



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 inductively coupled plasma optical emission spectrometry with electrothermal vaporisation (ETV-ICP-OES)

National foreword

This British Standard is the UK implementation of EN 15991:2025. It supersedes BS EN 15991:2015, which is withdrawn.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.

Contractual and legal considerations

This publication has been prepared in good faith, however no representation, warranty, assurance or undertaking (express or implied) is or will be made, and no responsibility or liability is or will be accepted by BSI in relation to the adequacy, accuracy, completeness or reasonableness of this publication. All and any such responsibility and liability is expressly disclaimed to the full extent permitted by the law.

This publication is provided as is, and is to be used at the recipient's own risk.

The recipient is advised to consider seeking professional guidance with respect to its use of this publication.

This publication is not intended to constitute a contract. Users are responsible for its correct application.

© The British Standards Institution 2025
Published by BSI Standards Limited 2025

ISBN 978 0 539 28141 5

ICS 81.060.10

Compliance with a British Standard cannot confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 30 September 2025.

Amendments/corrigenda issued since publication

Date	Text affected
------	---------------

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 inductively
coupled plasma optical emission spectrometry with
electrothermal vaporisation (ETV-ICP-OES)

Essai des matières premières céramiques et des
matériaux céramiques - Détermination directe des
fractions massiques d'impuretés dans les poudres et
les granulés de carbure de silicium par spectroscopie
d'émission optique avec plasma induit par haute
fréquence avec vaporisation électrothermique (ETV-
ICP-OES)

Prüfung keramischer Roh- und Werkstoffe - Direkte
Bestimmung der Massenanteile von
Spurenverunreinigungen in pulver- und kornförmigem
Siliciumcarbid mittels optischer
Emissionsspektroskopie mit induktiv gekoppeltem
Plasma und elektrothermischer Verdampfung (ETV-
ICP-OES)

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

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.

CEN members are the national standards bodies of 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 United Kingdom.



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

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

Contents	Page
1 Scope	4
2 Normative references	4
3 Terms and definitions	4
4 Principle	4
5 Spectrometry	5
6 Apparatus.....	7
7 Reagents and auxiliary material.....	7
8 Sampling and sample preparation.....	8
9 Calibration	8
10 Procedure.....	9
11 Emission lines and working range.....	10
12 Calculation of the results and evaluation	10
13 Reporting of results.....	11
14 Precision.....	11
14.1 Repeatability.....	11
14.2 Reproducibility.....	11
15 Test report.....	11
Annex A (informative) Results of interlaboratory study	12
Annex B (informative) Emission lines and working range	17
Annex C (informative) Possible interferences and their elimination	19
Annex D (informative) Information regarding the evaluation of the uncertainty of the mean value.....	22
Annex E (informative) Commercial certified reference materials.....	23
Annex F (informative) Calibration using aqueous solutions and powdered calibration samples	24
Bibliography	27

European foreword

This document (EN 15991:2025) 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 15991:2015.

EN 15991:2025 includes the following significant technical changes with respect to EN 15991:2015:

- Clause 2 and Clause 3 have been added, noting that they neither add any normative references nor terms and definitions to the document;
- Clause 4 adds significantly more detail about the analysis and process;
- Clause 5 provides additional information on the spectrometry methodology;
- 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 specifies a method for the determination of the mass fractions of the elements Al, Ca, Cr, Cu, Fe, Mg, Ni, Ti, V and Zr in powdered and granular silicon carbide.

Dependent on element, emission lines, plasma conditions and sample mass, this test method is applicable for mass fractions 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 and electrothermal vaporization (ETV-ICP-OES) 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 sample mass or by choosing emission lines with different sensitivity.

After adequate verification, this document 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, boron carbide (B_4C), silicon nitride (Si_3N_4), boron nitride (BN) and several metal oxides as well as with the determination of P and S in some of these materials.

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

For the determination of impurities in silicon carbide, ICP-OES is a suited method. The classic application of ICP-OES is based on the nebulization of sample solutions. For silicon carbide, sample digestion by wet-chemical methods is required to obtain these sample solutions, for example by melt-fusion or acid/pressure-decomposition. These sample digestion procedures are time-consuming, require the use of hazardous chemicals, deteriorate the detection limits due to the dilution of the sample and the possibility of introduction of impurities as well as analyte losses represents a source of systematic errors.

With ETV-ICP-OES, the impurities are measured directly from the powdered silicon carbide sample, thus avoiding sample digestion and the associated disadvantages. Compared to wet-chemical ICP-OES methods, ETV-ICP-OES requires more effort for method development and is therefore particularly suitable when many samples of one matrix are to be measured with high sample throughput and with high detection sensitivity.

In ETV-ICP-OES, sample introduction by nebulization of liquids is replaced by the electrothermal vaporization of solid samples at high temperatures in the graphite tube furnace of the ETV-system.

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-system. A suitable design of the furnace (see Figures 1 and 2) and an optimized gas flow in the transition area between graphite tube and transport tube (see Figure 3) ensure that the sample vapour is transferred into a form that is transported to the ICP-OES with almost no losses (see [5], [6], [7], [8], [10]). Elements, for example titanium or zirconium, forming refractory carbides that are incompletely or not evaporating need to be converted in the graphite tube furnace into a chemical form which easily evaporates. For this purpose, a suitable reaction gas (halogenating agent) is mixed to the argon carrier-gas stream (see Figures 1 and 2) which converts the carbides into volatile halides (see [1], [3], [5] and [10].)

The evaporation products containing the element traces are transported by the transport tube as dry aerosol to the ICP-torch, injected into the plasma and there excited for the emission of optical radiation (see Figure 2).

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.

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.

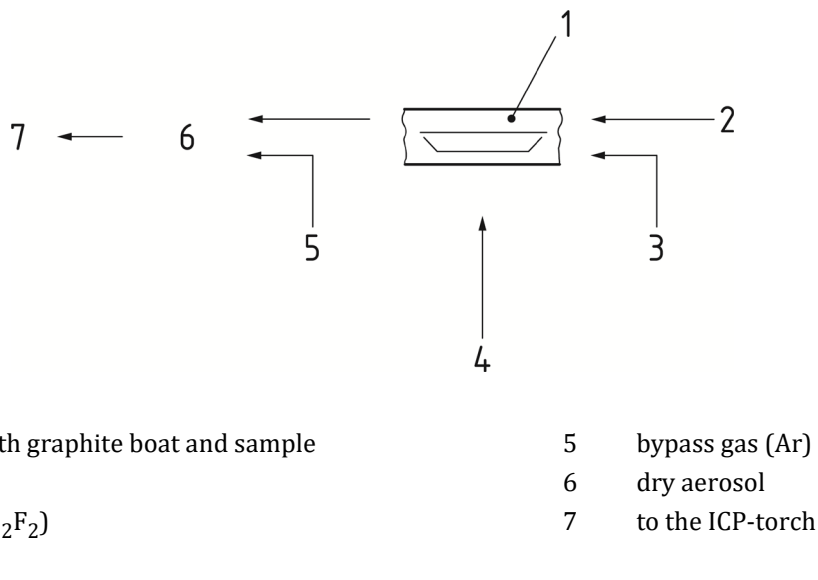
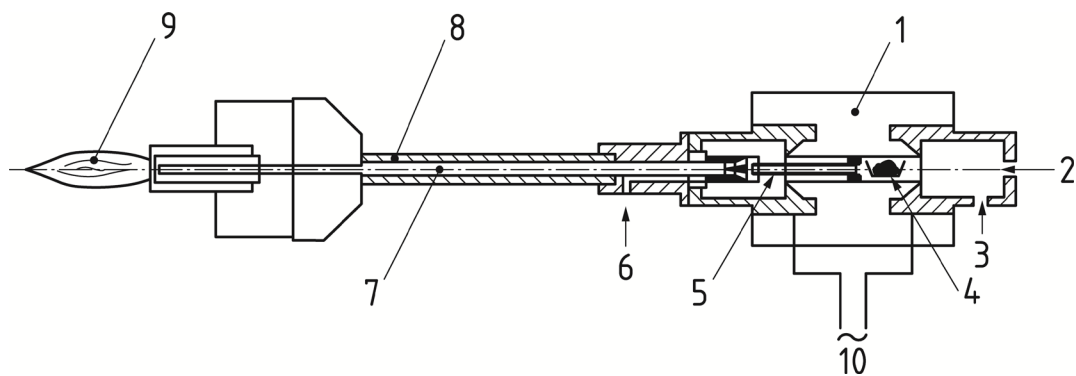


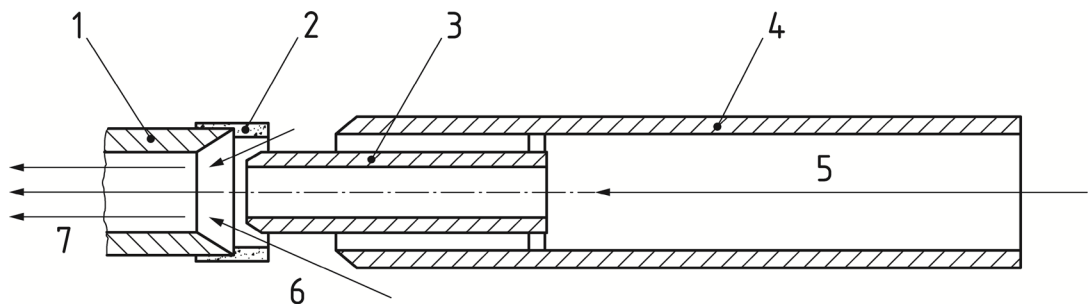
Figure 1 — Schematic representation of the ETV gas flows



Key

- | | | | |
|---|--|----|--|
| 1 | graphite tube furnace | 6 | bypass-gas (Ar) |
| 2 | pyrometer | 7 | dry aerosol to ICP-OES |
| 3 | carrier gas (Ar) + reaction gas (CCl ₂ F ₂) | 8 | transport tube |
| 4 | solid sample in graphite boat | 9 | inductively coupled plasma of the OES |
| 5 | evaporation products | 10 | power supply for graphite tube furnace |

Figure 2 — Schematic design of the ETV-system/ICP-OES coupling (example)



Key

- | | | | |
|---|-------------------------|---|--|
| 1 | alumina transport tube | 5 | carrier gas (Ar + CCl ₂ F ₂) and evaporation products of the sample |
| 2 | alumina transition ring | 6 | bypass gas (Ar) |
| 3 | nozzle of graphite tube | 7 | dry aerosol in laminar flow to ICP-OES |
| 4 | graphite tube | | |

Figure 3 — Schematic design of the transition area between graphite-tube and transport-tube (example)

NOTE Figure 1, Figure 2 and Figure 3 show a commercially available ETV-system.

6 Apparatus

6.1 Common laboratory instruments and laboratory instruments according to 6.2 to 6.7.

6.2 Inductively coupled plasma optical emission spectrometer (ICP-OES), simultaneous, with the possibility to register transient emission signals with a sampling rate of minimum 5 Hz and suited for the synchronised start of ETV vaporization cycle and registration of emission signals.

NOTE Especially for changing matrices the measurement of the spectral background near the emission lines is beneficial, because by this the systematic and stochastic contributions of the measurement uncertainty can be decreased, the latter only by simultaneous measurement of the background. The use of spectrometers equipped with area- or array-detectors is an advantage in such cases as they allow simultaneous background measurement.

6.3 Electrothermal vaporization (ETV) system, with graphite tube furnace with suited transition zone between graphite tube and transport tube for optimized aerosol formation, to be connected to the injector tube of the ICP-torch by a transport tube for example made of alumina (Al_2O_3), polytetrafluoroethylene (PTFE), perfluoroalkoxy (PFA) or polyvinyl chloride (PVC), with controlled gas flows (preferably with mass flow control) and furnace control (preferably with continuous temperature control of the graphite boat by a pyrometer) for a reproducible control of the ETV-system.

NOTE The use of an automatic sample changer improves the reproducibility of measurements.

6.4 Tweezers, self-closing, made of a material preventing contamination.

6.5 Micro spatula, made of a material preventing contamination.

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

NOTE An analytical balance with a resolution of 0,001 mg can improve the reproducibility of the measurements.

6.7 Mill or crusher, made of a material that does not contaminate the sample with any of the analytes to be determined, for example a mortar.

7 Reagents and auxiliary material

Only analytical grade reagents shall be used unless stated otherwise.

7.1 Sample boats, high-purity graphite (spectral grade) with low porosity, the size adapted to the graphite tube of the ETV-system.

7.2 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.)

7.3 Calibration solutions, aqueous single- or multi-element calibration solutions, prepared by dilution of commercially available standard-stock solutions with water to the required concentration.

7.4 Reaction gas, Dichlorodifluoromethane (CCl_2F_2) shall be used as reaction gas for simultaneous determination of the elements listed in Clause 1.

NOTE 1 The results of the interlaboratory study (see Annex A) were obtained using CCl_2F_2 as reaction gas.

NOTE 2 Inside the EU, according to the “EC-regulation on substances that deplete the ozone layer” (see [12]), the use of CCl_2F_2 is allowed for laboratory and analytical purposes. CCl_2F_2 is completely decomposed in the hot graphite furnace and in the downstream inductively coupled plasma. The use of CCl_2F_2 for laboratory and analysis purposes is, however, subject to registration at the European Commission (registration on the website of the European Commission, search for “labODS registry”).

Alternative reaction gases:

In general, compared to CCl_2F_2 the use of alternative reaction gases (halogenating agents) for analysis of silicon carbide can lead for some elements to a deterioration of release behaviour and transport efficiency. The suitability of an alternative reaction gas shall therefore be checked in advance.

The following alternative reaction gases have been used in practice for ETV-ICP-OES: trifluoromethane (CHF_3), tetrafluoromethane (CF_4), trichloromethane (CHCl_3), chlorodifluoromethane (CHClF_2), 1,1,1,2-tetrafluoroethane ($\text{C}_2\text{H}_2\text{F}_4$), sulphur hexafluoride (SF_6) und nitrogen trifluoride (NF_3). In practice, for CF_4 a comparable performance to CCl_2F_2 could be confirmed.

7.5 Carrier, bypass and shield gases, argon (Ar), purity $\geq 99,996\%$ (volume fraction).

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 $(110 \pm 10)^\circ\text{C}$ 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 sample material shall have a particle size of $\leq 50\ \mu\text{m}$. If necessary, the sample shall be crushed, milled and homogenized. For this, devices suited for the analytical task shall be applied. For porous materials, it shall be checked whether crushing is required.

Crushing is necessary if the transient analysis signals show an unusual long decay (tailing).

9 Calibration

The calibration shall be performed for each measuring cycle with calibration samples with defined mass fractions of the elements to be determined. The procedure shall be carried out in accordance with Clause 10. The calibration shall be carried out over a range adapted to the analytical task.

NOTE 1 This can be achieved by different masses of the same calibration sample or same masses of different calibration samples with different mass fractions of the analytes or by a combination of both possibilities.

Calibration samples and the sample to be analysed shall be measured under identical conditions.

Because of the low sample mass used and therefore the resulting scattering of the measurements, the number of calibration samples shall be adapted to the desired accuracy. Practically about 10 to 15 calibration samples have proved to be successful. For example, for 10 calibration samples, five different masses of two different calibration samples with different mass fractions of the analytes are required.

Preferably calibration samples of the same or similar material should be used, if possible certified reference materials (CRM) or matrix-adapted synthetic calibration samples.

The mass fractions of trace-impurities in the calibration samples should be in the same range as of the sample material.