
**Chemical analysis of silicon-carbide-
containing raw materials and refractory
products —**

Part 1:
**General information and sample
preparation**

*Analyse chimique des matières premières et des produits réfractaires
contenant du carbure de silicium —*

Partie 1: Informations générales et préparation des échantillons

ISO 21068-1:2008

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 21068-1 was prepared by Technical Committee ISO/TC 33, *Refractories*

ISO 21068 consists of the following parts, under the general title *Chemical analysis of silicon-carbide-containing raw materials and refractory products*:

- *Part 1: General information and sample preparation*
- *Part 2: Determination of loss on ignition, total carbon, free carbon and silicon carbide, total and free silica and total and free silicon*
- *Part 3: Determination of nitrogen, oxygen and metallic and oxidic constituents*

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Introduction

ISO 21068, Parts 1 to 3, have been developed from the combination of a Japanese standard JIS 2011 [2] and work items originally developed within CEN. As there is a wide variety of laboratory equipment in use, the most commonly used methods are described.

ISO 21068, Parts 1 to 3, are applicable to the analysis of all refractory products as classified in ISO 10081 [10] to [13] (shaped) and ISO 1927 [3] (unshaped) and raw materials containing carbon and/or silicon carbide. Therefore, ISO 21068, Parts 1 to 3, covers the full range of analysis from pure silicon carbide to oxidic refractory composition with low-content silicon carbide and/or nitrides. Primarily, ISO 21068, Parts 1 to 3, 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.

If free carbon is present, the standard includes different types of temperature treatment in order to determine the mass changes gravimetrically. Frequently, the resulting residue is used for other determinations.

The determination of other groups of analytes described in ISO 21068, Parts 1 to 3, are free metals, free silicon (S_{free}), free aluminum (Al_{free}), free magnesium (Mg_{free}), free iron (Fe_{free}) and the group of oxides from main to trace components.

ISO 21068, Parts 1 to 3, also describe the chemical analysis of silicon dioxide, total silicon, oxygen and nitrogen and other oxidic bound metals that typically occur in the materials.

It represents a listing of analytical methods which is approximately 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 bond independent. For carbon-containing ceramic raw materials and compositions ISO 21068, Parts 1 to 3, provide analytical methods for the determination of free carbon, and SiC in the presence of oxidic compounds in particular SiO_2 .

Because of the diversity of laboratory equipment, the three parts of ISO 21068 summarize broadly used analytical techniques which lead to equivalent results. In principle, the determination of carbon is based in all itemized methods on the oxygen extraction method, where carbon is oxidized at elevated temperatures. Thereafter carbon is analysed as CO_2 .

As well as carbon and carbidic compounds, metallic silicon, aluminium and magnesium are considered. While metallic silicon is in majority 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 oxidic bound components, such as Al_2O_3 , CaO, MgO, TiO_2 , Cr_2O_3 , ZrO_2 and alkalines, can be determined by XRF as described in ISO 12677, ICP-AES or wet chemical methods (see ISO 26845 [25], ISO 21587-1 [22] and ISO 21587-3 [24]). These results can be corrected by formulas provided by ISO 21068, Parts 1 to 3, in consideration of the values obtained by the determination of carbon, SiC, and metallic components.

ISO 21068, Parts 1 to 3, also provide 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.

ISO 21068, Parts 1 to 3, do not provide methods to distinguish quantitatively between different varieties of nitrides like silicon nitride, silicon oxy-nitride and sialon. For further information about the determination of this group of compounds, see EN 12698-2.

