



**SLOVENSKI STANDARD**  
**oSIST prEN 15979:2024**  
**01-september-2024**

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**Preskušanje keramičnih surovin in keramičnih materialov - Neposredno določevanje masnih frakcij nečistoč v prahu in zrnih silicijevega karbida z optično emisijsko spektrometrijo z vzbujanjem oblaka z enosmernim tokom (DCArc-OES)**

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)

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

Essai des matières premières céramiques et des matériaux céramiques - Détermination directe des fractions massiques d'impuretés dans les poudres et granulés de carbure de silicium par spectrométrie d'émission optique à excitation par arc de courant continu (DCArc-OES)

**Ta slovenski standard je istoveten z: prEN 15979**

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NORME EUROPÉENNE  
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**DRAFT**  
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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  
(DCArc-OES)**

Essai des matières premières et matériaux de base  
céramiques - Détermination directe des fractions  
massiques d'impuretés dans les poudres et granules 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  
optischer Emissionsspektrometrie und Anregung im  
Gleichstrombogen (DCArc-OES)

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 187.

If this draft becomes a European Standard, 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.

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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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

**CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels**

<b>Contents</b>	<b>Page</b>
European Foreword.....	4
1 Scope.....	5
2 Normative references.....	5
3 Terms and definitions .....	5
4 Principle .....	5
5 Spectrometry .....	6
6 Apparatus.....	6
7 Reagents .....	7
8 Sampling and sample preparation.....	7
9 Calibration .....	7
10 Procedure.....	8
11 Calculation .....	10
12 Expression of results.....	10
13 Precision.....	10
14 Test report.....	10
Annex A (informative) Results of interlaboratory study .....	12
Annex B (informative) Emission lines and working range .....	16
Annex C (informative) Possible interferences and their elimination .....	18
C.1 General.....	18
C.2 Spectral interferences .....	18
C.2.1 Line coincidences .....	18
C.2.2 Band coincidences .....	18
C.2.3 Background influences.....	19
C.2.4 Line reversal, self-absorption.....	19
C.2.5 Unspecific radiation .....	19
C.3 Non spectral interferences .....	19
C.3.1 Interferences by the physical characteristics of the sample .....	19
C.3.2 Interferences by depositions .....	19
C.3.3 Ionization interferences .....	19
C.3.4 Change of electric arc-plasma.....	20
C.3.5 Conclusion.....	20

<b>Annex D (informative) Information regarding the evaluation of the uncertainty of the mean value .....</b>	<b>21</b>
<b>Annex E (informative) Commercial certified reference materials .....</b>	<b>22</b>
<b>Bibliography .....</b>	<b>23</b>

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**prEN 15979:2024 (E)**

## **European foreword**

This document (prEN 15979:2024) has been prepared by Technical Committee CEN/TC 187 “Refractory products and materials”, the secretariat of which is held by BSI.

This document is currently submitted to the CEN Enquiry.

This document will supersede EN 15979:2011.

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## 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.

## 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-effective, 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] & [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  $(60 \pm 5)$  °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**,  $\text{CH}_2\text{Cl}_2$ .

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

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

NOTE Drying for 2 h is normally sufficient.

The particle size of sample material shall be  $\leq 150$   $\mu\text{m}$  (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 (see [4], [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 whereby for the graphical representation of the calibration functions the analyte masses are plotted on the x-axis and the measured net-intensities of the emission lines on the y-axis. If necessary, a quadratic regression can also be used.

## 10 Procedure

### 10.1 Standard procedure

The sample prepared in accordance with Clause 8 is filled into the carrier electrode. One of the three following procedures shall be applied:

- a) The sample material is filled into the carrier electrode using a small, precisely tailored funnel and applying mechanical compression;
- b) The carrier electrode is filled by repeatedly pressing the cup (orifice downwards) onto the sample material which is lying on a clean carrier (for example filter paper);
- c) A sub-sample of the sample material is weighed to the nearest 0,01 mg into the carrier electrode in a defined narrow weighing range, for example 4,5 mg - 5,5 mg. The mass of the weighed sub-sample has to be documented. Subsequently, the sub-sample has to be compacted in the cup of the carrier electrode by slightly striking it on a rigid underlay or by knocking with a spatula at the tweezers holding the carrier electrode.

Depending on dimension and shape of the carrier electrode the mass of the sub-sample can vary. In case of trace-impurities with mass fractions above the calibrated range, the sample mass can be reduced to a minimum of approximately 1 mg. In this case, a material of the same type, which does not contain the elements to be measured, has to be added to the carrier electrode and mixed with the sub-sample. The total mass of material in the carrier electrode shall correspond to that of the calibration sample (7.1). Instead of a pure material of the same matrix, spectral-grade graphite powder can be used.

To avoid contamination, the tweezers (6.4) shall only be used in the clamping area of the carrier electrode of the electrode holder of the DC-Arc system.

The carrier electrode has to be fixed in the optical path using the electrode holder of the DC-Arc-system. The distance to the upper counter electrode (cathode) has to be adjusted to the nearest 0,1 mm in a range of 3,5 mm to 4,0 mm.

NOTE 1 The distance between the electrodes can vary according to the diameter of the electrodes.

The position of the electrodes, and thus the arc discharge, has to be constant with respect to the optical axis of the optical system. Any change of the optical adjustment will lead to different results. Parts of the electrodes shall not be visible to the emission spectrometer. This is especially true for the upper electrode (cathode) whereas the lower electrode (anode), because of the high burn-off rate, normally remains a significantly shorter time in the optical path.

NOTE 2 Electrodes visible in the optical path result in a strong enhancement of the spectral background in some spectral ranges.

The arc discharge has to be started at the same time as the data acquisition of the emission spectrometer (6.2).

The evaporation or combustion of the sample in the DC-Arc has to be carried out preferably under shielding gas excluding any nitrogen. The mixing ratio of the shielding gas is about 70 parts by volume argon and 30 parts by volume oxygen at a constant gas flow of about  $(4 \pm 1)$  l/min.

The combustion can also be performed in oxygen or in air. If air is used, one shall pay attention to spectral interferences, for example CN-bands. In addition, a negative influence on the reproducibility can be expected.

**WARNING** — It is not safe to look into the arc plasma without eye protection (UV- and IR-radiation). Reflections on reflective areas can be dangerous too.