Testing of ceramic raw and basic materials - Direct determination of mass fractions of impurities in powders and granules of silicon carbide by OES by DC arc excitation

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NATIONAL FOREWORD

Käesolev Eesti standard EVS-EN 15979:2011 sisaldab Euroopa standardi EN 15979:2011 ingliskeelset teksti.	This Estonian standard EVS-EN 15979:2011 consists of the English text of the European standard EN 15979:2011.
Standard on kinnitatud Eesti Standardikeskuse 28.02.2011 käskkirjaga ja jõustub sellekohase teate avaldamisel EVS Teatajas.	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.
Euroopa standardimisorganisatsioonide poolt rahvuslikele liikmetele Euroopa standardi teksti kättesaadavaks tegemise kuupäev on 26.01.2011.	Date of Availability of the European standard text 26.01.2011.
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English Version

Testing of ceramic raw and basic materials - Direct determination of mass fractions of impurities in powders and granules of silicon carbide by OES by DC arc excitation

Essai des matières premières et matériaux de base céramiques - Détermination directe des fractions massiques d'impuretés dans les poudres et granulés de carbure de silicium par OES à l'excitation d'arc DC

Prüfung keramischer Roh- und Werkstoffe - Direkte Bestimmung der Massenanteile an Verunreinigungen in pulver- und kornförmigem Siliciumcarbid mittels OES und Anregung im Gleichstrombogen

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

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

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Foreword

This document (EN 15979:2011) 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of an identical text or by endorsement, at the latest by July 2011, and conflicting national standards shall be withdrawn at the latest by July 2011.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.

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1 Scope

This European Standard describes the method for the analysis of mass fractions of the impurities Al, B, Ca, Cr, Cu, Fe, Mg, Ni, Ti, V and Zr in powder- and grain-shaped silicon carbide of ceramic raw and basic materials. This application can also be extended to other metallic elements and other similar non-metallic powder- and grain-shaped materials such as carbides, nitrides, graphite, carbon blacks, cokes, carbon, as well as a number of further oxidic raw and basic materials after appropriate testing.

NOTE There are positive interferences for materials such as e.g. graphite, B_4C, BN, WC and several refractory metal oxides.

This testing procedure is applicable to mass fractions of the impurities mentioned above from approximately 1 mg/kg up to approximately 3 000 mg/kg, after verification. In some cases it may be possible to extend the range up to 5 000 mg/kg depending on element, wavelength, arc parameter, and sample weight.

2 Principle

The combustion and evaporation of the crushed sample material takes place in the arc in an atmosphere of mixed argon and oxygen or in air. The metallic traces in the arc plasma are excited to emission of light. The light is guided into a simultaneous emission spectrometer (e.g. by coupling via fibre-optics or directly). The light is split in its spectral lines and measured by applicable detectors like a photomultiplier, charge coupled device (CCD), and charge injection device (CID). The mass fractions of elements in the sample are calculated by comparison of the intensities of the element-specific spectral line with those of a calibration sample of identical material.

3 Spectrometry

The optical emission spectrometry is based on generation of line spectra of excited atoms or ions, in which each spectral line can be definitely related to an element and the line intensities are proportional to the mass fractions of elements in the measured sample (see [6], [7] & [12]).

Contrary to wet-chemical methods via solution the classical sample decomposition is replaced by evaporation end excitation in DC-Arc.

4 Apparatus

Ordinary laboratory apparatus and the following:

4.1 Emission spectrometer, simultaneous, preferably with time resolved registration of signal, and connected to DC-Arc-equipment.

4.2 Gas flushing device, for shielding gas and/or reaction gas of DC-Arc, e.g. gas-mixer with mass-flow controller and Stallwood-jet

NOTE When working with air, the shielding gas unit can be omitted.

4.3 Tweezers, self-locking.

4.4 Balance, an analytical balance at least capable of reading to the nearest 0,1mg. However, for small weight <10 mg, a five figure balance at least capable of reading to the nearest 0,01 mg shall be used.

4.5 Pressing tool, for compacting the sample into the electrode.