SLOVENSKI STANDARD
SIST EN ISO 21068-2:2008

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Kemijska analiza surovin in ognjevzdrţnih izdelkov s silicijevim karbidom - 2. del: Določevanje ostanka po ţarenju, celokupnega ogljika, prostega ogljika in silicijevega karbida, celokupne in proste silike in celokupnega in prostega silicija (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 (ISO 21068-2:2008)

Chemische Analyse von Siliciumcarbid enthaltenden Rohstoffen und feuerfesten Erzeugnissen - Teil 2: Bestimmung des Glühverlustes und Gehaltes an Gesamtkohlenstoff, freiem Kohlenstoff und Siliciumcarbid, des Gehaltes an gesamtem und freiem Silicium(IV)-oxid sowie an gesamtem und freiem Silicium (ISO 21068-2:2008)

Analyse chimique des matières premières et des produits réfractaires contenant du carbure de silicium - Partie 2: Détermination de la perte au feu, du carbone total, du carbone libre et du carbure de silicium, de la silice totale et libre, et du silicium total et libre (ISO 21068-2:2008)

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EUROPÄISCHE NORM

EN ISO 21068-2

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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 (ISO 21068-2:2008)

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Foreword

This document (EN ISO 21068-2:2008) has been prepared by Technical Committee ISO/TC 33 "Refractories" in collaboration with Technical Committee CEN/TC 187 “Refractory products and materials” the secretariat of which is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by February 2009, and conflicting national standards shall be withdrawn at the latest by February 2009.

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Endorsement notice

The text of ISO 21068-2:2008 has been approved by CEN as a EN ISO 21068-2:2008 without any modification.

INTERNATIONAL STANDARD

ISO 21068-2

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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

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

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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.

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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 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 [6] and work items originally developed within CEN. Because there is a wide variety of laboratory equipment in use, the most commonly used methods are described.

This part of ISO 21068 is applicable to the analysis of all refractory products classified in ISO 10081 [2] to [5] (shaped) and ISO 1927 [1] (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 oxidic 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.

If free carbon is present, this part of ISO 21068 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 this part of ISO 21068 are free metals, free silicon (Si_{free}), free aluminum (Al_{free}), free magnesium (Mg_{free}), free iron (Fe_{free}) and the group of oxides from main to trace components.

This part of ISO 21068 also describes the chemical analysis of SiO_2, total Si, oxygen and nitrogen and other oxidic-bound metals which typically occur in the materials.

This part of ISO 21068 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.

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

1 Scope

This part of ISO 21068 specifies analytical techniques for the determination of change in mass by thermal treatment at specified temperatures, and methods for the determination of the total carbon content, free carbon, silicon carbide, silicon, total silica and free silica content of silicon-carbide-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 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

ISO 9286:1997, *Abrasive grains and crude — Chemical analysis of silicon carbide*

ISO 10060, *Dense, shaped refractory products — Test methods for products containing carbon*

ISO 21068-1:2008, *Chemical analysis of silicon-carbide-containing raw materials and refractory products — Part 1: General information and sample preparation*

EN 12698-1:2007, *Chemical analysis of nitride bonded silicon carbide refractories — Part 1: Chemical methods*

3 Terms and definitions

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

4 Determination of change in mass

4.1 General

The determination of change in mass is defined as a loss or increase in mass caused by heat treatment. Several methods are distinguished based on the sample material, temperature and atmosphere. The determination of change in mass is carried out by a gravimetric method. Heat treatments in air will lead to a loss of volatile matter and carbon and an increase of mass due to oxidation, whereas a heat treatment in argon will only lead to a loss of volatile matter.

Normally, the residue is used for other determinations. Therefore, the change in mass is considered for the calculation of the analytical result. The analytical method which is applied to the residue depends on the matrix and the parameters to be determined. It is up to the user to select the appropriate analytical method.

Table 1 gives an overview of methods of determination of change in mass by heat pretreatments and their different applications.

Table 1 — Methods and application purpose of determination of change in mass

Short title of method	Temperature	Subclause	Application
Loss on drying (LOD_{250})	250 °C	4.2	Attached water and chemically combined water are removed, e.g. in clay containing plastic formulations
Loss on calcination in argon (LOI_{Ar})	750 °C	4.3	All volatile compounds out of pitch- or resin-bonded formulations are removed
Change in mass in air	200 °C	4.4	Volatile compounds are removed from resin-bonded formulations
	400 °C	4.4	Volatile compounds are removed from pitch-bonded formulations
Change in mass (LOI_{750})	750 °C	4.5	Both procedures are suitable to remove carbon (e.g. graphite) from refractory formulations. If fine-grained SiC is present, care should be taken because SiC may be oxidised as well.
Loss on ignition (LOI_{850})	850 °C	4.6	
Loss on ignition ($LOI_{1\,050}$)	1 050 °C	4.7	Loss or gain of mass; attached water, chemically combined water, carbon, organic compounds (e.g. pitch, resin), silicon carbide, and metals are removed

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

4.2.1 Principle

The test sample is heated at 250 °C ± 10 °C and the loss of mass from attached water is determined.

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 the sample into the container and spread it out flat. 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 an air bath 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 drying, in grams;

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

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

4.3.1 Principle

Pretreatment under argon at 750 °C to remove volatile matter. The loss of volatile matter is determined by a gravimetric method.

The residue (R_{Ar}) is normally used for determination of C_{total}, SiC and C_{free} (therefore these parameters will be additionally indexed with $_{Ar}$), and the change in mass has to be considered for the calculation of the result.

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, heatable and adjustable at (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, with an inner diameter ⩾ 16 mm, made from porcelain, sillimanite, quartz or other suitable material.

4.3.2.5 Open combustion boats, of unglazed ceramic material, the length of which is adapted to 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, should be made of material proofed against oxygen diffusion. Preferable materials are glass and copper. Silicone is unsuitable.

4.3.2.7 Test assembly

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