

---

---

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

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

iTeh Standards  
(<https://standards.iteh.ai>)  
Document Preview

ISO 17947:2014

<https://standards.iteh.ai/catalog/standards/iso/de225f6a-6640-47dd-890e-6ef04fba7f01/iso-17947-2014>



iTeh Standards  
(<https://standards.iteh.ai>)  
Document Preview

ISO 17947:2014

<https://standards.iteh.ai/catalog/standards/iso/de225f6a-6640-47dd-890e-6ef04fba7f01/iso-17947-2014>



**COPYRIGHT PROTECTED DOCUMENT**

© ISO 2014

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office  
Case postale 56 • CH-1211 Geneva 20  
Tel. + 41 22 749 01 11  
Fax + 41 22 749 09 47  
E-mail [copyright@iso.org](mailto:copyright@iso.org)  
Web [www.iso.org](http://www.iso.org)

Published in Switzerland

# Contents

Page

<b>Foreword</b>	<b>v</b>
<b>Introduction</b>	<b>vi</b>
<b>1 Scope</b>	<b>1</b>
<b>2 Normative references</b>	<b>1</b>
<b>3 Analytes and ranges</b>	<b>2</b>
<b>4 Preparation of test sample</b>	<b>2</b>
4.1 Sampling	2
4.2 Drying	2
4.3 Weighing	2
<b>5 Apparatus and reagents</b>	<b>2</b>
<b>6 Blank test</b>	<b>2</b>
<b>7 Determination of total silicon</b>	<b>3</b>
7.1 Classification of determination methods	3
7.2 Fusion-dehydration/insolubilization separation-gravimetry and ICP-OES	3
7.3 XRF using fused cast-bead method	5
<b>8 Determination of total nitrogen</b>	<b>5</b>
8.1 Classification of determination methods	5
8.2 Acid pressure decomposition-distillation separation-acidimetric titration method	5
8.3 Inert gas fusion-thermal conductivity method	10
8.4 Fusion-ammonia separation-acidimetric titration method	13
<b>9 Determination of aluminium, iron, and calcium</b>	<b>13</b>
9.1 Principle	13
9.2 Reagents	13
9.3 Apparatus and instrument	14
9.4 Procedure	14
9.5 Blank test	15
9.6 Drawing calibration curve	15
9.7 Calculation	15
<b>10 Determination of oxygen</b>	<b>16</b>
10.1 Principle	16
10.2 Reagents	16
10.3 Apparatus	16
10.4 Instrument	16
10.5 Procedure	16
10.6 Blank test	16
10.7 Calculation of calibration coefficient	16
10.8 Calculation	17
<b>11 Determination of carbon</b>	<b>17</b>
11.1 Classification of determination methods	17
11.2 Combustion (RF furnace)-IR absorption spectrometry	17
11.3 Combustion (resistance furnace)-coulometry	20
11.4 Combustion (resistance furnace)-gravimetry	20
11.5 Combustion (resistance furnace)-conductometry	20
<b>12 Determination of fluorine and chlorine</b>	<b>20</b>
12.1 Principle	20
12.2 Reagents	20
12.3 Apparatus and instruments	21
12.4 Procedure	21
12.5 Blank test	22

12.6	Drawing calibration curve.....	22
12.7	Calculation.....	22
<b>13</b>	<b>Reporting analytical values.....</b>	<b>23</b>
13.1	Number of analyses.....	23
13.2	Evaluation of analytical values.....	23
13.3	Expression of analytical values.....	23
<b>14</b>	<b>Test report.....</b>	<b>24</b>
<b>Annex A</b> (informative)	<b>List of commercial certified reference materials .....</b>	<b>25</b>
<b>Annex B</b> (informative)	<b>Analytical results obtained from a round robin test.....</b>	<b>26</b>
<b>Annex C</b> (informative)	<b>Spectral lines for ICP-OES.....</b>	<b>31</b>
<b>Bibliography</b>	<b>.....</b>	<b>32</b>

iTeh Standards  
(<https://standards.iteh.ai>)  
Document Preview

ISO 17947:2014

<https://standards.iteh.ai/catalog/standards/iso/de225f6a-6640-47dd-890e-6ef04fba7f01/iso-17947-2014>

## 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 documents 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](http://www.iso.org/directives)).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see [www.iso.org/patents](http://www.iso.org/patents)).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 206, *Fine ceramics*.

## Document Preview

ISO 17947:2014

<https://standards.iteh.ai/catalog/standards/iso/de225f6a-6640-47dd-890e-6ef04fba7f01/iso-17947-2014>

## Introduction

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

iTeh Standards  
(<https://standards.iteh.ai>)  
Document Preview

ISO 17947:2014

<https://standards.iteh.ai/catalog/standards/iso/de225f6a-6640-47dd-890e-6ef04fba7f01/iso-17947-2014>

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

## 1 Scope

This International Standard specifies the methods for the chemical analysis of fine silicon nitride powders used as the raw material for fine ceramics.

This International Standard stipulates the determination methods of total silicon, total nitrogen, aluminium, iron, calcium, oxygen, carbon, fluorine, and chlorine in fine silicon nitride powders.

## 2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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, *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 21068-3, *Chemical analysis of silicon-carbide-containing raw materials and refractory products — 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, *Chemical analysis of refractories — General requirements for wet chemical analysis, atomic absorption spectrometry (AAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES) methods*

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

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

### 3 Analytes and ranges

Analytes and ranges specified in this International Standard shall be as follows.

- a) Total silicon (T. Si), range of 30 % to 70 % (mass fraction)
- b) Total nitrogen (T. N), range of 30 % to 45 % (mass fraction)
- c) Aluminium (Al), range of 0,001 % to 0,6 % (mass fraction)
- d) Iron (Fe), range of 0,001 % to 0,6 % (mass fraction)
- e) Calcium (Ca), range of 0,001 % to 0,03 % (mass fraction)
- f) Oxygen (O), range of 0,05 % to 5 % (mass fraction)
- g) Carbon (C), range of 0,01 % to 6 % (mass fraction)
- h) Fluorine (F), range of 0,001 % to 0,2 % (mass fraction)
- i) Chlorine (Cl), range of 0,001 % to 0,2 % (mass fraction)

### 4 Preparation of test sample

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

#### 4.1 Sampling

Take the sample in accordance with ISO 8656-1.

#### 4.2 Drying

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 an air bath at  $110\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$  for 2 h without a lid, and then cool in a desiccator (desiccant: magnesium perchlorate for drying) with a lid for 1 h.

#### 4.3 Weighing

Weigh the sample of the required quantity to the nearest 0,1 mg using a balance.

### 5 Apparatus and reagents

Unless otherwise specified in each determination, use ordinary laboratory apparatus for chemical analysis listed in ISO 26845, Clause 4, as necessary. Reagents should conform to the requirements of ISO 6353-1, ISO 6353-2, and ISO 6353-3, as appropriate. Unless otherwise specified in each determination, use corresponding reagents of analytical grade listed in ISO 26845, Clause 5, as necessary.

### 6 Blank test

Blank test shall be carried out by using identical quantities of reagents, conditions, and procedures throughout each determination to correct the analytical values obtained.



## 7 Determination of total silicon

### 7.1 Classification of determination methods

Total 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 ICP-OES
- Method B: XRF using fused cast-bead method

### 7.2 Fusion-dehydration/insolubilization separation-gravimetry and ICP-OES

#### 7.2.1 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 using gravimetry as silicon dioxide converted after ignition, whereas soluble silicon in the filtrate is determined using ICP-OES. The sum of them represents the total silicon.

#### 7.2.2 Reagents

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

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

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

**7.2.2.3 Hydrochloric acid (1+1), (1+4), (1+50)**, prepared by diluting hydrochloric acid with water, respectively.

**7.2.2.4 Sulfuric acid (1+1), (1+4)**, prepared by diluting sulfuric acid with water, respectively.

**7.2.2.5 Cellulose powder**.

**7.2.2.6 Polyethylene oxide solution [0,05 % (m/V)]**, prepared by dissolving polyethylene oxide with water.

**7.2.2.7 Hydrofluoric acid**, concentration of 48 %.

#### 7.2.3 Apparatus and instruments

Use ordinary laboratory apparatus and instruments for chemical analysis in accordance with ISO 26845, Clause 4.

**7.2.3.1 Platinum dish**.

**7.2.3.2 Platinum crucible**.

**7.2.3.3 Burner**, capable of heating at 1 100 °C.

**7.2.3.4 Muffle furnace**, capable of being operated at 1 100 °C.

**7.2.3.5 Balance**, readable to 0,1 mg.

**7.2.3.6 Inductively coupled plasma optical emission spectrometer (ICP-OES).**

#### **7.2.4 Procedure**

The procedure shall be as follows. The procedure described in ISO 21068-2, Clause 8 can be alternatively used.

##### **7.2.4.1 Fusion of sample**

Weigh 0,30 g of the sample and 2,0 g of sodium carbonate, anhydrous into a platinum dish and mix well. Start to heat carefully and increase the temperature gradually to 1 000 °C to completely fuse the sample using a burner or in a muffle furnace.

##### **7.2.4.2 Separation of silicon**

Add 20 ml of hydrochloric acid (1+1) to dissolve the melt on a hot plate. Silicon dioxide will appear to be jellified and precipitated at this stage. There are two methods to separate the precipitated silicon dioxide.

- a) Dehydrate carefully the precipitate to dryness in order to prevent it from spattering and add 5 ml of hydrochloric acid and 20 ml of water to dissolve any salt mixed with the precipitate. Filtrate the precipitate with a filter paper and wash with hot hydrochloric acid (1+50) several times and then with hot water sufficiently until it contains no salt. Receive the filtrate and washings together in a volumetric flask and make constant volume. Preserve this precipitate for gravimetry of insoluble silicon and the solution for the ICP-OES determination of soluble silicon, respectively.
- b) After eduction of jellified silicon dioxide, add 0,05 g of cellulose powder and 10 ml of polyethylene oxide solution to agglomerate silicon dioxide for easy filtration. Filtrate and wash in the same procedure, and then preserve this precipitate for gravimetry of insoluble silicon and the solution for the ICP-OES determination of soluble silicon, respectively.

##### **7.2.4.3 Gravimetry for insoluble silicon**

Transfer the precipitate embedded in the filter paper together into a platinum crucible and ignite at 1 100 °C after charring and ashing the paper. Weigh the crucible. Moisten the precipitate in the crucible with a few drops of water and sulfuric acid (1+1) and add 10 ml of hydrofluoric acid. Then evaporate to dryness on a hot plate to remove all of silicon dioxide, ignite it at 1 100 °C and weigh the crucible again. The loss of mass after hydrofluoric acid treatment shall be the amount of insoluble silicon dioxide.

##### **7.2.4.4 ICP-OES for soluble silicon**

Aspirate an aliquot of the preserved solution into an Ar plasma of ICP-OES to determine soluble silicon in the sample.

#### **7.2.5 Blank test**

Run blank determinations according to the operations of [7.2.4.1](#) to [7.2.4.4](#) without taking a sample.

#### **7.2.6 Drawing calibration curve**

For ICP-OES, prepare calibration solutions to span the range of concentration of silicon in the test solution. Each calibration solution shall have a similar matrix to the test solution.

With those calibration solutions, draw calibration curves for soluble silicon to establish the relation between the emission intensity and the amount of silicon.

### 7.2.7 Calculation

With the amount of insoluble silicon in 7.2.4.3, soluble silicon in 7.2.4.4 and the blank test in 7.2.5, calculate the content of total silicon according to Formula (1).

$$T.Si = [(m_1 - m_0) + (A_1 - A_0)] / m \times 0,4674 \times 100 \quad (1)$$

where

$T.Si$  is the content of total silicon in the sample, % (mass fraction);

$m_1$  is the amount of insoluble silicon dioxide in the sample, g;

$m_0$  is the amount of insoluble silicon dioxide in the blank test, g;

$A_1$  is the amount of soluble silicon dioxide in the sample, g;

$A_0$  is the amount of insoluble silicon dioxide in the blank test, g;

$m$  is the weighed amount of the sample, g.

### 7.3 XRF using fused cast-bead method

The procedure shall be in accordance with EN 12698-2.

## 8 Determination of total nitrogen

### 8.1 Classification of determination methods

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

- Method A: Acid pressure decomposition–distillation separation–acidimetric titration method
- Method B: Inert gas fusion–thermal conductivity method
- Method C: Fusion–ammonia separation–acidimetric titration method

### 8.2 Acid pressure decomposition–distillation separation–acidimetric titration method

#### 8.2.1 Principle

A sample is decomposed in a pressure decomposition vessel with a mixture of hydrofluoric acid and sulfuric acid to convert nitrogen into ammonia. Add boric acid and transfer the solution into a distillation flask. Add sodium hydroxide and perform steam distillation. React the distilled ammonia with a known amount of amidosulfuric acid and back-titrate the excess of amidosulfuric acid with a standardized sodium hydroxide solution.

#### 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 Hydrofluoric acid.

### 8.2.2.3 Sulfuric acid.

8.2.2.4 Sodium hydroxide, more than 97,0 % (mass fraction) of purity.

8.2.2.5 Sodium hydroxide solution (500 g/l), prepared by dissolving sodium hydroxide in water.

8.2.2.6 Amidosulfuric acid, more than 99,0 % (mass fraction) of purity.

8.2.2.7 0,1 mol/l amidosulfuric acid solution, prepared by weighing 10,0 g of amidosulfuric acid and dissolving in water to make 1 000 ml. Calculate the factor of this solution according to Formula (2).

$$F = m \times P / (9,7095 \times 100) \quad (2)$$

where

$F$  is the factor of the 0,1 mol/l amidosulfuric acid solution;

$m$  is the weighed amount of amidosulfuric acid, g;

$P$  is the purity of amidosulfuric acid, % (mass fraction).

8.2.2.8 0,1 mol/l sodium hydroxide solution, prepared by dissolving sodium hydroxide in water in accordance with ISO 21068-3, 5.2.2.8. Take exactly 50 ml of the 0,1 mol/l amidosulfuric acid solution in a beaker (200 ml) and dilute with water to about 100 ml. Titrate this solution with the 0,1 mol/l sodium hydroxide solution using a pH meter. Take the end point as pH 5,5 and determine the volume of the titrant consumed. Calculate the factor of this solution according to Formula (3).

$$F_1 = F \times 50,00 / V \quad (3)$$

ISO 17947:2014

where <https://standards.iteh.ai/catalog/standards/iso/de225f6a-6640-47dd-890e-6ef04fba7f01/iso-17947-2014>

$F_1$  is the factor of the 0,1 mol/l sodium hydroxide solution;

$F$  is the factor of the 0,1 mol/l amidosulfuric acid solution;

$V$  is the titration volume of the 0,1 mol/l sodium hydroxide solution, ml.

### 8.2.2.9 Boric acid.

8.2.2.10 Ammonium sulfate, more than 99,9 % (mass fraction) of purity.

## 8.2.3 Apparatus

Use ordinary laboratory apparatus for chemical analysis and the following.

### 8.2.3.1 Platinum crucible.

### 8.2.3.2 Pressure decomposition vessel, on the market.

An example is shown in [Figure 1](#). Use the vessels for the exclusive use in this analysis only to avoid cross-contamination by nitrogen. If the vessel which has ever contacted with nitric acid is used, the lower values of nitrogen can be obtained.