



**International
Standard**

ISO 21068-3

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

**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, du nitrure de
silicium, de l'oxynitrure de silicium et du SiAlON —*

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

**Second edition
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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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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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 21068-3:2008), which has been technically revised.

The main changes are as follows:

- methods described in ISO 12698-1:2007 for the dosage of free aluminium, total nitrogen and free alumina 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.

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.

Introduction

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.

This document is also applicable to the analysis of SiC raw materials.

Except the XRD method specified in ISO 21068-4, all chemical methods specified in this document are only validated for SiC raw materials. For refractory products 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 raw materials containing carbon and/or silicon carbide this document applies after appropriate verification for any matrix composition.

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

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

1 Scope

This document specifies analytical techniques for the determination of total nitrogen and nitrogen calculated as silicon nitride, total oxygen, and metallic and oxidic components in silicon carbide raw materials and refractory products.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 determination of 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 spectrophotometry (FAAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES)*

ISO 12677, *Chemical analysis of refractory products by X-ray fluorescence (XRF) — Fused cast-bead method*

ISO 16169, *Preparation of silicon carbide and similar materials for analysis by ISO 12677*

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 determination of 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 atomic emission spectrometry (ICP-AES)*

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

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

ISO 21068-3:2024(en)

ISO 21079-2, *Chemical analysis of refractories containing alumina, zirconia, and silica — Refractories containing 5 percent to 45 percent 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 percent to 45 percent of ZrO₂ (alternative to the X-ray fluorescence method) — Part 3: Flame atomic absorption spectrophotometry (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*

3 Terms and definitions

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

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 Determination of nitrogen and oxygen

4.1 General

For oxygen only the inert-gas fusion method is given; for nitrogen, calculated usually as Si₃N₄, several different methods are described.

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 (TC) and infrared (IR) absorption detection

4.2.1 Principle

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

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

The sample decomposes, releasing any oxygen and nitrogen present. The nitrogen released remains as elemental nitrogen, while oxygen combines with the carbon of the graphite crucible to form carbon monoxide. The sample gases are carried on a helium carrier gas either to a copper oxide catalyst, which converts carbon monoxide to carbon dioxide, and then to an infrared absorption cell which measures the carbon dioxide present or are measured directly without catalyst as carbon monoxide. The gas stream is then passed through sodium hydroxide to remove carbon dioxide, and magnesium perchlorate to remove any moisture present, and finally through a thermal conductivity cell to quantify the nitrogen.

Because the sample will invariably be in the form of a powder, it should be enclosed in a small tin capsule before placing it in the graphite crucible to prevent any loss of sample during analysis.