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

Part 3:
**Determination of nitrogen, oxygen
and metallic and oxidic constituents**

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

*Partie 3: Dosage de l'azote, de l'oxygène et des constituants
métalliques et oxydés*



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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 21068-3 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 oxide constituents*

Introduction

ISO 21068, Parts 1 to 3, have been developed from the combination of a Japanese standard JIS 2011 [8] and work items originally developed within CEN. Because there is a wide variety of laboratory equipment in use, the most commonly used methods are described.

This part of ISO 21068 is applicable to the analysis of all refractory products as classified in ISO 10081 (all parts) [3], [4], [5], [6] (shaped) and ISO 1927 [1] (unshaped) and raw materials containing carbon and/or silicon carbide. Therefore, this part of ISO 21068 covers the full range of analysis from pure silicon carbide to oxidic refractory composition with a low content of silicon carbide and/or nitrides. Primarily, this part of ISO 21068 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, this part of ISO 21068 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 this part of ISO 21068 are free metals, free silicon (Si_{free}), free aluminum (Al_{free}), free magnesium (Mg_{free}), free iron (Fe_{free}) and the group of oxides from main to trace components.

This part of ISO 21068 also describes the chemical analysis of SiO_2 , total Si, oxygen and nitrogen and other oxidic bound metals which typically occur in the materials.

This part of ISO 21068 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.

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

Part 3: Determination of nitrogen, oxygen and metallic and oxidic constituents

1 Scope

This part of ISO 21068 specifies methods for the determination of total nitrogen and nitrogen calculated as silicon nitride, total oxygen, and free metallic and oxidic components in silicon carbide raw materials and refractory products.

It applies only to silicon carbide materials that are not bonded with nitrogen. Nitride-bonded silicon carbide refractories are covered in EN 12698-1.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 10058-1, *Chemical analysis of magnesite and dolomite refractory products (alternative to the X-ray fluorescence method) — Part 1: Apparatus, reagents, dissolution and gravimetric silica*

ISO 10058-2, *Chemical analysis of magnesite and dolomite refractory products (alternative to the X-ray fluorescence method) — Part 2: Wet chemical analysis*

ISO 10058-3, *Chemical analysis of magnesite and dolomite refractory products (alternative to the X-ray fluorescence method) — Part 3: Flame atomic absorption spectrometry (FAAS) and inductively coupled plasma emission spectrometry (ICP-AES)*

ISO 12677, *Chemical analysis of refractory products by XRF — Fused cast bead method*

ISO 20565-1, *Chemical analysis of chrome-bearing refractory products and chrome-bearing raw materials (alternative to the X-ray fluorescence method) — Part 1: Apparatus, reagents, dissolution and gravimetric silica*

ISO 20565-2, *Chemical analysis of chrome-bearing refractory products and chrome-bearing raw materials (alternative to the X-ray fluorescence method) — Part 2: Wet chemical analysis*

ISO 20565-3, *Chemical analysis of chrome-bearing refractory products and chrome-bearing raw materials (alternative to the X-ray fluorescence method) — Part 3: Flame atomic absorption spectrometry (FAAS) and inductively coupled plasma emission spectrometry (ICP-AES)*

ISO 21068-1:2008, *Chemical analysis of silicon-carbide-containing raw materials and refractory products — Part 1: General information and sample preparation*

ISO 21068-2:2008, *Chemical analysis of silicon-carbide-containing raw materials and refractory products — Part 2: Determination of loss on ignition, total carbon, free carbon and silicon carbide, total and free silica and total and free silicon*

ISO 21079-1, *Chemical analysis of refractories containing alumina, zirconia and silica — Refractories containing 5 % to 45 % of ZrO₂ (alternative to the X-ray fluorescence method) — Part 1: Apparatus, reagents and dissolution*

ISO 21079-2, *Chemical analysis of refractories containing alumina, zirconia and silica — Refractories containing 5 % to 45 % of ZrO₂ (alternative to the X-ray fluorescence method) — Part 2: Wet chemical analysis*

ISO 21079-3, *Chemical analysis of refractories containing alumina, zirconia and silica — Refractories containing 5 % to 45 % of ZrO₂ (alternative to the X-ray fluorescence method) — Part 3: Flame atomic absorption spectrometry (FAAS) and inductively coupled plasma emission spectrometry (ICP-AES)*

ISO 21587-1, *Chemical analysis of aluminosilicate refractory products (alternative to the X-ray fluorescence method) — Part 1: Apparatus, reagents, dissolution and gravimetric silica*

ISO 21587-2, *Chemical analysis of aluminosilicate refractory products (alternative to the X-ray fluorescence method) — Part 2: Wet chemical analysis*

ISO 21587-3, *Chemical analysis of aluminosilicate refractory products (alternative to the X-ray fluorescence method) — Part 3: Inductively coupled plasma and atomic absorption spectrometry methods*

ISO 26845, *Chemical analysis of refractories — General requirements for wet chemical analysis, atomic absorption spectrometry (AAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES) methods*

EN 12698-1:2007, *Chemical analysis of nitride bonded silicon carbide refractories — Part 1: Chemical methods*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 21068-1 apply.

4 Determination of nitrogen and oxygen

4.1 General

For oxygen only, the IR detection method is given; for nitrogen, several different methods are described, calculated nominally as Si₃N₄.

NOTE The calculation of nitrogen as Si₃N₄ is only applicable in the case where other nitride species are absent or too low to detect by XRD, see ISO 21068-1. Otherwise, nitrogen is reported as total nitrogen.

4.2 Combined determination of nitrogen and oxygen by an analyser with thermal conductivity (CR) and infrared absorption (IR) detection

4.2.1 Principle

The method uses inert-gas fusion analysis. A preweighed sample is placed in a graphite crucible positioned between the electrodes of an impulse furnace. 5 kW of power (typically) is passed through the crucible generating a temperature of approximately 2 800 °C.

NOTE 1 Furnace temperatures can be varied by increasing and decreasing current/voltage.