



SLOVENSKI STANDARD
oSIST prEN ISO 21068-1:2023
01-julij-2023

Kemijska analiza surovin in ognjevzdržnih izdelkov, ki vsebujejo silicijev karbid, silicijev nitrid, silicijev oksinitrid in sialon - 1. del: Splošne informacije in priprava vzorcev (ISO/DIS 21068-1:2023)

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/DIS 21068-1:2023)

Chemische Analyse von Rohstoffen und feuerfesten Erzeugnissen, die Siliziumcarbid, Siliziumnitrid, Siliziumoxynitrid und Sialon enthalten - Teil1: Allgemeine Angaben und Probenvorbereitung (ISO/DIS 21068-1:2023)

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 et préparation des échantillons (ISO/DIS 21068-1:2023)

Ta slovenski standard je istoveten z: prEN ISO 21068-1

ICS:

71.040.40	Kemijska analiza	Chemical analysis
81.080	Ognjevzdržni materiali	Refractories

oSIST prEN ISO 21068-1:2023 **en,fr,de**

DRAFT INTERNATIONAL STANDARD

ISO/DIS 21068-1

ISO/TC 33

Secretariat: **BSI**

Voting begins on:
2023-06-01

Voting terminates on:
2023-08-24

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

Part 1: General information and sample preparation

ICS: 81.080

iTeh STANDARD PREVIEW
(standards.iteh.ai)

[oSIST prEN ISO 21068-1:2023](https://standards.iteh.ai/catalog/standards/sist/eda428dc-e535-4800-893a-a9f7270d98aa/osist-pren-iso-21068-1-2023)

<https://standards.iteh.ai/catalog/standards/sist/eda428dc-e535-4800-893a-a9f7270d98aa/osist-pren-iso-21068-1-2023>

This document is circulated as received from the committee secretariat.

THIS DOCUMENT IS A DRAFT CIRCULATED FOR COMMENT AND APPROVAL. IT IS THEREFORE SUBJECT TO CHANGE AND MAY NOT BE REFERRED TO AS AN INTERNATIONAL STANDARD UNTIL PUBLISHED AS SUCH.

IN ADDITION TO THEIR EVALUATION AS BEING ACCEPTABLE FOR INDUSTRIAL, TECHNOLOGICAL, COMMERCIAL AND USER PURPOSES, DRAFT INTERNATIONAL STANDARDS MAY ON OCCASION HAVE TO BE CONSIDERED IN THE LIGHT OF THEIR POTENTIAL TO BECOME STANDARDS TO WHICH REFERENCE MAY BE MADE IN NATIONAL REGULATIONS.

RECIPIENTS OF THIS DRAFT ARE INVITED TO SUBMIT, WITH THEIR COMMENTS, NOTIFICATION OF ANY RELEVANT PATENT RIGHTS OF WHICH THEY ARE AWARE AND TO PROVIDE SUPPORTING DOCUMENTATION.

ISO/CEN PARALLEL PROCESSING



Reference number
ISO/DIS 21068-1:2023(E)

© ISO 2023

iTeh STANDARD PREVIEW (standards.iteh.ai)

[oSIST prEN ISO 21068-1:2023](https://standards.iteh.ai/catalog/standards/sist/eda428dc-e535-4800-893a-a9f7270d98aa/osist-pren-iso-21068-1-2023)

<https://standards.iteh.ai/catalog/standards/sist/eda428dc-e535-4800-893a-a9f7270d98aa/osist-pren-iso-21068-1-2023>



COPYRIGHT PROTECTED DOCUMENT

© ISO 2023

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

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

Published in Switzerland

Contents

	Page
Foreword.....	iv
Introduction.....	v
1 Scope.....	1
2 Normative references.....	2
3 Terms and definitions.....	2
4 Sampling and preparation of the test sample.....	3
4.1 General.....	3
4.2 Sample pre-treatment.....	4
4.2.1 General.....	4
4.2.2 Crushing.....	4
4.2.3 Thermal pre-treatment – Drying.....	4
5 Preliminary analyses.....	4
6 Expression of results.....	5
7 Test report.....	5
Bibliography.....	6

iTeh STANDARD PREVIEW (standards.iteh.ai)

[oSIST prEN ISO 21068-1:2023](https://standards.iteh.ai/catalog/standards/sist/eda428dc-e535-4800-893a-a9f7270d98aa/osist-pren-iso-21068-1-2023)

<https://standards.iteh.ai/catalog/standards/sist/eda428dc-e535-4800-893a-a9f7270d98aa/osist-pren-iso-21068-1-2023>

ISO/DIS 21068-1:2023(E)

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 raw materials and refractory products containing silicon-carbide, silicon-nitride, silicon-oxynitride and sialon*:

- *Part 1: General information and sample preparation*
- *Part 2: Determination of volatile components, total carbon, free carbon, silicon carbide, total and free silicon, free and surface silica*
- *Part 3: Determination of nitrogen, oxygen and metallic and oxide constituents*
- *Part 4: XRD methods*

Introduction

ISO 21068, Parts 1 to 4, have been developed from the combination of EN 12698:2007, Parts 1 [8] and 2 [9], and ISO 21068:2008, Parts 1 to 3 [10 to 12]. The latter has been originally developed from the combination of Japanese standard JIS R 2011:2007 [2] and work items developed within CEN. Because there is a wide variety of laboratory equipment in use, the most commonly used methods are described.

The new ISO 21068, Part 4, is derived from the European standard EN 12698-2:2007 [9] 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.

ISO 21068, Parts 1 to 4, are applicable to the analysis of all refractory products as classified in ISO 10081, Parts 1 to 4 [3 to 6], (shaped) and ISO 1927-1 (unshaped) and raw materials containing carbon and/or silicon carbide. Therefore, ISO 21068, Parts 1 to 4, covers the full range of analysis from pure silicon carbide to oxide refractory composition with low-content silicon carbide and/or nitrides. Primarily, ISO 21068, Parts 1 to 4, 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. The new Part 4 includes details of sample preparation and general principles for qualitative and quantitative analysis of mineralogical phase composition. Quantitative determination of α - Si_3N_4 , β - Si_3N_4 , Si_2ON_2 , AlN , and SiAlON are described.

If free carbon is present, the standard includes different temperature treatment 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 4, 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.

ISO 21068, Parts 1 to 4, also describe 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 ISO 21068, Parts 1 to 4, provide analytical methods for the determination of free carbon, and SiC in the presence of oxide compounds in particular SiO_2 .

Due to the diversity of laboratory equipment, the four parts of ISO 21068 summarize broadly used analytical techniques which lead to equivalent results. For example, the determination of carbon is based in all described methods on the reaction of carbon with oxygen at elevated temperatures to CO_2 . Thus, carbon is analysed as 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 Al_2O_3 , CaO , MgO , TiO_2 , Cr_2O_3 , ZrO_2 and alkalis, can be determined by XRF, ICP-OES or wet chemical methods (see ISO 12677, ISO 26845 [16], ISO 21587 Parts 1-3 [13 to 15]). These results can be corrected by formulas provided by ISO 21068, Parts 1 to 4, in consideration of the values obtained by the determination of carbon, SiC, and metallic components.

ISO 21068, Parts 1 to 4, 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/DIS 21068-1:2023(E)

ISO 21068, Parts 1 to 4, also provide methods to distinguish quantitatively between different varieties of nitrides like silicon nitride, silicon oxy-nitride and sialon.

iTeh STANDARD PREVIEW (standards.iteh.ai)

[oSIST prEN ISO 21068-1:2023](https://standards.iteh.ai/catalog/standards/sist/eda428dc-e535-4800-893a-a9f7270d98aa/osist-pren-iso-21068-1-2023)

<https://standards.iteh.ai/catalog/standards/sist/eda428dc-e535-4800-893a-a9f7270d98aa/osist-pren-iso-21068-1-2023>

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

Part 1: General information and sample preparation

1 Scope

This part of ISO 21068 gives definitions and specifies techniques for the preparation of samples for the chemical analysis of silicon-carbide-containing raw materials and refractory products including:

- a) SiC raw materials;
- b) graphite brick containing silicon carbide;
- c) silicon carbide brick (includes the bricks containing silicon nitride, silicon oxynitride, sialon);
- d) refractories containing carbon and/or silicon carbide mixed with clay;
- e) refractories containing carbon and/or silicon carbide mixed with silica (and fused silica);
- f) refractories containing carbon and/or silicon carbide mixed with high alumina material;
- g) refractories containing carbon and/or silicon carbide mixed with magnesia (and dolomite);
- h) refractories containing carbon and/or silicon carbide mixed with chrome mineral or magnesia-chrome materials;
- i) refractories containing carbon and/or silicon carbide except those described in a) to g) above.

The items of chemical analysis described in ISO 21068, Parts 1 to 3 are as follows:

- 1) loss on drying (LOD);
- 2) loss on ignition (LOI);
- 3) total carbon, C_{total} ;
- 4) free carbon, C_{free} ;
- 5) silicon carbide, SiC;
- 6) free silicon (Si_{free});
- 7) free aluminium (Al_{free});
- 8) free magnesium (Mg_{free});
- 9) free iron (Fe_{free});
- 10) silicon(IV) dioxide (SiO_2);
- 11) aluminium oxide (Al_2O_3);
- 12) iron(III) oxide (Fe_2O_3);

ISO/DIS 21068-1:2023(E)

- 13) titanium(IV) oxide (TiO_2);
- 14) calcium oxide (CaO);
- 15) magnesium oxide (MgO);
- 16) sodium oxide (Na_2O);
- 17) potassium oxide (K_2O);
- 18) chromium(III) oxide (Cr_2O_3);
- 19) zirconium oxide (ZrO_2);
- 20) boron oxide (total boron calculated as B_2O_3);
- 21) nitrogen;
- 22) oxygen;
- 23) nitrides (undifferentiated: Si_3N_4 , AlN, BN, sialon, oxy-nitrides, etc.).

ISO 21068-4 describes XRD methods for the determination of mineralogical phases typically apparent in nitride and oxy-nitride bonded silicon carbide refractory products.

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 1927-1, *Monolithic (unshaped) refractory products — Part 1: Introduction and classification*

ISO 5022, *Shaped refractory products — Sampling and acceptance testing*

ISO 8656-1, *Refractory products — Sampling of raw materials and unshaped products — Part 1: Sampling scheme*

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

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
Monolithic refractory materials
 Mixtures consisting of an aggregate and a bond or bonds, prepared ready for use either directly in the condition in which they are supplied or after the addition of one or more suitable liquids. Monolithic materials are defined in ISO 1927-1.

Note 1 to entry: Monolithic refractory materials can contain metallic, organic or ceramic fibre material.

Note 2 to entry: These mixtures are either dense or insulating. Insulating mixtures are those whose true porosity is not less than 45 % when determined in accordance with EN 1094-4 [1] using a test piece fired to specified conditions.

3.2
Dense shaped refractory materials
 Pre-shaped and fired or tempered refractory compositions to obtain a ceramic or a carbon-based bond.

Note 1 to entry: Dense shaped refractory materials are classified in accordance with ISO 10081 Part 1 to 4 [3]–[6].