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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

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)

Reference number
ISO 21587-3:2007(E)

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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*

INTERNATIONAL STANDARD **ISO 21587-3:2007(E)**

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 (SiO_2)
- aluminium oxide (Al_2O_3)
- iron(III) oxide (total iron oxide calculated as Fe_2O_3)
- titanium(IV) oxide (TiO_2)
- manganese(II) oxide (MnO)
- calcium oxide (CaO)
- magnesium oxide (MgO)
- sodium oxide (Na_2O)
- potassium oxide (K_2O)
- chromium(III) oxide (Cr_2O_3)
- zirconium oxide (ZrO_2)
- phosphorous(V) oxide (P_2O_5)

This part of ISO 21587 gives alternatives to the X-ray flluorescence (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, Al_2O_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 No.	**Matrix solution 1** ml	**Matrix solution 2 or 2′** ml	**Dilute standard silicon(IV) oxide solution** ml	**Concentration of solution** SiO_2 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 (S1′d).

Spray a portion of diluted stock solution (S1d) or (S1′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.

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 (S1′d) is referred to as diluted blank solution (B1d) or (B1′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_{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:

$$w_{\mathrm{SiO_2}} = \frac{(m_1 - m_2) + (m_3 - m_4) \times \frac{500}{10}}{m} \times 100 \qquad (1)$$

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_2O_3 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_2O_3 0,04 mg/ml, TiO_2 0,04 mg/ml, MnO 0,01 mg/ml, Cr_2O_3 0,01 mg/ml, ZrO_2 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_2O_3	TiO_2	MnO	Cr_2O_3	ZrO_2
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 (S1′dSCY).

Spray a portion of stock solution (S1dScY) or (S1′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 (B1dScY) or (B′1dScY).

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, $w_{Fe_2O_3}$, 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.

$$w_{Fe_2O_3} \frac{m_1 - m_2}{m} \times \frac{500}{10} \times 100 \qquad (2)$$

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

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), $w_{\mathrm{TiO_2}}$, 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.

$$w_{\mathrm{TiO_2}} = \frac{m_1 - m_2}{m} \times \frac{500}{10} \times 100 \tag{3}$$

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_{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.

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

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.

Table 3 — Example of Series 1 solution for calibration (mass fraction of aluminium oxide is 30 %)

Solution for calibration No.	Aluminium oxide solution I ml	Hydrochloric acid (1+1) ml	Mixed standard solution 1 ml	Concentration of solution mg/100 ml			
				CaO	MgO	Na_2O	K_2O
1	3	5	0	0	0	0	0
2	3	5	2	0,2	0,2	0,2	0,2
3	3	5	4	0,4	0,4	0,4	0,4
4	3	5	6	0,6	0,6	0,6	0,6
5	3	5	8	0,8	0,8	0,8	0,8
6	3	5	10	1.0	1.0	1.0	1.0
7	3	5	20	2,0	2,0	2,0	2,0
8	3	5	30	3,0	3,0	3,0	3,0
9	3	5	40	4,0	4,0	4,0	4,0
10	3	5	50	5,0	5,0	5,0	5,0
11	3	5	60	6,0	6,0	6,0	6,0

7.3 Procedure

Spray a portion of stock solution (S4) (4.3.4.3 in ISO 21587-1:2007) into the argon plasma flame of an ICP/AE spectrometer, and measure the emission intensity at a wavelength of 393,37 nm, for example.

7.4 Blank test

Carry out the procedure described in 7.3 using blank solution (B4).

7.5 Plotting the calibration graph

Carry out the procedure described in 7.3 using Series 1 solutions for calibration. Plot the relation between the emission intensity and the mass of calcium oxide, and prepare the calibration graph by adjusting the curve so that it passes through the point of origin.

7.6 Calculation

Calculate the mass fraction of calcium oxide, w_{CaO}, as a percentage, using the following equation. Use the mass of calcium oxide derived from the emission intensity obtained in 7.3 and 7.5, and the calibration described in 7.6.

$$w_{\text{CaO}} = \frac{m_1 - m_2}{m} \times 100 \tag{5}$$

where

m_1 is the mass, in g, of calcium oxide in stock solution (S4);

m_2 is the mass, in g, of calcium oxide in blank solution (B4);

m is the mass, in g, of the test portion (4.3.4.3 in ISO 21587-1:2007).

8 Determination of magnesium oxide by ICP/AES

8.1 Principle

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

8.2 Procedure

Spray a portion of stock solution (S4) obtained in 4.3.4.3 in ISO 21587-1:2007 into the argon plasma flame of an ICP/AE spectrometer, and measure the intensity at a wavelength of 279,55 nm.

8.3 Blank test

Carry out the procedure described in 8.2 with blank solution (B4) obtained in 4.3.4.4 in ISO 21587-1:2007.

8.4 Plotting the calibration graph

Use Series 1 solutions for calibration described in 10.2.2 in ISO 21587-2:2007. Carry out the procedure described in 8.2 and plot the relation between the emission intensity and the mass of magnesium oxide, and prepare the calibration graph by adjusting the curve so that it passes through the point of origin.

8.5 Calculation

Calculate the mass fraction of magnesium oxide, w_{MgO}, as a percentage, using the following equation by using the mass of magnesium oxide derived from the emission intensity obtained in 8.2 and 8.3, and the calibration described in 8.4.

$$w_{\mathrm{MgO}} = \frac{m_1 - m_2}{m} \times 100 \tag{6}$$

where

m_1 is the mass, in g, of magnesium oxide in stock solution (S4);

m_2 is the mass, in g, of magnesium oxide in blank solution (B4);

m is the mass, in g, of the test portion in 4.3.4.2 in ISO 21587-1:2007.

9 Determination of sodium oxide by ICP/AES

9.1 Principle

The emission intensity of sodium is measured by an ICP/AE spectrometer on stock solution (S4).

9.2 Procedure

Spray a portion of stock solution (S4) obtained in 4.3.4.3 in ISO 21587-1:2007 into the argon plasma flame of an ICP/AE spectrometer, and measure the intensity at a wavelength of 589,00 nm.

9.3 Blank test

Carry out the procedure described in 9.2 with blank solution (B4) obtained in 4.3.4.4 in ISO 21587-1:2007.

9.4 Plotting the calibration graph

Carry out the procedure described in 9.2 using Series 1 solutions for calibration described in 10.2.2 in ISO 21587-2:2007. Plot the relation between the emission intensity and the mass of sodium oxide. Prepare the calibration graph by adjusting the curve so that it passes through the point of origin.

9.5 Calculation

Calculate the mass fraction of sodium oxide, w_{Na_2O}, as a percentage, using the following equation by using the mass of sodium oxide derived from the emission intensity obtained in 9.2 and 9.3, and the calibration described in 9.4.

$$w_{Na_2O} = \frac{m_1 - m_2}{m} \times 100 \tag{7}$$

where

m_1 is the mass, in g, of sodium oxide in stock solution (S4);

m_2 is the mass, in g, of sodium oxide in blank solution (B4);

m is the mass, in g, of the test portion in 4.3.4.2 in ISO 21587-1:2007.

10 Determination of potassium oxide by ICP/AES

10.1 Principle

The emission intensity of potassium is measured by an ICP/AE spectrometer on stock solution (S4).

10.2 Procedure

Spray a portion of stock solution (S4) obtained in 4.3.4.3 in ISO 21587-1:2007 into the argon plasma flame of an ICP/AE spectrometer, and measure the intensity at a wavelength of 766,49 nm.

10.3 Blank test

Carry out the procedure described in 10.2 using blank solution (B4) obtained in 4.3.4.4 in ISO 21587-1:2007.

10.4 Plotting the calibration graph

Carry out the procedure described in 10.2 using Series 1 solutions for calibration described in 10.2.2 in ISO 21587-2:2007. Plot the relation between the emission intensity and the mass of potassium oxide. Prepare the calibration graph by adjusting the curve so that it passes through the point of origin.

10.5 Calculation

Calculate the mass fraction of potassium oxide, w_{K_2O}, as a percentage, using the following equation by using the mass of potassium oxide derived from the emission intensity obtained in 10.2 and 10.3, and the calibration described in 10.4.

$$w_{K_2O} = \frac{m_1 - m_2}{m} \times 100 \tag{8}$$

where

m_1 is the mass, in g, of potassium oxide in stock solution (S4);

m_2 is the mass, in g, of potassium oxide in blank solution (B4);

m is the mass, in g, of the test portion in 4.3.4.2 in ISO 21587-1:2007.

11 Determination of chromium(III) oxide by ICP/AES

11.1 Principle

The emission intensity of chromium in stock solution (S1dScY) or (S′1dScY) is measured by an ICP/AE spectrometer.

11.2 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 205,55 nm, for example.

11.3 Blank test

Carry out the procedure described in 11.2 using blank solution (B1dScY) or (B′1dScY) obtained in 4.4.

11.4 Plotting the calibration graph

Carry out the procedure described in 11.2, using Series 3 solutions for calibration as described in 4.2.4. Plot the relation between the emission intensity and the mass of chromium(III) oxide, and prepare the calibration graph by adjusting the curve so that it passes through the point of origin.

11.5 Calculation

Calculate the mass fraction of chromium(III) oxide, $w_{\mathrm{Cr_2O_3}}$, as a percentage, using the following equation, using the mass of chromium(III) oxide derived from the emission intensity obtained in 11.2 and 11.3, and the calibration described in 11.4.

$$w_{\mathrm{Cr_2O_3}} = \frac{m_1 - m_2}{m} \times \frac{500}{10} \times 100 \qquad (9)$$

where

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

m_2 is the mass, in g, of chromium(III) oxide in blank solution (B1d) or (B′1d);

m is the mass, in g, of the test portion described in 4.2.2.3 or 4.2.3.3 in ISO 21587-1:2007.

12 Determination of zirconium oxide by ICP/AES

12.1 Principle

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

12.2 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 257,14 nm, for example.

12.3 Blank test

Carry out the procedure described in 12.2 using blank solution (B1d or B′1d) obtained in 4.4.

12.4 Plotting the calibration graph

Carry out the procedure described in 12.2, using Series 3 solutions for calibration as described in 4.2.4. Plot the relation between the emission intensity and the mass of zirconium oxide, and prepare the calibration graph by adjusting the curve so that it passes through the point of origin.

12.5 Calculation

Calculate the mass fraction of zirconium oxide, $w_{\mathrm{ZrO_2}}$, as a percentage,, using the following equation, using the mass of zirconium oxide derived from the emission intensity obtained in 12.2 and 12.3, and the calibration described in 12.4.

$$w_{\mathrm{ZrO_2}} = \frac{m_1 - m_2}{m} \times \frac{500}{10} \times 100 \qquad (10)$$

where

m_1 is the mass, in g, of zirconium oxide in the aliquot portion of stock solution (S1dScY) or (S′1dScY);

m_2 is the mass, in g, of zirconium oxide in blank solution (B1dScY or B′1dScY);

m is the mass, in g, of the test portion described in 4.2.2.3 or 4.2.3.3 in ISO 21587-1:2007

13 Determination of phosphorus(V) oxide by ICP/AES

13.1 Principle

The emission intensity of phosphorus is measured by an ICP emission spectrometer on stock solution (S1dScY) or (S′1dScY).

13.2 Reagents

Prepare the following reagents in addition to any in ISO 26845 and ISO 21587-1 that are required.

13.2.1 Aluminium oxide, Al_2O_3 1 mg/ml.

Wash the surface of the aluminium (minimum 99,9 % by mass) with hydrochloric acid (1+3) and dissolve the oxidized layer, and subsequently wash with water, ethanol, and diethyl ether in succession. Then dry in a desiccator. Weigh 0,529 2 g of aluminium into a platinum dish (e.g. 100 ml), and cover with a watch glass, then add 50 ml of hydrochloric acid (1+1), and heat to dissolve the metal on a steam bath. After cooling, dilute to 1 l in a volumetric flask with water.

13.2.2 Magnesium oxide, MgO 10 mg/ml.

Weigh 6,0 g of magnesium (minimum 99,9 % by mass) with mass fraction pf silicon less than 0,001 % into a platinum dish (e.g. 150 ml) and cover with a watch glass. Then, add 100 ml of hydrochloric acid (1+1) and heat on a steam bath until the metal has dissolved. After cooling, dilute to 1 l in a volumetric flask with water.

13.2.3 **Calcium oxide**, CaO 10 mg/ml.

Transfer 18 g of calcium carbonate (99,9 % by mass min. with mass fraction of silicon less than 0,001 %) into a platinum crucible (e.g. 150 ml). Cover with a watch glass, gradually add 60 ml of hydrochloric acid (1+1) to dissolve the carbonate. Cool, transfer to a 1 l volumetric flask, and dilute to the mark with water.

13.2.4 Matrix solution 2 or 2′.

See 3.2.3.

13.2.5 Series 4 solution for calibration.

Transfer appropriate volumes of dilute standard phosphorus(V) oxide solution (0,04 mg/ml) to each of several 100 ml volumetric flasks. Add appropriate volumes of the matrix solution 2 or 2′ and dilute to the mark with water. Typical examples of preparation are shown in Table 4.

In Table 4, an example of the preparation of solutions is shown. Depending on the composition of the sample, and the types and capabilities of instrument used, an appropriate solution series for calibration should be prepared.

NOTE In Series 3 solution for calibration, standard phosphorus(V) oxide solution may be added, instead of the standard zirconium oxide solution.

Table 4 — Example of the Series 4 solution for calibration

Solution for calibration No.	**Matrix solution 2 or 2′** ml	**Aluminium oxide solution**[a] (1mg/ml) ml	**Internal standard solution** ml	**Dilute standard phosphorus(V) oxide solution** ml	**Concentration of P_2O_5 solution** mg/100 ml
1	10	1,0	5	0	0,00
2	10	1,0	5	1	0,04
3	10	1,0	5	2	0,08
4	10	1,0	3	3	0,12
5	10	1,0	5	4	0,16
6	10	1,0	5	5	0,20
7	10	1,0	5	10	0,40
8	10	1,0	5	15	0,60
9	10	1,0	5	20	0,80
10	10	1,0	5	30	1,20

[a] 1 ml of aluminium oxide solution corresponds to 5 % by mass. In this case, the mass fraction of aluminium oxide is 5 %.

13.3 Procedure

Spray a portion of the solution (S1ScY) or (S′1ScY) into the argon plasma flame of an ICP emission spectrometer, and measure the emission intensity at a wavelength of 213,62 nm, for example. If necessary, measure the emission intensity of an internal standard element.

13.4 Blank test

Carry out the procedure described in 13.3 using blank test solution (B1dScY) or (B′1dScY).

13.5 Plotting the calibration graph

Carry out the procedure described in 13.3 using Series 4 solutions and plot the relation between the emission intensity and the mass of phosphorus(V) oxide as the calibration graph.

13.6 Calculation

Calculate the mass fraction of phosphorus (V) oxide, $w_{P_2O_5}$, as a percentage, using the following equation and the mass of phosphorus(V) oxide derived from the emission intensity obtained in 13.3 and 13.4, and the calibration described in 13.5.

$$w_{P_2O_5} = \frac{m_1 - m_2}{m} \times \frac{500}{V} \times 100 \qquad (11)$$

where

m_1 is the mass, in grams, of phosphorus(V) oxide in the aliquot portion of stock solution (S1dScY) or (S′1dScY);

m_2 is the mass, in grams, of phosphorus(V) oxide in the aliquot portion of blank solution (B1dScY or B′1dScY);

V is the aliquot portion volume, in ml, of stock solution (S1) or (S′1) described in 13.3;

m is the mass, in grams, of the test portion in 4.2.2.2 or 4.2.3.2 in ISO 21587-1:2007.

14 Determination of calcium by AAS

14.1 Principle

The determination is carried out on the sample decomposed by removing the silicon(IV) oxide with hydrofluoric acid. A portion of the solution is sprayed into the flame of an atomic absorption spectrometer, and the emission intensity of calcium is measured.

14.2 Reagents

Prepare the following reagents in addition to any in ISO 26845 and ISO 21587-1 that are required.

14.2.1 **Aluminium oxide solution**, Al_2O_3, 10 mg/ml.

Wash the surface of a sufficient amount of aluminium metal (purity of more than 99,9 % by mass) with hydrochloric acid (1+4) to dissolve the oxidized layer. Then wash with water, ethanol and diethyl ether in succession, and dry in a desiccator. Weigh 5,3 g of aluminium and transfer to a 250 ml beaker. Cover with a watch glass, add 50 ml of hydrochloric acid (1+1), and heat to dissolve. After cooling, dilute to 1 l in a volumetric flask with water.

14.2.2 **Series 5 solution for calibration**.

Transfer aliquot portions of mixed standard solution II to several 100 ml volumetric flasks. To each, add 5 ml of hydrochloric acid (1+1), 10 ml of lanthanum solution, and an appropriate amount of standard solution 1 of aluminium oxide, and dilute to the mark with water. A typical example of solutions for calibration is shown in Table 5.

Table 5 — Example of Series 5 solution for calibration (the mass fraction of aluminium oxide is 30 %)

Solution for calibration No.	Aluminium oxide solution ml	Hydrochloric acid (1+1) ml	Lanthanum solution ml	Mixed standard solution 1 ml	Concentration of solution (mg/100 ml)			
					CaO	MgO	Na_2O	K_2O
1	6	5	10	0	0	0	0	0
2	6	5	10	2	0,2	0,2	0,2	0,2
3	6	5	10	4	0,4	0,4	0,4	0,4
4	6	5	10	6	0,6	0,6	0,6	0,6
5	6	5	10	8	0,8	0,8	0,8	0,8
6	6	5	10	10	1,0	1,0	1,0	1,0
7	6	5	10	20	2,0	2,0	2,0	2,0
8	6	5	10	30	3,0	3,0	3,0	3,0
9	6	5	10	40	4,0	4,0	4,0	4,0
10	6	5	10	50	5,0	5,0	5,0	5,0
11	6	5	10	60	6,0	6,0	6,0	6,0

14.3 Procedure

Spray a portion of stock solution (S3) (4.3.3.4 in ISO 21587-1:2007) into the dinitrogen oxide-acetylene flame of an atomic absorption (AA) spectrometer, measure at a wavelength of 422 nm.

NOTE When the concentration of stock solution (S3) exceeds the upper limit of calibration of the standards, transfer an appropriate volume (x ml) of stock solution (S3) into a 100 ml volumetric flask.

Add $5{,}0 - 5{,}0' \left(\frac{x}{100} \right)$ ml of hydrochloric acid (1+1) and $10{,}0 - 10{,}0' \left(\frac{x}{100} \right)$ ml of lanthanum solution. Dilute to the mark with water, and measure with this solution.

14.4 Blank test

Carry out the procedure described in 14.3 using blank solution (B3) (4.3.3.5 in ISO 21587-1:2007).

NOTE When the concentration of stock solution (S3) exceeds the upper limit of calibration of the standards, the blank solution (B3) is prepared using the same procedure as that for the stock solution in the note in 14.3.

14.5 Plotting the calibration graph

Carry out the procedure described in 14.3 using Series 5 calibration solutions (14.2) for calibration. Plot the relation between the absorbance and mass of calcium oxide, and prepare the calibration graph by adjusting the curve so that it passes through the point of origin.

NOTE When the concentration of stock solution (S3) exceeds the upper limit of calibration of the standards, adjust the amount of aluminium oxide solution in Series 5 calibration solutions to the same concentration of aluminium oxide as in the stock solution after dilution.

14.6 Calculation

Calculate the mass fraction of calcium oxide, w_{CaO}, as a percentage, using the following equation. Use the mass of calcium oxide derived from the absorbance obtained in 14.3 and 14.4, and the calibration described in 14.5.

$$w_{\mathrm{CaO}} = \frac{m_1 - m_2}{m} \times \frac{100}{V} \times 100 \tag{12}$$

where

m_1 is the mass, in g, of calcium oxide in the aliquot portion of stock solution (S3) or the diluted stock solution;

m_2 is the mass, in g, of calcium oxide in the aliquot portion of blank solution (B3) or the diluted blank solution;

V is the volume, in ml, of the aliquot portion taken from stock solution (S3) (In the case where the whole solution is used, that is, no aliquot portion, use 100 ml);

m is the mass, in g, of the test portion (4.3.3.3 in ISO 21587-1:2007).

15 Determination of magnesium oxide by AAS

15.1 Principle

The absorbance of magnesium in stock solution (S3) is measured by an AA spectrometer.

15.2 Procedure

Spray a portion of stock solution (S3) obtained in 4.3.3.4 in ISO 21587-1:2007 into a flame, and measure the absorbance of magnesium at a wavelength of 285,2 nm.

15.3 Blank test

Carry out the procedure described in 15.2 using blank solution (B3) obtained in 4.3.3.5 in ISO 21587-1:2007.

15.4 Plotting the calibration graph

Use Series 5 solution (14.2.2) for the procedure described in 15.2. Then plot the relation between the absorbance and mass of magnesium oxide, and prepare the calibration by parallel displacement of the line to pass through the original point.

15.5 Calculation

Calculate the mass fraction of magnesium oxide, w_{MgO}, as a percentage, using the following equation with the mass of magnesium oxide derived from the absorbance obtained in 15.2 and 15.3, and the calibration described in 15.4.

$$w_{\mathrm{MgO}} = \frac{m_1 - m_2}{m} \times \frac{500}{V} \times 100 \tag{13}$$

where

m_1 is the mass, in g, of magnesium oxide in stock solution (S3) or the diluted stock solution;

m_2 is the mass, in g, of magnesium oxide in blank solution (B3) or the diluted blank solution;

V is the aliquot portion volume, in ml, of stock solution (S3);

NOTE In the case where the whole solution is used, i.e. there is no aliquot portion, use V = 100 ml.

m is the mass, in g, of the test portion in 4.3.3.3 in ISO 21587-1:2007.

16 Determination of sodium oxide by AAS

16.1 Principle

The absorbance of sodium is measured in stock solution (S3) by an AA spectrometer.

16.2 Procedure

Spray a portion of stock solution (S3) in 4.3.3.4 in ISO 21587-1:2007 into the air-acetylene flame of an AA spectrometer, and measure the absorbance at a wavelength of 589,0 nm.

NOTE If the concentration of sodium oxide is too high in the stock solution, a wavelength of 589,6 nm or 330,2 nm may be used.

16.3 Blank test

Carry out the procedure in 16.2 using blank solution (B3) obtained in 4.3.3.5 in ISO 21587-1:2007.

16.4 Plotting the calibration graph

Carry out the procedure described in 16.2 using Series 5 solution for calibration as described in 14.2.2, and plot the relation between the absorbance and mass of sodium oxide. Prepare the calibration graph by adjusting the curve so that it passes through the point of origin.

NOTE In the case where the note in 16.2 applies, adjust Series 5 solutions for calibration to the corresponding concentration of sodium oxide and use the same wavelength as for the stock solution.

16.5 Calculation

Calculate the mass fraction of sodium oxide, $w_{\mathrm{Na_2O}}$, as a percentage, by using the following equation with the mass of sodium oxide derived from the absorbance obtained in 16.2 and 16.3, and the calibration described in 16.4.

$$w_{\mathrm{Na_2O}} = \frac{m_1 - m_2}{m} \times \frac{100}{V} \times 100 \qquad (14)$$

where

m_1 is the mass, in g, of sodium oxide in stock solution (S3) or the diluted stock solution;

m_2 is the mass, in g, of sodium oxide in blank solution (B3) or the diluted blank solution;

V is the aliquot portion volume, in ml, of stock solution (S3);

NOTE In the case where the whole solution is used, i.e. there is no aliquot portion, use V = 100 ml.

m is the mass, in g, of the test portion in 4.3.3.3 in ISO 21587-1:2007.

17 Determination of potassium oxide by AAS

17.1 Principle

The absorbance of potassium is measured in stock solution (S3) by an AA spectrometer.

17.2 Procedure

Spray a portion of stock solution (S3) obtained in 4.3.3.4 in ISO 21587-1:2007. into the air-acetylene flame of an AA spectrometer, and measure the absorbance at a wavelength of 766 nm.

NOTE If the concentration of potassium oxide is too high in the stock solution, a wavelength of 769 nm or 404,4 nm may be used.

17.3 Blank test

Carry out the procedure described in 17.2 using blank solution (B3) obtained in 4.3.3.5 in ISO 21587-1:2007.

17.4 Plotting the calibration graph

Carry out the procedure described in 17.2 using Series 5 solution for calibration as described in 14.2.2. Plot the relation between the absorbance and mass of potassium oxide, and prepare the calibration graph by adjusting the curve so that it passes through the point of origin.

NOTE In the case of too high a concentration of potassium oxide in the stock solution, a Series 5 solution is prepared corresponding to the concentration, and the calibration is prepared by using the same wavelength as the stock solution.

17.5 Calculation

Calculate the mass fraction of potassium oxide, $w_{\mathrm{K_2O}}$, as a percentage. Use the following equation with the mass of potassium oxide derived from the absorbance obtained in 17.2 and 17.3, and the calibration described in 17.4.

$$w_{\mathrm{K_2O}} = \frac{m_1 - m_2}{m} \times \frac{100}{V} \times 100 \tag{15}$$

where

m_1 is the mass, in g, of potassium oxide in stock solution (S3) or the diluted stock solution;

m_2 is the mass, in g, of potassium oxide in blank solution (B3) or the diluted blank solution;

V is the aliquot portion volume, in ml, of stock solution (S3)

NOTE In the case where the whole solution is used, i.e. there is no aliquot portion, use V = 100 ml.

m is the mass, in g, of the test portion in 4.3.3.3 in ISO 21587-1:2007.

18 Determination of manganese oxide by AAS

18.1 Principle

The absorbance of manganese is measured in an aliquot portion of stock solution (S1) or (S′1) by an atomic absorption spectrometer.

18.2 Reagents

Prepare the following reagents in addition to any in ISO 26845 and ISO 21587-1 that are required.

18.2.1 Aluminium oxide solution, Al_2O_3, 4 mg/ml.

Wash the surface of a sufficient amount of aluminium metal (purity of more than 99,9 % by mass) with hydrochloric acid (1+4) to dissolve the oxidized layer. Then wash with water, ethanol and diethyl ether in succession, and dry in a desiccator. Weigh 2,1 g of aluminium and transfer it to a 250 ml beaker. Cover with a watch glass, add 25 ml of hydrochloric acid (1+1), and heat to dissolve. After cooling, dilute to 1 l in a volumetric flask with water.

18.2.2 Mixed standard solution 3, MnO and Cr_2O_3, each 0,02 mg/ml.

Transfer 20 ml each of the standard manganese(**II**) oxide solution (1 mg/ml) and standard chromium(**III**) oxide solution (1 mg/ml) into a 1 000 ml volumetric flask and dilute to the mark with water.

18.2.3 Series 6 solutions for calibration.

These solutions are prepared following a procedure similar to that used for Series 2 solution for calibration (3.2.4). Add 20 ml of matrix solution 2 or 2″ and 10 ml of lanthanum solution. An example is shown in Table 6.

Table 6 — Example of Series 6 solution for calibration (mass fraction of aluminium oxide is 30 %)

Solution for calibration No.	Matrix solution 2 or 2′ ml	Aluminium oxide solution ml	Lanthanum solution ml	Mixed standard solution 3 ml	Concentration of solution Mg/100 ml	
					MnO	Cr_2O_3
1	20	3	10	0	0,00	0,00
2	20	3	10	1	0,02	0,02
3	20	3	10	2	0,04	0,04
4	20	3	10	3	0,06	0,06
5	20	3	10	4	0,08	0,08
6	20	3	10	5	0,10	0,10
7	20	3	10	10	0,20	0,20
8	20	3	10	15	0,30	0,30
9	20	3	10	20	0,40	0,40

NOTE Matrix solution 2 is used for stock solution (S1) and matrix solution 2′ is used for stock solution (S′1).

18.3 Procedure

Transfer a 20 ml aliquot portion of stock solution (S1) or (S′1), prepared as in 4.2.2.3 or 4.2.3.3 in ISO 21587-1:2007 to a 100 ml volumetric flask, add 10 ml of lanthanum solution, and dilute to the mark with water. This solution is designated as stock solution (S1dLa) or stock solution (S′1dLa).

Spray a portion of stock solution (S1dLa) or (S′1dLa) into the flame of an AA spectrometer, measure at a wavelength of 297,5 nm.

18.4 Blank test

Carry out the procedure described in 18.3 with blank solution (B1) or (B′1) obtained in 4.2.2.4 or 4.2.3.4 in ISO 21587-1:2007. The solution corresponding to stock solutions (S1dLa) and (S′1dLa) is designated as blank solution (B1dLa) or (B′1dLa).

18.5 Plotting the calibration graph

Treat an aliquot portion of Series 6 solution for calibration (18.2.2) as described in 18.3, and plot the relation between the absorbance and mass of manganese(II) oxide. Prepare the calibration graph by adjusting the curve so that it passes through the point of origin.

18.6 Calculation

Calculate the mass fraction of manganese(II) oxide, w_{MnO}, as a percentage, using the following equation with the mass of manganese(II) oxide which is derived from the absorbance described in 18.3 and 18.4, and the calibration described in 18.5.

$$w_{\mathrm{MnO}} = \frac{m_1 - m_2}{m} \times \frac{500}{20} \times 100 \qquad (16)$$

where

m_1 is the mass, in g, of manganese(II) oxide in the aliquot portion of stock solution (S1dLa) or (S′1dLa);

m_2 is the mass, in g, of manganese(II) oxide in the aliquot portion of blank solution (B1dLa) or (B′1dLa);

m is the mass, in g, of the test portion described in 4.2.2.2 or 4.2.3.2 in ISO 21587-1:2007.

19 Determination of chromium(III) oxide by AAS

19.1 Principle

The absorbance for chromium in stock solution (S1dLa) or S′1dLa) (18.3) is measured by an AA spectrometer.

19.2 Procedure

Spray a portion of stock solution (S1dLa) or (S′dLa) obtained in 18.3 into the air-acetylene flame of an AA spectrometer, and measure the absorbance for chromium at a wavelength of 357,9 nm.

19.3 Blank test

Transfer blank solution (B1dLa) or (B′1dLa) obtained in 18.4 and carry out the procedure described in 19.2.

19.4 Plotting the calibration graph

Carry out the procedure described in 19.2 using Series 6 solution (18.2.3) for calibration as described in 18.5. Plot the relation between the absorbance and mass of chromium(III) oxide. Prepare the calibration graph by adjusting the curve so that it passes through the point of origin.

19.5 Calculation

Calculate the mass fraction of potassium oxide, $w_{\mathrm{Cr_2O_3}}$, as a percentage. Use the following equation with the mass of chromium(III) oxide derived from the absorbance obtained in 19.2 and 19.3, and the calibration described in 19.4.

$$w_{\mathrm{Cr_2O_3}} = \frac{m_1 - m_2}{m} \times \frac{500}{20} \times 100 \qquad [17]$$

where

m_1 is the mass, in g, of chromium(III) oxide in the aliquot portion of stock solution (S1dLa) or (S′1dLa);

m_2 is the mass, in g, of chromium(III) oxide in the aliquot portion of blank solution (B1dLa) or (B′1dLa);

m is the mass, in g, of the test portion described in 4.2.2.2 or 4.2.3.2 in ISO 21587-1:2007.

20 Test report

Prepare a test report in accordance with ISO 26845.

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