SLOVENSKI STANDARD
oSIST prEN ISO 21068-3:2023

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Nadomešča:
SIST EN ISO 21068-3:2008

Kemijska analiza surovin in ognjevzdržnih izdelkov, ki vsebujejo silicijev karbid, silicijev nitrid, silicijev oksinitrid in sialon - 3. del: Določevanje dušika, kisika ter kovinskih in oksidnih sestavin (ISO/DIS 21068-3:2023)

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 (ISO/DIS 21068-3:2023)

Chemische Analyse von Rohstoffen und feuerfesten Erzeugnissen, die Siliziumcarbid, Siliziumnitrid, Siliziumoxynitrid und Sialon enthalten - Teil 3: Bestimmung des Gehaltes an Stickstoff, Sauerstoff sowie metallischen und oxidischen Bestandteilen (ISO/DIS 21068-3:2023)

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 3: Dosage de l'azote, de l'oxygène et des constituants métalliques et oxydés (ISO/DIS 21068-3:2023)

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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

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 21068-3 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 [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 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.

This part of ISO 21068 is also applicable to the analysis of SiC raw materials.

Except the XRD method specified in Part 4 all chemical methods specified in this standard are only validated for SiC raw materials. For 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 this standard apply 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 part of ISO 21068 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 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-OES)*

ISO 12677, *Chemical analysis of refractory products by 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 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-OES)*

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 21068-2, *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 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 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*

EN 15979, *Testing of ceramic raw and basic materials – Direct determination of mass fractions of impurities in powders and granules of silicon carbide by OES by DC arc excitation*

3 Terms and definitions

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

4 Determination of nitrogen and oxygen

4.1 General

For oxygen only the inert-gas fusion method is given; for nitrogen, calculated usually as Si_3N_4 several different methods are described.

NOTE The calculation of nitrogen as Si_3N_4 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 1 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.

For materials difficult to decompose, a fluxing agent shall be added to the sample. A suitable agent is oxygen free nickel capsule or nickel wire basket.

4.2.2 Reagents

Only chemicals the analytical purity of which is known and sufficient for the analytical purpose, as well as distilled water or water which has been completely deionized by means of an ion exchange process shall be used.

4.2.2.1 Tin capsule, of suitable dimensions and oxygen and nitrogen free.

4.2.2.2 High temperature graphite crucibles, of suitable size, recommended by the instrument producer.

4.2.2.3 Nickel capsules or basket, of suitable dimensions and oxygen and nitrogen free.

4.2.2.4 Acetic acid, 96 wt%

4.2.2.5 Nitric acid, 65 wt%

4.2.2.6 Hydrochloric acid, 32 wt%

4.2.2.7 Acetone

4.2.2.8 Carbon dioxide, 99,998 % pure.

4.2.2.9 Nitrogen, 99,998 % pure.

4.2.2.10 Helium, 99,998 % pure.

4.2.3 Apparatus

Ordinary laboratory apparatus and the following.

4.2.3.1 Combined nitrogen/oxygen analyser, commercially available.

NOTE If no combined analyser for nitrogen and oxygen is available, a separate nitrogen and/or oxygen analyser can be used.

4.2.3.2 Analytical balance, capable of measuring to the nearest 0,01 mg

4.2.4 Nickel pre-treatment

If nickel capsules or baskets are used, surface oxygen shall be removed by the following cleaning procedure:

Prepare a solution containing approximately 75 ml of acetic acid (4.2.2.4), 25 ml of nitric acid (4.2.2.5) and 1,5 ml of hydrochloric acid (4.2.2.6). In a well-ventilated fume cupboard, heat the solution to a temperature of 55 °C ± 5 °C, immerse the nickel capsule or basket in the heated solution for 30 s to 60 s, remove the nickel capsule or basket from the solution and rinse immediately in running water. Immerse the nickel capsule or basket in chemically pure acetone (4.2.2.7), dry thoroughly and place the cleaned nickel capsules or basket in a desiccator.

4.2.5 Calibration

Referring to the instrument operation manual, the calibration can be achieved by two methods:

a) using primary standards or certified reference materials;

b) by injection of known volumes of pure carbon dioxide (4.2.2.8) and nitrogen (4.2.2.9) into the detection system.

If b) is used, it is recommended to additionally analyze a certified reference material to verify the performance of the electrode furnace, associated chemicals and detection system.

4.2.6 Procedure

4.2.6.1 General

Operate the instrument in accordance with the instrument operation manual.

4.2.6.2 Determination

Prepare and dry the sample as specified in Clause 4 of ISO 21068-1. Weigh it, to the nearest 0,01 mg, into the capsule (4.2.2.1) and seal it, taking care to expel any air present.

NOTE A typical sample mass is approximately 50 mg. However, in practice, the sample mass is determined by a combination of the dynamic range of the analyser and the magnitude of the concentration of oxygen and nitrogen present.

Place the tin capsule including the sample into the loading-mechanism of the analyser. If nickel is used, the tin capsule is firstly put into the nickel capsule or basket.

Carry out the analysis in two stages:

a) heat the graphite crucible to a temperature at least as high as that used for the analysis, for a period of time sufficient to allow any entrapped oxygen and nitrogen to be expelled;

b) drop the sample into the graphite crucible and perform the analysis.

At least three determinations per sample shall be carried out.

4.2.6.3 Blank determinations

The blank shall be the mean of at least three determinations.

4.2.6.4 Calculation

Calculate the mass fraction of nitrogen or oxygen, w_a, expressed as a percentage, using Equation (1).

$$w_a = w_m - b \tag{1}$$

where

w_m is the mass fraction of nitrogen or oxygen, respectively, measured in the sample, expressed as a percentage;

b is the average blank determination of nitrogen or oxygen respectively, expressed as a percentage by mass.

Report the results as the mean of three determinations.