in Clause 1. The results of the interlaboratory study (see Annex A) were obtained using CCl₂F₂ 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).

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