

---

---

**Chemical analysis of refractories  
containing alumina, zirconia, and silica —  
Refractories containing 5 % to 45 %  
of ZrO<sub>2</sub> (alternative to the X-ray  
fluorescence method) —**

Part 3:

**Flame atomic absorption  
spectrophotometry (FAAS)  
and inductively coupled plasma emission  
spectrometry (ICP-AES)**

<https://standards.iteh.ai/catalog/standards/sist/bb12c12b-4ec6-4ec6-8a06-208fab4925f3/iso-21079-3-2008>

*Analyse chimique des matériaux réfractaires contenant de l'alumine, de la zirconie et de la silice — Matériaux réfractaires contenant de 5 % à 45 % de ZrO<sub>2</sub> (méthode alternative à la méthode par fluorescence de rayons X) —*

*Partie 3: Méthodes par spectrométrie d'absorption atomique dans la flamme (FAAS) et spectrométrie d'émission atomique avec plasma induit par haute fréquence (ICP-AES)*



**PDF disclaimer**

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

**iTeh STANDARD PREVIEW**  
**(standards.iteh.ai)**

ISO 21079-3:2008

<https://standards.iteh.ai/catalog/standards/sist/bb2e12b-4ee6-4ec8-8a06-208fab4925f3/iso-21079-3-2008>



**COPYRIGHT PROTECTED DOCUMENT**

© ISO 2008

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing 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

Foreword.....	iv
<b>1 Scope .....</b>	<b>1</b>
<b>2 Normative references .....</b>	<b>1</b>
<b>3 Instrumental methods using ICP-AES .....</b>	<b>1</b>
<b>3.1 Determination of residual silica in solution by ICP-AES .....</b>	<b>1</b>
<b>3.2 Determination of silicon(IV) oxide, aluminium oxide, iron(III) oxide, titanium(IV) oxide, calcium oxide, magnesium oxide, chromium(III) oxide and hafnium oxide using stock solutions (S1), (S'1) or (S''1) by ICP-AES .....</b>	<b>3</b>
<b>3.3 Determination of calcium oxide, magnesium oxide, sodium oxide and potassium oxide using stock solutions (S4) by ICP-AES .....</b>	<b>7</b>
<b>4 Instrumental methods using Flame Atomic Absorption Spectrometry (FAAS) .....</b>	<b>8</b>
<b>4.1 Determination of calcium oxide, magnesium oxide, sodium oxide and potassium oxide using stock solutions (S3) by FAAS .....</b>	<b>8</b>
<b>4.2 Determination of chromium(III) oxide by FAAS spectrometry .....</b>	<b>10</b>
<b>5 Calculation and expression of test results .....</b>	<b>12</b>
<b>6 Test report .....</b>	<b>12</b>
<b>Bibliography .....</b>	<b>13</b>

<https://standards.itech.ai/catalog/standards/sist/bb2e12b-4ec6-4ec8-8a06-208fab4925f3/iso-21079-3-2008>  
 ISO 21079-3:2008

## 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 21079-3 was prepared by Technical Committee ISO/TC 33, *Refractories*.

ISO 21079 consists of the following parts, under the general title *Chemical analysis of refractories containing alumina, zirconia, and silica — Refractories containing 5 % to 45 % of ZrO<sub>2</sub> (alternative to the X-ray fluorescence method)*:

- *Part 1: Apparatus, reagents and dissolution* [ISO 21079-3:2008](https://standards.iteh.ai/catalog/standards/sist/bb2e12b-4ee6-4ec8-8a06-208fab4925f3/iso-21079-3-2008)
- *Part 2: Wet chemical analysis* <https://standards.iteh.ai/catalog/standards/sist/bb2e12b-4ee6-4ec8-8a06-208fab4925f3/iso-21079-3-2008>
- *Part 3: Flame atomic absorption spectrophotometry (FAAS) and inductively coupled plasma emission spectrometry (ICP-AES)*

# Chemical analysis of refractories containing alumina, zirconia, and silica — Refractories containing 5 % to 45 % of ZrO<sub>2</sub> (alternative to the X-ray fluorescence method) —

## Part 3: Flame atomic absorption spectrophotometry (FAAS) and inductively coupled plasma emission spectrometry (ICP-AES)

### 1 Scope

This part of ISO 21079 specifies flame atomic absorption spectrophotometry (FAAS) and inductively coupled plasma emission spectrometry (ICP-AES) methods for the analysis of AZS (alumina, zirconia, and silica) refractory products (containing 5 % to 45 % of ZrO<sub>2</sub>) and raw materials.

This part of ISO 21079 is not applicable to MgO-based refractories.

This part of ISO 21079 gives alternatives to the X-ray fluorescence (XRF) method given in ISO 12677.

[ISO 21079-3:2008](#)

### 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 21079-1:2008, *Chemical analysis of refractories containing alumina, zirconia and silica — Refractories containing 5 % to 45 % of ZrO<sub>2</sub> (alternative to the X-ray fluorescence method) — Part 1: Apparatus, reagents and dissolution*

ISO 26845, *Chemical analysis of refractories — General requirements for wet chemical analysis, atomic absorption spectrometry (AAS) and inductively coupled plasma (ICP) methods*

### 3 Instrumental methods using ICP-AES

#### 3.1 Determination of residual silica in solution by ICP-AES

##### 3.1.1 Principle

The residual silica remaining in solution in solutions (S1) or (S'1) is determined using ICP-AES.

##### 3.1.2 Reagents

###### 3.1.2.1 General

Prepare the reagents given in 3.1.2.2 to 3.1.2.4. and those specified in ISO 26845, as necessary.

**3.1.2.2 Diluted silicon(IV) oxide standard solution, SiO<sub>2</sub> 0,08 mg/ml.**

Transfer 20 ml of the silicon(IV) oxide standard solution (SiO<sub>2</sub> 1 mg/ml) into a 250 ml volumetric flask, and dilute to the mark with water.

**3.1.2.3 Matrix solution 1 or 1'.**

Carry out the procedure given in 9.2.2.3 or 9.2.3.3 of ISO 21079-1:2008 without the sample, but omit heating the fusion mixture or anhydrous sodium carbonate. Designate the solution equivalent to stock solution (S1) or (S'1) as matrix solution 1 or 1' as applicable.

**3.1.2.4 Series 2 solution for calibration.**

Transfer appropriate aliquot portions of diluted silicon(IV) oxide standard solution (0,08 mg/ml) precisely into several 100 ml volumetric flasks, in accordance with the composition of the samples. Add 10 ml each of matrix solution 1 or 1' and dilute to the mark with water.

Table 1 shows a typical example of the preparation of solutions. An appropriate series of solutions for calibration should be prepared, depending on the compositions of the samples and the types and capabilities of instrument used.

**Table 1 — Example of series 2 solution for calibration**

Solution No.	Matrix solution 1 or 1' ml	Diluted silicon(IV) oxide standard solution ml	Concentration of solution SiO <sub>2</sub> mg/100 ml
1	10	0	0
2	10	5	0,4
3	10	10	0,8
4	10	15	1,2
5	10	20	1,6
6	10	25	2,0

**3.1.3 Procedure**

Determine the silicon(IV) oxide remaining in solution (S1) or (S'1) (see Annex A of ISO 21079-1:2008) as follows.

Transfer 10 ml of stock solution (S1) or (S'1) to a 100 ml volumetric flask, and dilute to the mark with water. Designate this solution for the determination of dissolved silicon(IV) oxide as diluted stock solution (S1d) or (S'1d).

Spray a portion of diluted stock solution (S1d or S'1d) into the argon plasma flame of the inductively coupled plasma atomic emission (ICP-AE) spectrometer, and measure the emission intensity at an appropriate wavelength, for example, 251,611 nm.

**3.1.4 Blank test**

Carry out the procedure in 3.1.3 with blank solution (B1) or (B'1) (see Annex A of ISO 21079-1:2008). Designate the equivalent diluted blank solution to diluted stock solution (S1d) or (S'1d) as diluted blank solution (B1d or B'1d).

### 3.1.5 Plotting the calibration graph

Using the series 2 solution for calibration, carry out the emission procedure described in 3.1.3. Plot the relation between the emission intensity and mass of oxide, prepare the calibration graph by adjusting the curve so that it passes through the point of origin.

A new calibration should be carried out using the range of calibration and blank solutions for each set of determinations.

### 3.1.6 Calculation

Calculate the mass fraction of silicon(IV) oxide,  $w(\text{SiO}_2)$ , expressed as a percentage, using Equation (1) with the amount of silicon(IV) oxide derived from the values obtained from 3.1.3 and 3.1.4 and the calibration prepared as described in 3.1.5.

$$w(\text{SiO}_2) = \frac{(m_1 - m_2) + (m_s - m_b) \times \frac{500}{10}}{m} \times 100 \quad (1)$$

where

$m$  is the mass of the test portion obtained in accordance with 9.2.2.2 or 9.2.3.2 of ISO 21079-1:2008, in grams;

$m_1$  is the mass obtained in accordance with 9.2.2.3 or 9.2.3.3 of ISO 21079-1:2008, in grams;

$m_2$  is the mass obtained in accordance with 9.2.2.4 or 9.2.3.4 of ISO 21079-1:2008, in grams;

$m_s$  is the mass of silicon(IV) oxide in diluted stock solution (S1d or S'1d) as described in 3.1.3, in grams;

$m_b$  is the mass of silicon(IV) oxide in diluted blank solution (B1d or B'1d) as described in 3.1.4, in grams.

## 3.2 Determination of silicon(IV) oxide, aluminium oxide, iron(III) oxide, titanium(IV) oxide, calcium oxide, magnesium oxide, chromium(III) oxide and hafnium oxide using stock solutions (S1), (S'1) or (S''1) by ICP-AES

### 3.2.1 Principle

The emission intensity of silicon(IV) oxide, aluminium oxide, iron(III) oxide, titanium(IV) oxide, calcium oxide, magnesium oxide, chromium(III) oxide and hafnium oxide are measured by the ICP-AE spectrometer for stock solutions (S1), (S'1) or (S''1). The ICP-AES method is applicable to the components in (S1), (S'1) or (S''1) as given in Table 2.

Table 2 — Application range

Component	Range % by mass
SiO <sub>2</sub>	0,1 to 10
Al <sub>2</sub> O <sub>3</sub>	1 to 10
Fe <sub>2</sub> O <sub>3</sub>	0,01 to 2
TiO <sub>2</sub>	0,01 to 5
CaO	0,01 to 2
MgO	0,01 to 2
Cr <sub>2</sub> O <sub>3</sub>	0,01 to 3
HfO <sub>2</sub>	0,01 to 2

NOTE 1 When solutions (S1) or (S'1) are used, the SiO<sub>2</sub> is residual silica. When solution (S''1) is used, the SiO<sub>2</sub> is all of the silicon(IV) oxide.

NOTE 2 This method cannot be applied to silicon(IV) oxide and aluminium oxide contents of more than 10 % by mass.

### 3.2.2 Reagents

#### 3.2.2.1 General

Prepare the reagents given in 3.2.2.2 to 3.2.2.7 and those specified in ISO 26845, as necessary.

#### 3.2.2.2 Scandium standard solution, Sc 1,0 mg/ml.

Dry about 0,2 g of scandium oxide at 110 °C ± 5 °C for 60 min, cool in a desiccator. Weigh 0,153 4 g of this, transfer to a 100 ml beaker, gradually add 10 ml of hydrochloric acid (1+1) to dissolve, and dilute precisely to 1 l in a volumetric flask with water.

A suitable commercial standard solution may be used.

#### 3.2.2.3 Yttrium standard solution, Y 1,0 mg/ml.

Dry about 0,2 g of yttrium oxide at 110 °C ± 5 °C for 60 min, cool in a desiccator. Weigh 0,127 0 g of this, transfer to a 100 ml beaker. Dissolve by gradually adding 10 ml of hydrochloric acid (1+1) to dissolve, and dilute precisely to 1 l in a volumetric flask with water.

A suitable commercial standard solution may be used.

#### 3.2.2.4 Internal standard solution.

Transfer 10 ml of yttrium or scandium standard solution into a volumetric 100 ml flask, and dilute to the mark with water. Prepare this solution immediately prior to use.

#### 3.2.2.5 Mixed standard solution 2, SiO<sub>2</sub> 0,1 mg/ml, Al<sub>2</sub>O<sub>3</sub> 0,10 mg/ml, Fe<sub>2</sub>O<sub>3</sub> 0,02 mg/ml, CaO 0,02 mg/ml, MgO 0,2 mg/ml, HfO<sub>2</sub> 0,02 mg/ml, Cr<sub>2</sub>O<sub>3</sub> 0,03 mg/ml and TiO<sub>2</sub> 0,05 mg/ml.

Transfer 50 ml each of the silicon(IV) oxide and the aluminium oxide, 10 ml each of the iron(III) oxide, the calcium oxide, the magnesium oxide and the hafnium oxide, 15 ml of the chromium(III) oxide, and 25 ml of the titanium(IV) oxide standard solutions into a 500 ml volumetric flask, and dilute to the mark with water.



### 3.2.2.6 Matrix solution 1, 1' or 1''.

Carry out the procedure given in 9.2.2.3, 9.2.3.3 or 9.2.4.3 of ISO 21079-1:2008 without the sample, but omit heating the fusion mixture or anhydrous sodium carbonate. Designate the solution equivalent to stock solution (S1), (S'1) or (S''1) as matrix solution 1, 1' or 1'' as applicable.

### 3.2.2.7 Series 3 solutions for calibration.

Transfer appropriate aliquot portions of mixed standard solution 2 into each of several 100 ml volumetric flasks. Add 20 ml of matrix solution 1, matrix solution 1' or matrix solution 1'' and 5 ml of internal standard solution, respectively, and dilute to the mark with water.

An example of the preparation of solutions is shown in Table 3. Prepare an appropriate solution series for calibration, in accordance with the composition of the sample, and the type and capabilities of the instrument used.

Table 3 — Example of series 3 solution for calibration

Solution No.	Matrix solution ml	Internal standard solution ml	Mixed standard solution 2 ml	Concentration of solution mg/100 ml							
				SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	CaO	MgO	Cr <sub>2</sub> O <sub>3</sub>	HfO <sub>2</sub>
1	20	5	0	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00
2	20	5	2	0,20	0,20	0,04	0,10	0,04	0,04	0,06	0,04
3	20	5	5	0,50	0,50	0,10	0,25	0,10	0,10	0,15	0,10
4	20	5	10	1,00	1,00	0,20	0,50	0,20	0,20	0,30	0,20
5	20	5	15	1,50	1,50	0,30	0,75	0,30	0,30	0,45	0,30
6	20	5	20	2,00	2,00	0,40	1,00	0,40	0,40	0,60	0,40

### 3.2.3 Procedure

Transfer a 20 ml aliquot portion of stock solution (S1), (S'1) or (S''1) (see Annex A of ISO 21079-1:2008), into a 100 ml volumetric flask. Add 5 ml of internal standard solution, and dilute to the mark with water. Designate this solution as stock solution (S1dScY), (S'1dScY) or (S''1dScY).

Spray a portion of stock solution (S1dScY), (S'1dScY) or (S''1dScY) into the argon plasma flame of an ICP-AE spectrometer, and measure the emission intensity of each element and internal standard element at the appropriate wavelength.

Examples of appropriate wavelengths are given in Table 4.