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**Chemical analysis of aluminosilicate  
refractory products (alternative to the  
X-ray fluorescence method) —**

**Part 3:  
Inductively coupled plasma and atomic  
absorption spectrometry methods**

iTeh STANDARD PREVIEW

*Analyse chimique des produits réfractaires d'aluminosilicates (méthode  
alternative à la méthode par fluorescence de rayons X) —*

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

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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 21587-3 was prepared by Technical Committee ISO/TC 33, *Refractories*.

ISO 21587 consists of the following parts under the general title *Chemical analysis of aluminosilicate refractory products (alternative to the X-ray fluorescence method)*:

- *Part 1: Apparatus, reagents, dissolution and gravimetric silica*
- *Part 2: Wet chemical analysis*
- *Part 3: Inductively coupled plasma and atomic absorption spectrometry methods*

# Chemical analysis of aluminosilicate refractory products (alternative to the X-ray fluorescence method) —

## Part 3: Inductively coupled plasma and atomic absorption spectrometry methods

### 1 Scope

This part of ISO 21587 specifies inductively coupled plasma/atomic emission (ICP/AE) spectrometry and flame atomic absorption (FAA) spectrometry methods for the chemical analysis of aluminosilicate refractory products and raw materials.

The methods are applicable to the determination of the following:

- silicon(IV) oxide ( $\text{SiO}_2$ )
- aluminium oxide ( $\text{Al}_2\text{O}_3$ )
- iron(III) oxide (total iron oxide calculated as  $\text{Fe}_2\text{O}_3$ )
- titanium(IV) oxide ( $\text{TiO}_2$ )
- manganese(II) oxide ( $\text{MnO}$ )
- calcium oxide ( $\text{CaO}$ )
- magnesium oxide ( $\text{MgO}$ )
- sodium oxide ( $\text{Na}_2\text{O}$ )
- potassium oxide ( $\text{K}_2\text{O}$ )
- chromium(III) oxide ( $\text{Cr}_2\text{O}_3$ )
- zirconium oxide ( $\text{ZrO}_2$ )
- phosphorous(V) oxide ( $\text{P}_2\text{O}_5$ )

This part of ISO 21587 gives alternatives to the X-ray fluorescence (XRF) method given in ISO 12677:2003, *Chemical analysis of refractory products by XRF — Fused cast bead method*.

## 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 21587-1:2007, *Chemical analysis of aluminosilicate refractory products (alternative to the X-ray fluorescence method) — Part 1: Apparatus, reagents, dissolution and gravimetric silica*

ISO 21587-2:2007, *Chemical analysis of aluminosilicate refractory products (alternative to the X-ray fluorescence method) — Part 2: Wet chemical analysis*

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

## 3 Determination of residual silica in solution by ICP/AES

### 3.1 Principle

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

### 3.2 Reagents

Prepare the reagents specified in ISO 26845 and ISO 21587-1 and the following.

#### 3.2.1 Aluminium oxide solution, $\text{Al}_2\text{O}_3$ 5 mg/ml.

Weigh 2,65 g of aluminium (purity, more than 99,5% by mass, having Si, less than 0,001 % by mass) into a platinum dish (e.g. 100 ml). Cover the dish with a watch glass, add approximately 100 ml of hydrochloric acid (1+1) and heat on a steam bath in order to dissolve the metal. After cooling, dilute to 1 l in a volumetric flask with water.

#### 3.2.2 Matrix solution 1.

Transfer an appropriate aliquot portion of the aluminium oxide solution (5 mg/ml) into a 500 ml volumetric flask and dilute to the mark with water. Prepare this solution freshly whenever needed.

NOTE The amount of the aluminium oxide solution to be used is determined by the chemical composition of the sample. For example, use 35 ml for the aluminium oxide solution of a sample containing 35 % by mass of aluminium oxide.

#### 3.2.3 Matrix solution 2 or 2'.

Carry out the procedure given in 4.2.2.3 or 4.2.3.3 of ISO 21587-1:2007 without the sample, but omit heating the fusion mixture or anhydrous sodium carbonate. The solution equivalent to stock solution (S1) or (S'1) is referred to as matrix solution 2 or 2', as applicable.

#### 3.2.4 Series 2 solution for calibration.

Transfer appropriate amounts of aliquot portions of dilute standard silicon(IV) oxide solution (0,2 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 and either matrix solution 2 or 2', respectively, and dilute to the mark with water. Table 1 is shown as an example.

NOTE In Table 2, a typical example of the preparation of solutions is shown. Prepare an appropriate series of solutions for calibration in accordance with the compositions of the samples, and the type and capabilities of instrument used.

Table 1 — Example of Series 2 solution for calibration

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

### 3.3 Procedure

Determine the silicon(IV) oxide remaining in solution (S1) or (S'1) (4.2.2.3 or 4.2.3.3 in ISO 21587-1:2007) 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. This solution, for the determination of dissolved silicon(IV) oxide, is referred to as diluted stock solution (S1d) or (S'1'd).

Spray a portion of diluted stock solution (S1d) or (S'1'd) into the argon plasma flame of the inductively coupled plasma/atomic emission (ICP/AE) spectrometer, and measure the emission intensity at, for example, the wavelength of 251,61 nm.

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### 3.4 Blank test

Carry out the procedure in 3.3 with blank solution (B1) or (B'1). The diluted blank solution equivalent to diluted stock solution (S1d) or (S'1'd) is referred to as diluted blank solution (B1d) or (B'1'd).

### 3.5 Plotting the calibration graph

Using the calibration solution Series 2, carry out the emission procedure described in 3.3. Plot the relation between the emission intensity and the mass of oxide, and 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 solution solutions for each set of determinations.

### 3.6 Calculation

Calculate the mass fraction of silicon(IV) oxide,  $w_{\text{SiO}_2}$ , as a percentage, from the amount of silicon(IV) oxide derived from the figures obtained from 3.3 and 3.4 and the calibration described in 3.5 using the equation:

$$w_{\text{SiO}_2} = \frac{(m_1 - m_2) + (m_3 - m_4) \times \frac{500}{10}}{m} \times 100 \quad (1)$$

where

- $m$  is the mass, in g, of the test portion (4.2.2.2 or 4.2.3.2 in ISO 21587-1:2007);
- $m_1, m_2$  are the mass, in g, differences in 4.2.2.3 or 4.2.3.3 in ISO 21587-1:2007;
- $m_3$  is the mass, in g, of silicon(IV) oxide in diluted stock solution (S1d or S'1d) as described in 3.3.
- $m_4$  is the mass, in g, of silicon(IV) oxide in diluted blank solution (B1d or B'1d) as described in 3.4.

## 4 Determination of iron(III) oxide by ICP/AES

### 4.1 Principle

The emission intensity of iron is measured by an ICP/AE spectrometer on solutions S1 or S'1, obtained from 4.2.2.3 or 4.2.3.3 of ISO 21587-1:2007.

### 4.2 Reagents

Any of the reagents described in 4.1, 4.2 and 4.3 of ISO 21587-1:2007 and the following are required.

#### 4.2.1 Aluminium oxide solution, Al<sub>2</sub>O<sub>3</sub> 2 mg/ml.

Dilute aluminium oxide solution (10 mg/ml) with water to a concentration of one-fifth.

#### 4.2.2 Mixed standard solution 2, Fe<sub>2</sub>O<sub>3</sub> 0,04 mg/ml, TiO<sub>2</sub> 0,04 mg/ml, MnO 0,01 mg/ml, Cr<sub>2</sub>O<sub>3</sub> 0,01 mg/ml, ZrO<sub>2</sub> 0,01 mg/ml.

Transfer 40 ml each of the standard iron(III) oxide solution (1 mg/ml) and the standard titanium(IV) oxide solution (1 mg/ml), and 10 ml each of the standard manganese(II) oxide solution (1 mg/ml), the standard chromium(III) oxide solution (1 mg/ml) and the standard zirconium oxide solution (1 mg/ml), to a 1 000 ml volumetric flask and dilute to the mark with water.

#### 4.2.3 Matrix solution 2 or 2'.

See 3.2.3.

#### 4.2.4 Series 3 solutions for calibration.

Transfer appropriate aliquot portions of mixed standard solution 2 into each of several 100 ml volumetric flasks. Add 10 ml of matrix solution 2 or matrix solution 2', 5 ml of internal standard solution (Sc 0,1 mg/ml, Y 0,1 mg/ml), and a specified amount of aluminium oxide solution (2 mg/ml), respectively, and dilute to the mark with water.

NOTE An example of the preparation of solutions is shown in Table 2. Prepare an appropriate series of solutions for calibration in accordance with the composition of the samples, and the type and capabilities of instrument used.



**Table 2 — Example of Series 3 solution for calibration  
(mass fraction of aluminium oxide is 30 %)**

Solution for calibration No.	Matrix solution 2 or 2' ml	Internal standard solution ml	Aluminium oxide solution ml	Mixed standard solution 2 ml	Concentration of solution				
					mg/100 ml				
					Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	MnO	Cr <sub>2</sub> O <sub>3</sub>	ZrO <sub>2</sub>
1	10	5	3	0	0,00	0,00	0,00	0,00	0,00
2	10	5	3	1	0,04	0,04	0,01	0,01	0,01
3	10	5	3	2	0,08	0,08	0,02	0,02	0,02
4	10	5	3	3	0,12	0,12	0,03	0,03	0,03
5	10	5	3	4	0,16	0,16	0,04	0,04	0,04
6	10	5	3	5	0,20	0,20	0,05	0,05	0,05
7	10	5	3	10	0,40	0,40	0,10	0,10	0,10
8	10	5	3	15	0,60	0,60	0,20	0,20	0,20
9	10	5	3	20	0,80	0,80	0,30	0,30	0,30

#### 4.3 Procedure

Transfer a 10 ml aliquot portion of stock solution (S1) or (S'1), as prepared in 4.2.2.3 or 4.2.3.3 of ISO 21587-1:2007, into a 100 ml volumetric flask. Add 5 ml of internal standard solution (Sc 0,1 mg/ml, Y 0,1 mg/ml), and dilute to the mark with water. This solution is designated as stock solution (S1'dScY) or (S'1'dScY).

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Spray a portion of stock solution (S1'dScY) or (S'1'dScY) into the argon plasma flame of an ICP/AE spectrometer, and measure the iron emission intensity at the appropriate wavelength, for example, 259,94 nm.

#### 4.4 Blank test

Transfer a 10 ml aliquot portion of blank solution (B1) or (B'1), (4.2.2.4 or 4.3.2.4 in ISO 21587-1:2007) and carry out the procedure given in 4.3. The solution corresponding to stock solution (B1) or (B'1) is designated as blank solution (B1'dScY) or (B'1'dScY).

#### 4.5 Plotting the calibration graph

Use Series 3 solutions for calibration. Carry out the procedure described in 4.3 and plot the relation between the emission intensity and the mass of iron(III) oxide. Prepare the calibration graph by adjusting the curve so that it passes through the point of origin.

#### 4.6 Calculation

Calculate the mass fraction of iron(III) oxide,  $w_{\text{Fe}_2\text{O}_3}$ , as a percentage, using the following equation, with iron(III) oxide that is derived from the emission intensity in 4.3 and 4.4, and the calibration in 4.5.

$$w_{\text{Fe}_2\text{O}_3} = \frac{m_1 - m_2}{m} \times \frac{500}{10} \times 100 \quad (2)$$

where

$m_1$  is the mass, in g, of iron(III) oxide in stock solution (S1dScY) or (S'1dScY);

$m_2$  is the mass, in g, of iron(III) oxide in blank solution (B1dScY) or (B'1dScY);

$m$  is the mass, in g, of the test portion in 4.2.2.2 or 4.3.2.2 of ISO 21587-1:2007.

## 5 Determination of titanium(IV) oxide by ICP/AES

### 5.1 Principle

The emission intensity of the titanium is measured by an ICP/AE spectrometer on stock solution (S1dScY) or (S'1dScY).

### 5.2 Procedure

Spray a portion of stock solution (S1dScY) or (S'1dScY) (4.3) into the argon plasma flame of an ICP/AE spectrometer, and measure the titanium emission intensity at a wavelength of 334,94 nm.

### 5.3 Blank test

Carry out the procedure described in 5.2 for blank solution (B1dScY) or (B'1dScY) obtained in 4.4.

### 5.4 Plotting the calibration graph (standards.iteh.ai)

Using the Series 3 solutions (4.2.4), carry out the procedure described in 5.2, and plot the relation between the emission intensity and titanium(IV) oxide. Prepare the calibration graph by adjusting the curve so that it passes through the point of origin.

### 5.5 Calculation

Calculate the mass fraction of titanium oxide(IV),  $w_{\text{TiO}_2}$ , as a percentage, using the following equation with the amount of titanium(IV) oxide derived from the emission intensity obtained in 5.2 and 5.3, and the calibration in 5.4.

$$w_{\text{TiO}_2} = \frac{m_1 - m_2}{m} \times \frac{500}{10} \times 100 \quad (3)$$

where

$m_1$  is the mass, in g, of titanium(IV) oxide in stock solution (S1dScY) or (S'1dScY);

$m_2$  is the mass, in g, of titanium(IV) oxide in blank solution (B1dScY) or (B'1dScY);

$m$  is the mass, in g, of the test portion described in 4.2.2.2 or 4.3.2.2 of ISO 21587-1:2007.

## 6 Determination of manganese(II) oxide by ICP/AES

### 6.1 Principle

The emission intensity of manganese from stock solution (S1dScY) or (S'1dScY) is measured by an ICP/AE spectrometer.

## 6.2 Procedure

Spray a portion of stock solution (S1dScY) or (S'1dScY) obtained in 4.3 into the argon plasma flame of an ICP/AE spectrometer, and measure the emission intensity at a wavelength of 257,61 nm, for example.

## 6.3 Blank test

Carry out the procedure described in 6.2 using blank solution (B1dScY) or (B'1dScY) obtained in 4.4.

## 6.4 Plotting the calibration graph

Carry out the procedure described in 6.2 using Series 3 solutions for calibration (4.2.4). Plot the relation between the emission intensity and the mass of manganese(II) oxide, and prepare the calibration graph by adjusting the curve so that it passes through the point of origin.

## 6.5 Calculation

Calculate the mass fraction of manganese(II) oxide,  $w_{\text{MnO}}$ , as a percentage, using the following equation. Use the mass of manganese(II) oxide that is derived from the emission intensity obtained in 6.2. and 6.3. and the calibration in 6.4.

$$w_{\text{MnO}} = \frac{m_1 - m_2}{m} \times \frac{500}{10} \times 100 \quad (4)$$

where

$m_1$  is the mass, in g, of manganese(II) oxide in stock solution (S1dScY) or (S'1dScY);

$m_2$  is the mass, in g, of manganese(II) oxide in blank solution (B1dScY) or (B'1dScY).

$m$  is the mass, in g, of the test portion described in 4.2.2.2 or 4.3.2.2 of ISO 21587-1:2007.

## 7 Determination of calcium oxide by ICP/AES

### 7.1 Principle

The emission intensity of calcium in solution (S4) is measured by an ICP/AE spectrometer.

### 7.2 Series 1 solution for calibration

See 10.2.2 in ISO 21587-2:2007. Transfer aliquot portions of mixed standard solution 1 to each of several 100 ml volumetric flasks. To each, add 5 ml of hydrochloric acid (1+1) and an appropriate amount of standard solution I of aluminium oxide, and dilute to the mark with water. Typical examples of preparation are shown in Table 3.