



BSI Standards Publication

Testing of ceramic raw materials and ceramic materials — Direct determination of mass fractions of impurities in powders and granules of silicon carbide by optical emission spectrometry by direct current arc excitation (DCArc-OES)

National foreword

This British Standard is the UK implementation of EN 15979:2025. It supersedes BS EN 15979:2011, which is withdrawn.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 committee manager.

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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 optical
emission spectrometry by direct current arc excitation
(DCArc-OES)

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
(DCArc-OES)

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

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

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

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European foreword

This document (EN 15979:2025) 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of an identical text or by endorsement, at the latest by March 2026, and conflicting national standards shall be withdrawn at the latest by March 2026.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 15979:2011.

EN 15979:2025 includes the following significant technical changes with respect to EN 15979:2011:

- Clause 2 and Clause 3 have been added, noting that they neither add any normative references nor terms and definitions to the document;
- Clause 9 adds more detail about calculation of the calibration functions;
- subclause 10.2 adds more detail about the procedure using addition of carrier;
- Clause 14 now provides the completeness of the information required by CEN/CENELEC Internal Regulations Part 3;
- Annex A, Tables A.1 to A.4 now provides the correct printing of variables required by CEN/CENELEC Internal Regulations Part 3;
- 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 describes a method for the analysis of mass fractions of the impurities Al, B, Ca, Cr, Cu, Fe, Mg, Ni, Ti, V and Zr in powdered and grain-shaped silicon carbide of ceramic raw materials and ceramic materials. This application can also be extended to other metallic elements and other similar non-metallic powdered 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 is positive experience with materials such as, for example, graphite, boron carbide (B_4C), boron nitride (BN), tungsten carbide (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 is possible to extend the range up to 5 000 mg/kg depending on element, emission lines, DC arc parameters, and sample mass.

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

With DC arc-OES, the impurities are measured directly from the powdered silicon carbide sample, thus avoiding the disadvantages of the usually applied wet-chemical digestion of silicon carbide such as high time-consumption, the use of hazardous chemicals, dilution of the sample and the possibility of systematic errors due to introduction of impurities as well as analyte losses. Compared to wet-chemical ICP-OES methods, DC arc-OES requires more effort for method development and is therefore particularly suitable when many samples of one matrix are to be measured. With DC arc-OES, impurities in silicon carbide can be measured cost-effectively, with high sample throughput and with a detection sensitivity down to the lower mg/kg level.

In DC arc-OES, the powdered silicon carbide sample is evaporated and excited in the direct current arc-plasma of the DC arc-system. The combustion and evaporation of the powdered sample material takes place in the arc-plasma in an atmosphere of oxygen, mixed argon and oxygen or in air. The metallic traces in the silicon carbide sample are excited in the arc-plasma to emission of optical radiation. The optical radiation from the DC arc-system is guided into a simultaneous emission spectrometer by coupling via fibre-optics or directly. If the coupling takes place via fibre-optics, the simultaneous emission spectrometer can be a commercially available ICP-OES device, provided that the measurement can be started with the plasma switched off.

NOTE As the fibre-optics used for coupling is usually made of quartz, emission lines with wavelengths of less than around 220 nm can no longer be measured due to absorption.

Literature on DC arc-OES see [1], [3], [4], [5], [6], [8], [9] and [10].

5 Spectrometry

Optical emission spectrometry is based on the generation of line spectra of excited atoms or ions, where each emission line is associated with an element and the line intensities are proportional to the mass fractions of the elements in the analysed sample (see [6], [7] and [12]).

In a simultaneous emission spectrometer in, for example Paschen-Runge- or Echelle-configuration, the optical radiation is dispersed. The intensities of suited emission lines or background positions are registered with applicable detectors like photomultipliers (PMT), charge coupled devices (CCD), complementary metal-oxide semiconductor (CMOS), charge injection devices (CID), and serial coupled devices (SCD). By comparison of the intensities of the element-specific emission lines of the sample with calibration samples of known composition, the mass fractions of the trace elements in the sample are determined.

6 Apparatus

6.1 Common laboratory equipment, and the following:

6.2 Emission spectrometer, simultaneous, with the possibility to register transient emission signals with a sampling rate of at least 5 Hz, suitable for the synchronised start of the DCArc-program and the registration of the emission signals and possibility for coupling to the DCArc-system.

NOTE 1 If the coupling of the DCArc-system to the emission spectrometer takes place via fibre-optics, the simultaneous emission spectrometer can be a commercially available ICP-OES device, provided that the measurement can be started with the plasma switched off.

6.3 DCArc-system, with Stallwood jet, controlled gas-flows for argon and oxygen, preferably with mass-flow control, and controlled setting of arc-current, preferably with the possibility of programmable current vs. time programs with freely definable ramp- and hold-steps.

NOTE 2 When working with air, Stallwood jet and gas-flow controllers can be omitted.

6.4 Tweezers, self-closing.

6.5 Analytical balance, with a resolution of at least 0,01 mg.

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

6.7 Drying cabinet, adjustable to $(60 \pm 5) ^\circ\text{C}$.

NOTE 3 A drying cabinet will only be required if wet mixed and subsequently dried samples are used.

6.8 Stirring balls made out of polytetrafluoroethylene (PTFE), diameter 6 mm, for example.

NOTE 4 Stirring balls will be required if wet mixed and subsequently dried samples are used.

6.9 Plastic vessel, with screw cap, volume 25 ml, made of, for example, polyethylene (PE) or polypropylene (PP).

NOTE 5 Plastic vessels will be required if mixed and subsequently dried samples are used.

6.10 High resistance carbon electrodes or graphite electrodes, spectral-grade, peak-shaped or elliptical counter electrode (cathode), cup-shaped carrier electrode (anode) with groove or taper.

7 Reagents

Only analytical grade reagents shall be used unless stated otherwise.

7.1 Calibration samples with well-defined mass fractions of trace-impurities, preferably certified reference materials (CRM).

NOTE Certified reference materials are available for main-, minor- and trace components (see Annex E).

7.2 Oxygen, purity $\geq 99,99$ % (volume fraction).

7.3 Argon, purity $\geq 99,99$ % (volume fraction).

7.4 Dichloromethane, CH_2Cl_2 .

NOTE Dichloromethane will be required if wet mixed and subsequently dried samples are used.

8 Sampling and sample preparation

Sampling shall be performed in a way that the sample to be analysed is representative for the total amount of material.

NOTE 1 For example, according to ISO 5022 [13], ISO 8656-1 [14] or ISO 21068-1 [15].

If the sample is not received in a dry state, it shall be dried at (110 ± 10) °C until constant mass is achieved ($<0,5$ % variation). The sample is then cooled down to room temperature and stored in a desiccator.

NOTE 2 Drying for 2 h is normally sufficient.

The particle size of sample material shall be ≤ 150 μm (100 mesh). If necessary, the sample shall be crushed, milled and homogenized. Regarding possible contamination, the material of the equipment used shall be suited for the analytical task.

9 Calibration

Calibration shall be carried out for each measuring cycle with calibration samples (7.1) of defined mass fractions of traces-impurities in accordance with Clause 10. Calibration samples shall be measured at the beginning and at the end of the measuring cycle. Within the calibration range at least a three-point calibration shall be performed (as shown, for example, in [4] and [8]).

Calibration samples (7.1) of identical or similar material as the unknown sample, if possible certified reference materials or matrix matched synthetic calibration samples, shall be used. The mass fractions of trace-impurities in the calibration samples should be within in the range of those of the sample material.

The calibration function for each emission line shall be calculated using the analyte masses of the calibration samples used and the net-intensities of the emission line measured for these analyte masses. A monotonically increasing slope of the calibration functions with a sufficiently high gradient is essential.

NOTE The calculation of the calibration functions is usually carried out as linear regression. If necessary, a quadratic regression can also be used.