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

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

iTeh STANDARD PREVIEW

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

*Partie 1: Informations générales et préparation des échantillons*

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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*

## 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 ( $C_{\text{total}}$ ) and free carbon ( $C_{\text{free}}$ ) 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 ( $S_{\text{free}}$ ), free aluminum ( $Al_{\text{free}}$ ), free magnesium ( $Mg_{\text{free}}$ ), free iron ( $Fe_{\text{free}}$ ) 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.

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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.

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

## 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) graphite brick containing silicon carbide;
- b) silicon carbide brick (includes the bricks containing silicon nitride);
- c) refractories containing carbon and/or silicon carbide mixed with clay;
- d) refractories containing carbon and/or silicon carbide mixed with silica (and fused silica);
- e) refractories containing carbon and/or silicon carbide mixed with high alumina material;
- f) refractories containing carbon and/or silicon carbide mixed with magnesia (and dolomite);
- g) refractories containing carbon and/or silicon carbide mixed with chrome mineral or magnesia-chrome materials;
- h) 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,  $C_{\text{total}}$ ;
- 4) free carbon,  $C_{\text{free}}$ ;
- 5) silicon carbide, SiC;
- 6) free silicon ( $\text{Si}_{\text{free}}$ );
- 7) free aluminium ( $\text{Al}_{\text{free}}$ );
- 8) free magnesium ( $\text{Mg}_{\text{free}}$ );
- 9) free iron ( $\text{Fe}_{\text{free}}$ );
- 10) silicon(IV) oxide ( $\text{SiO}_2$ );

- 11) aluminium oxide ( $\text{Al}_2\text{O}_3$ );
- 12) iron(III) oxide (total iron oxide calculated as  $\text{Fe}_2\text{O}_3$ );
- 13) titanium(IV) oxide ( $\text{TiO}_2$ );
- 14) calcium oxide ( $\text{CaO}$ );
- 15) magnesium oxide ( $\text{MgO}$ );
- 16) sodium oxide ( $\text{Na}_2\text{O}$ );
- 17) potassium oxide ( $\text{K}_2\text{O}$ );
- 18) chromium(III) oxide ( $\text{Cr}_2\text{O}_3$ );
- 19) zirconium oxide ( $\text{ZrO}_2$ );
- 20) boron oxide (total boron calculated as  $\text{B}_2\text{O}_3$ );
- 21) nitrogen;
- 22) oxygen;
- 23) nitrides (undifferentiated:  $\text{Si}_3\text{N}_4$ , AlN, BN, sialon, oxy-nitrides, etc.).

The range of determination specified in this part of ISO 21068 is given in Table 1.

**Table 1 — Range of determination**

Component	Range % by mass
LOI	-10 to 99
$C_{\text{total}}$	0,01 to 99
$C_{\text{free}}$	0,01 to 99
SiC	3 to 99
$\text{Si}_{\text{free}}$	0,1 to 10
$\text{Al}_{\text{free}}$	0,1 to 10
$\text{Mg}_{\text{free}}$	0,1 to 10
$\text{Fe}_{\text{free}}$	0,1 to 10
$\text{SiO}_2$	0,1 to 95
$\text{Al}_2\text{O}_3$	0,1 to 95
$\text{Fe}_2\text{O}_3$	0,1 to 15
$\text{TiO}_2$	0,1 to 5
CaO	0,1 to 60
MgO	0,1 to 95
$\text{Na}_2\text{O}$	0,1 to 5
$\text{K}_2\text{O}$	0,1 to 5
$\text{Cr}_2\text{O}_3$	0,1 to 40
$\text{ZrO}_2$	0,1 to 1
$\text{B}_2\text{O}_3$	0,1 to 5
$\text{Si}_3\text{N}_4$	0,1 to 35



## 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 31-0, *Quantities and units — Part 0: General principles*

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 12677:2003, *Chemical analysis of refractory products by XRF — Fused cast bead method*

EN 12698-2, *Chemical analysis of nitride bonded silicon carbide refractories — Part 2: XRD methods*

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*

## 3 Terms and definitions

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

### 3.1

#### unshaped refractory materials (standards.iteh.ai)

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

[ISO 21068-1:2008](#)

NOTE 1 Unshaped refractory materials can contain metallic, organic or ceramic fibre material.

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NOTE 2 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

presheared and burned or tempered refractory compositions to obtain a ceramic or a carbon-based bond

NOTE Dense shaped refractory materials are classified in accordance with ISO 10081 [10] to [13].

### 3.3

#### moisture

water which is not chemically bound in refractory raw materials and products, from moisture absorption during storage, or residual water used for the preparation of a refractory castable mix

### 3.4

#### volatile components

components which are evaporated at 750 °C under an argon atmosphere

NOTE In general, chemically bound water (hydraulic and phosphate-bond unshaped refractories), hydroxyl groups and organic components with low vapour pressure, as present in pitch, tar, resin or other organic binder, are removed.

### 3.5

#### refractories containing carbon and/or silicon-carbide

formulations containing refractory components provided as shaped or unshaped products containing SiC and/or carbon

NOTE Whereas carbon can be available as graphite, organic binder (e.g. pitch, tar, resin, carbon black), the SiC content in refractory material can vary from less than 1 % by mass to almost 100 % by mass.