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BSI Standards Publication

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)

National foreword

This British Standard is the UK implementation of EN 15991:2015. It supersedes BS EN 15991:2011 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee RPI/1, Refractory products and materials.

A list of organizations represented on this committee can be obtained on request to its secretary.

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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 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.

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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of an identical text or by endorsement, at the latest by May 2016 and conflicting national standards shall be withdrawn at the latest by May 2016.

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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_4C, Si_3N_4, 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_2F_2) shall be used as halogenating agent. Compared to other halogen containing carbon compounds CCl_2F_2 provides optimum analyte release and transport efficiency. CCl_2F_2 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_2F_2 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).