SLOVENSKI STANDARD
oSIST prEN ISO 21068-2:2023

01-julij-2023

Kemijska analiza surovin in ognjevzdržnih izdelkov, ki vsebujejo silicijev karbid, silicijev nitrid, silicijev oksinitrid in sialon - 2. del: Določevanje hlapnih sestavin, celotnega ogljika, prostega ogljika, silicijevega karbida, celotnega in prostega silicija, prostega in površinskega silicija (ISO/DIS 21068-2:2023)

Chemical analysis of raw materials and refractory products containing silicon-carbide, silicon-nitride, silicon-oxynitride and sialon - Part 2: Determination of volatile components, total carbon, free carbon, silicon carbide, total and free silicon, free and surface silica (ISO/DIS 21068-2:2023)

Chemische Analyse von Rohstoffen und feuerfesten Erzeugnissen, die Siliciumcarbid, Siliciumnitrid, Siliciumoxinitrid und Sialon enthalten - Teil2: Bestimmung des Gehaltes an flüchtigen Bestandteilen, Gesamtkohlenstoff, freiem Kohlenstoff, Siliziumcarbid, gesamtem und freiem Silizium, freiem und anhaltendem Siliziumdioxid (ISO/DIS 21068-2:2023)

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 2: Dosage des composés volatils, du carbone total, du carbone libre, du carbure de silicium, du silicium total et libre et de la silice libre et superficielle (ISO/DIS 21068-2:2023)

Ta slovenski standard je istoveten z: **prEN ISO 21068-2**

ICS:

71.040.40	Kemijska analiza	Chemical analysis
81.080	Ognjevzdržni materiali	Refractories

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

Part 2:
Determination of volatile components, total carbon, free carbon, silicon carbide, total and free silicon, free and surface silica

ICS: 81.080

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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 21068-2 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 [1] 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 classified in ISO 10081, Parts 1 to 4 [2] to [5] (shaped) and ISO 1927-1 [6] (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 oxide 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. 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 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 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, ISO 21587 Parts 1-3). 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 21068, Parts 1 to 4, also provide methods to distinguish quantitatively between different varieties of nitrides like silicon nitride, silicon oxy-nitride and sialon.

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

Part 2:
Determination of volatile components, total carbon, free carbon, silicon carbide, total and free silicon, free and surface silica

1 Scope

This part of ISO 21068 specifies analytical techniques for the determination of volatile components by thermal treatment at specified temperatures, and methods for the determination of the total carbon content, free carbon, silicon carbide, total and free silicon and free and surface silica content of silicon-carbide, silicon-nitride and silicon-oxynitride containing raw materials and 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 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

ISO 9286, *Abrasive grains and crude — Chemical analysis of silicon carbide*

ISO 12677, *Chemical analysis of refractory products by X-ray fluorescence (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 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 ZrO_2 (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 percent to 45 percent of ZrO_2 (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 ZrO_2 (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*

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*

3 Terms and definitions

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

4 Determination of volatile components by gravimetric methods

4.1 General

The determination of volatile components is defined as change in mass caused by heat treatment of the sample at a defined temperature. The change in mass is measured by weighing.

Table 1 gives an overview of methods for determination of volatile components.

Table 1 — Methods for determination of volatile components

Title of method	Temperature	Subclause	Application
Loss on drying (LOD_{250})	250 °C	4.2	Attached water and chemically combined water are removed, for example in clay containing plastic formulations
Loss on ignition in argon (LOI_{Ar})	750 °C	4.3	All volatile compounds from pitch- or resin-bonded formulations are removed

4.2 Determination of the loss on drying at 250 °C (LOD_{250})

4.2.1 Principle

The test sample is heated at 250 °C ± 10 °C and the change in mass is determined gravimetrically.

4.2.2 Apparatus

4.2.2.1 Heat-resistant container, for example with dimensions 200 mm × 150 mm × 30 mm and made from stainless steel.

4.2.3 Procedure

Heat the heat-resistant container at 250 °C ± 10 °C for 30 min. Cool in a desiccator, weigh and record its empty mass, m_0, to the nearest 0,01 g.

Transfer 100 g to 600 g of the sample into the container and spread it out evenly. Then weigh and record the mass, m_1, of the container and sample to the nearest 0,01 g.

Place the container without a lid in air and heat it at 250 °C ± 10 °C for 16 h. Allow to cool in a desiccator. Weigh and record the mass, m_2, of the container plus the dried sample to the nearest 0,01 g.

4.2.4 Calculation

Calculate the loss on drying at 250 °C, LOD_{250}, as a percentage by mass, using Equation (1).

$$LOD_{250} = \frac{m_1 - m_2}{m_1 - m_0} \times 100 \quad (1)$$

where

m_0 is the mass of the empty container, in grams;

m_1 is the mass of the container plus the sample before heating, in grams;

m_2 is the mass of the container plus the sample after heating, in grams.

4.3 Determination of the loss on ignition in argon (LOI_{Ar})

4.3.1 Principle

The sample is heated in an argon atmosphere at 750 °C to remove volatile matter. The change in mass is determined gravimetrically.

NOTE The residue can be used for determination of C_{total}, SiC and C_{free} in organic matter containing materials. The change in mass during heating in argon must be considered for the calculation of C_{total}, SiC and C_{free}.

4.3.2 Apparatus

Ordinary laboratory apparatus and the following.

4.3.2.1 U-tube, with ground stoppers and filled with magnesium perchlorate.

4.3.2.2 Resistance furnace, capable of reaching (750 ± 25) °C, in the centre of the heating zone.

4.3.2.3 Thermocouple with display, registering up to 1 200 °C.

4.3.2.4 Ceramic tube, with cones or other gastight connector, of suitable diameter, made from porcelain, sillimanite, quartz or other suitable material.

4.3.2.5 Open combustion boats, of unglazed ceramic material, the length of which matches the oven's zone of constant temperature. The boats shall be broad enough to accommodate the amount of sample required for the determination.

4.3.2.6 Gas flowmeter, with an upper scale reading of around 20 l/h.

The argon-conducting parts, such as tubes and connections, must be made of material proofed against oxygen diffusion. Preferable materials are glass and copper. Silicone is unsuitable.

4.3.3 Test assembly

The test assembly is set up as shown in Figure 1.