



## ISO 21068-1

# Chemical analysis of raw materials and refractory products containing silicon-carbide, silicon-nitride, silicon-oxynitride and sialon —

## Part 1: General information, terminology and sample preparation

*Analyse chimique des matières premières et des produits réfractaires contenant du carbure de silicium, nitrure de silicium, oxynitride de silicium et sialon —*

*Partie 1: Informations générales, terminologie et préparation des échantillons*

Second edition  
2024-05

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Published in Switzerland

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This document was prepared by Technical Committee ISO/TC 33, *Refractories*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 187, *Refractory products and materials*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 21068-1:2008), which has been editorially and technically revised.

The main changes are as follows:

- The Scope was adjusted according to the Scope of the revised versions of ISO 21068-2, ISO 21068-3 and ISO 21068-4;
- Table 1 was removed as this is now part of the revised versions of ISO 21068-2, ISO 21068-3 and ISO 21068-4;
- The list of documents in [Clause 2](#) has been adjusted;
- [Clause 3](#) was adjusted to the revised versions of ISO 21068-2, ISO 21068-3 and ISO 21068-4;
- The weighing procedure has been removed as it can be assumed as general knowledge of the user of the ISO 21068 series;
- Bibliography was adjusted to this document.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at [www.iso.org/members.html](http://www.iso.org/members.html).

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The ISO 21068 series has been developed from the combination of EN 12698-1:2007<sup>[1]</sup> and EN 12698-2:2007<sup>[2]</sup> and ISO 21068-1:2008<sup>[3]</sup>, ISO 21068-2:2008<sup>[4]</sup> and ISO 21068-3:2008<sup>[5]</sup>. The last three standards have been originally developed from the combination of Japanese standard JIS R 2011:2007<sup>[6]</sup> and work items developed within CEN. Because there is a wide variety of laboratory equipment in use, the most commonly used methods are described.

ISO 21068-4 is derived from EN 12698-2:2007<sup>[2]</sup> describing XRD methods for the determination of mineralogical phases typically apparent in nitride and oxynitride bonded silicon carbide refractory products using a Bragg-Brentano diffractometer.

The ISO 21068 series is applicable to the analysis of all refractory products as classified in ISO 10081-1<sup>[7]</sup>, ISO 10081-2<sup>[8]</sup>, ISO 10081-3<sup>[9]</sup> and ISO 10081-4<sup>[10]</sup> (shaped) and ISO 1927-1<sup>[11]</sup> (unshaped) and raw materials containing carbon and/or silicon carbide. Therefore, the ISO 21068 series covers the full range of analysis from pure silicon carbide to oxide refractory composition with low-content silicon carbide and/or nitrides. Primarily, the ISO 21068 series provides methods to distinguish between different carbon bound types like total carbon ( $C_{\text{total}}$ ) and free carbon ( $C_{\text{free}}$ ) and derives from these two the silicon carbide content. ISO 21068-4 includes details of sample preparation and general principles for qualitative and quantitative analysis of mineralogical phase composition. Quantitative determination of  $\alpha$ - $\text{Si}_3\text{N}_4$ ,  $\beta$ - $\text{Si}_3\text{N}_4$ ,  $\text{Si}_2\text{ON}_2$ , AlN, and SiAlON are described.

If free carbon is present, ISO 21068-2 includes different temperature treatments to determine the mass changes gravimetrically. Frequently, the resulting residue is used for other determinations.

The determination of other groups of analytes described in the ISO 21068 series are free metals, free silicon ( $\text{Si}_{\text{free}}$ ), free aluminium ( $\text{Al}_{\text{free}}$ ), free magnesium ( $\text{Mg}_{\text{free}}$ ), free iron ( $\text{Fe}_{\text{free}}$ ) and the group of oxides from main to trace components.

The ISO 21068 series also describes the determination of silicon dioxide, total silicon, oxygen and nitrogen and other oxide bound metals that typically occur in the materials.

It represents a listing of analytical methods which is generally structured according to material composition. However, it is still the user who should prove the applicability of the method depending on the material and analytical requirements.

The most broadly used analytical techniques such as X-ray fluorescence spectroscopy (XRF) and inductively coupled plasma-optical emission spectrometry (ICP-OES) suffer from the disadvantage that the analytical results are chemical species independent. For carbon-containing ceramic raw materials and compositions, the ISO 21068 series provides analytical methods for the determination of free carbon, and SiC in the presence of oxide compounds in particular  $\text{SiO}_2$ .

Due to the diversity of laboratory equipment, the ISO 21068 series summarizes broadly used analytical techniques which lead to equivalent results. For example, the determination of carbon is based on all described methods on the reaction of carbon with oxygen at elevated temperatures to  $\text{CO}_2$ . Thus, carbon is analysed as  $\text{CO}_2$ .

As well as carbon and carbide compounds, metallic silicon, aluminium and magnesium are considered. While metallic silicon is mainly a precursor material which remains after the production process of SiC in the raw material, metallic aluminium is added as an antioxidant in carbon-containing refractory formulations.

Mostly oxide bound components, such as  $\text{Al}_2\text{O}_3$ , CaO, MgO,  $\text{TiO}_2$ ,  $\text{Cr}_2\text{O}_3$ ,  $\text{ZrO}_2$  and alkalis, can be determined by XRF, ICP-OES or wet chemical methods (see ISO 12677, ISO 26845<sup>[12]</sup>, ISO 21587-1<sup>[13]</sup>, ISO 21598-2<sup>[14]</sup> and ISO 21587-3<sup>[15]</sup>). These results can be corrected by formulae provided by the ISO 21068 series, in consideration of the values obtained by the determination of carbon, SiC, and metallic components.

The ISO 21068 series also provides methods for qualitative and quantitative determinations of the nitrogen content and the determination of oxygen. Thereby only the total content of nitrogen and oxygen is given; a precise determination of non-carbide components (oxides and nitrides) is not possible in this way.

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