



International
Standard

ISO 17947-1

**Fine ceramics (advanced ceramics,
advanced technical ceramics) —
Methods for chemical analysis of
silicon nitride powders —**

**Part 1:
Wet chemical methods, X-ray
fluorescence (XRF) using the fused
cast-bead method, carrier-gas hot
extraction (CGHE) and combustion
methods**

*Céramiques techniques — Méthodes pour l'analyse chimique de
poudres de nitrure de silicium —*

*Partie 1: Méthodes chimiques par voie humide, fluorescence de
rayons X (XRF) utilisant la méthode de la perle fondue, extraction
à chaud sous gaz vecteur (CGHE) et méthodes de combustion*

**First edition
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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 206, *Fine ceramics*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 184, *Advanced technical ceramics*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This first edition of ISO 17947-1 cancels and replaces ISO 17947:2014, which has been technically revised.

A list of all parts in the ISO 17947 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

This document has been developed from Japanese Industrial Standard JIS R 1603:2007^[1] with reference to CEN ENV 14226:2002^[2] and ASTM C1494-01:2007^[3], and is applicable to the chemical analysis of silicon nitride raw powders for fine ceramics use. This document covers both major and minor constituents, such as silicon, nitrogen, and some trace metallic and non-metallic elements.

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Fine ceramics (advanced ceramics, advanced technical ceramics) — Methods for chemical analysis of silicon nitride powders —

Part 1:

Wet chemical methods, X-ray fluorescence (XRF) using the fused cast-bead method, carrier-gas hot extraction (CGHE) and combustion methods

1 Scope

This document specifies methods for the determination of silicon, nitrogen, aluminium, iron, calcium, oxygen, carbon, fluorine, and chlorine in silicon nitride powders.

This document is applicable for the chemical analysis of silicon nitride powders used as raw material for fine ceramics.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 2828, *Aluminium oxide primarily used for the production of aluminium — Determination of fluorine content — Alizarin complexone and lanthanum chloride spectrophotometric method*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 6353-1, *Reagents for chemical analysis — Part 1: General test methods*

ISO 6353-2, *Reagents for chemical analysis — Part 2: Specifications — First series*

ISO 6353-3, *Reagents for chemical analysis — Part 3: Specifications — Second series*

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

ISO 21068-2:2024, *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 21068-3:2024, *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 21438-2, *Workplace atmospheres — Determination of inorganic acids by ion chromatography — Part 2: Volatile acids, except hydrofluoric acid (hydrochloric acid, hydrobromic acid and nitric acid)*

ISO 21438-3, *Workplace atmospheres — Determination of inorganic acids by ion chromatography — Part 3: Hydrofluoric acid and particulate fluorides*

ISO 26845:2008, *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

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

4 Analytes and ranges

Analytes and ranges specified in this document shall be as follows.

- Silicon (Si), range of 30 % to 70 % (mass fraction)
- Nitrogen (N), range of 30 % to 45 % (mass fraction)
- Aluminium (Al), range of 0,001 % to 0,6 % (mass fraction)
- Iron (Fe), range of 0,001 % to 0,6 % (mass fraction)
- Calcium (Ca), range of 0,001 % to 0,03 % (mass fraction)
- Oxygen (O), range of 0,05 % to 5 % (mass fraction)
- Carbon (C), range of 0,01 % to 6 % (mass fraction)
- Fluorine (F), range of 0,001 % to 0,2 % (mass fraction)
- Chlorine (Cl), range of 0,001 % to 0,2 % (mass fraction)

NOTE The results of an interlaboratory test conducted using three silicon nitride powder samples are listed in [Annex B](#).

5 Preparation of test sample

5.1 General

The method of preparing samples shall be in accordance with ISO 8656-1 unless otherwise mutually agreed upon between the analyser and the customer.

5.2 Sampling

Sampling shall be in accordance with ISO 8656-1.

5.3 Drying

The following procedure shall be used: Take about 10 g of the sample into a flat-type weighing bottle (60 mm × 30 mm) and spread it uniformly over the bottom of the bottle. Place the bottle in a drying oven at 110 °C ± 5 °C for 2 h without a lid, and then cool in a desiccator (desiccant: magnesium perchlorate for drying) with a lid for 1 h.

5.4 Weighing

The required quantity of the sample shall be weighed to the nearest 0,1 mg using a balance.

6 Apparatus and reagents

Unless otherwise specified for each determination, ordinary laboratory apparatus for chemical analysis as listed in ISO 26845:2008, Clause 4 may be used. Reagents shall conform to the requirements of ISO 6353-1, ISO 6353-2, and ISO 6353-3. Unless otherwise specified for each determination, reagents of analytical grade listed in ISO 26845:2008, Clause 5, shall be used.

7 Blank test

Blank test shall be carried out without the sample by using identical quantities of reagents, conditions, and procedures throughout each determination to correct the analytical values obtained.

8 Determination of silicon

8.1 Classification of determination methods

Silicon shall be determined by either of the following methods. If analytical results with four figures are required, use the method A. If analytical results with two or three figures are required, the method B can be used.

- Method A: Fusion–dehydration/insolubilization separation–gravimetry and inductively coupled plasma optical emission spectrometry (ICP-OES)
- Method B: XRF using fused cast-bead method

8.2 Fusion-dehydration/insolubilization separation-gravimetry and ICP-OES

8.2.1 Principle

A sample is fused with alkaline carbonate and the melt is treated with an acid to separate into two parts of silicon, insoluble silicon and soluble silicon, by filtration. Insoluble silicon is determined gravimetrically as silicon dioxide after ignition of the insoluble residue, whereas soluble silicon is determined in the filtrate using ICP-OES. The sum of them represents the content of silicon in the sample.

8.2.2 Reagents

Reagents of analytical grade shall be used. Reagent solutions shall be preserved in plastic bottles.

8.2.2.1 Water, of grade 1 or superior specified in ISO 3696.

8.2.2.2 Sodium carbonate, Na_2CO_3 , anhydrous, specified in ISO 6353-3 or that of higher grade.

8.2.2.3 Hydrochloric acid (1+1), (1+4), (1+50), HCl, concentrated hydrochloric acid ($c(\text{HCl}) = 32 \%$, mass fraction) diluted (1+1), (1+4) and (1+50) by volume with water ([8.2.2.1](#)).

8.2.2.4 Sulfuric acid (1+1), (1+4), H_2SO_4 , concentrated sulfuric acid ($c(\text{H}_2\text{SO}_4) = 96 \%$, mass fraction) diluted (1+1) and (1+4) by volume with water ([8.2.2.1](#)).

8.2.2.5 Cellulose powder.