



ISO 21068-2

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

**Part 2:
Determination of volatile components, total carbon, free carbon, silicon carbide, total and free silicon, free and surface silica**

Analyse chimique des matières premières et des produits réfractaires contenant du carbure de silicium, du nitrure de silicium, de l'oxynitride de silicium et du SiAlON —

Partie 2: Dosage des composés volatils, du carbone total, du carbone libre, du carbure de silicium, du silicium total et libre et de la silice libre et superficielle

**Second edition
2024-06**

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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This second edition cancels and replaces the first edition (ISO 21068-2:2008), which has been technically revised.

The main changes are as follows:

- methods described in ISO 12698-1:2007 for the determination of free carbon, silicon carbide and free silica have been included in this document;
- methods that are no longer used in practice have been removed;
- normative references and bibliography have been updated;
- document has been editorially revised.

A list of all parts in the ISO 21068 series can be found on the ISO website.

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The ISO 21068 series has been developed from the combination of EN 12698-1:2007^[1] and EN 12698-2:2007^[2] and ISO 21068-1:2008,^[3] ISO 21068-2:2008^[4] and ISO 21068-3:2008.^[5] The last three standards have been originally developed from the combination of Japanese standard JIS R 2011:2007^[6] 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^[2] describing XRD methods for the determination of mineralogical phases typically apparent in nitride and oxy-nitride 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,^[7] ISO 10081-2,^[8] ISO 10081-3 ^[9] and ISO 10081-4^[10] (shaped) and ISO 1927-1^[11] (unshaped) and for 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_{total}) and free carbon (C_{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 α -Si₃N₄, β -Si₃N₄, Si₂ON₂, 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 (Si_{free}), free aluminium (Al_{free}), free magnesium (Mg_{free}), free iron (Fe_{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 SiO₂.

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 CO₂. Thus, carbon is analysed as CO₂.

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 Al₂O₃, CaO, MgO, TiO₂, Cr₂O₃, ZrO₂ and alkalis, can be determined by XRF, ICP-OES or wet chemical methods (see ISO 12677^[13], ISO 26845^[23], ISO 21587-1^[20], ISO 21587-2^[21] and ISO 21587-3^[22]). 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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