INTERNATIONAL
STANDARD

ISO
20565-3

First edition
2008-12-01

Chemical analysis of chrome-bearing refractory products and chrome-bearing raw materials (alternative to the X-ray fluorescence method) —

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

Analyse chimique des produits réfractaires contenant du chrome et des matières premières contenant du chrome (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)

Reference number
ISO 20565-3:2008(E)

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Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
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Foreword

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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 20565-3 was prepared by Technical Committee ISO/TC 33, *Refractories*, in collaboration with Technical Committee CEN/TC 187, *Refractory products and materials*.

ISO 20565 consists of the following parts, under the general title *Chemical analysis of chrome-bearing refractory products and chrome-bearing raw materials (alternative to the X-ray fluorescence method)*:

- *Part 1: Apparatus, reagents, dissolution and determination of gravimetric silica*
- *Part 2: Wet chemical analysis*
- *Part 3: Flame atomic absorption spectrometry (FAAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES)*

INTERNATIONAL STANDARD **ISO 20565-3:2008(E)**

Chemical analysis of chrome-bearing refractory products and chrome-bearing raw materials (alternative to the X-ray fluorescence method) —

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

1 Scope

This part of ISO 20565 specifies flame atomic absorption spectrometry (FAAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES) methods for the chemical analysis of chrome-bearing refractory products and chrome-bearing raw materials.

It is applicable in the ranges of determination given in Table 1.

ISO 20565 gives alternatives to the X-ray fluorescence (XRF) method given in ISO 12677.

Table 1 — Range of determination (% by mass)

Component	Range
SiO_2	0,5 to 10
Al_2O_3	2 to 30
Fe_2O_3	0,5 to 25
TiO_2	0,01 to 1
MnO	0,01 to 1
CaO	0,01 to 3
MgO	15 to 85
Na_2O	0,01 to 1
K_2O	0,01 to 1
Cr_2O_3	2 to 60
ZrO_2	0,01 to 0,5
P_2O_5	0,01 to 5
LOI	−0,5 to 5
NOTE These values are after the loss on ignition (LOI) has been taken into account.	

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 20565-1:2008, *Chemical analysis of chrome-bearing refractory products and chrome-bearing raw materials (alternative to the X-ray fluorescence method) — Part 1: Apparatus, reagents, dissolution and determination of gravimetric silica*

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*

3 Instrumental methods using inductively coupled plasma emission spectrometry (ICP-AES)

3.1 Determination of residual silica in stock solutions (S1) by ICP-AES

3.1.1 Principle

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

3.1.2 Reagents

Prepare the following reagents in addition to any reagents described in ISO 20565-1:2008, Clause 5, that are necessary.

3.1.2.1 Diluted silicon(IV) oxide standard solution, SiO_2 0,08 mg/ml.

Transfer 20 ml of the silicon(IV) oxide standard solution (SiO_2 1 mg/ml) into a 250 ml volumetric flask and dilute to the mark with water.

3.1.2.2 Matrix solution 2.

Carry out the procedure given in ISO 20565-1:2008, 9.2.2.3, without the sample, but omit heating the fusion mixture or anhydrous sodium carbonate. The equivalent solution to stock solution (S1) is referred to as matrix solution 2.

3.1.2.3 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 2 (3.1.2.2) and dilute to the mark with water.

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

Table 2 — Example of series 2 solution for calibration

Solution No.	Matrix solution 2 ml	Diluted silicon(IV) oxide standard solution ml	Concentration of solution SiO_2 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) (see ISO 20565-1) as follows.

Transfer 10 ml of stock solution (S1) 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).

Spray a portion of diluted stock solution (S1d) into the argon plasma flame of the ICP-AE spectrometer, and measure the emission intensity at, for example, the wavelength of 251,611 nm.

3.1.4 Blank test

Carry out the procedure in 3.1.3 with blank solution (B1) (see ISO 20565-1). The equivalent diluted blank solution to diluted stock solution (S1d) is referred to as diluted blank solution (B1d).

3.1.5 Plotting of calibration graph

Calibrate the ICP-AE spectrometer using the series 2 solution (3.1.3.3) and 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 solution solutions for each set of determinations.

3.1.6 Calculation

Calculate the mass fraction of silicon(IV) oxide, $w_{\mathrm{SiO_2}}$, as a percentage, using Equation (1). Use the mass of silicon(IV) oxide derived from the figures obtained from 3.1.3 and 3.1.4 and the calibration prepared in 3.1.5.

$$w_{\mathrm{SiO_2}} = \frac{(m_1 - m_2) + (m_{\mathrm{s}} - m_{\mathrm{b}}) \times \frac{250}{10}}{m} \times 100 \qquad (1)$$

where

m is the mass of the test portion (see ISO 20565-1), in grams (g);

m_1 is the first mass from ISO 20565-1:2008, 9.2.2.3.3, in grams (g);

m_2 is the second mass from ISO 20565-1:2008, 9.2.2.3.3,, in grams (g);

m_{s} is the mass of silicon(IV) oxide in diluted stock solution (S1d) as described in 3.1.3, in grams (g);

m_{b} is the mass of silicon(IV) oxide in diluted blank solution (B1d) as described in 3.1.4, in grams (g).

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

3.2.1 Principle

The emission intensities of silicon(IV) oxide, aluminium oxide, iron(III) oxide, titanium(IV) oxide, manganese(II) oxide, calcium oxide, magnesium oxide, chromium(III) oxide and zirconium oxide are measured by an ICP-AE spectrometer for stock solutions (S1) (see ISO 20565-1) or (S′1) (see ISO 20565-1). This method should be applied to components in (S1) or (S′1) in accordance with Table 3.

Table 3 — Application range (% by mass)

Component	Range
SiO_2	0,1 to 10
Al_2O_3	0,05 to 10
Fe_2O_3	0,01 to 10
TiO_2	0,01 to 1
MnO	0,01 to 1
CaO	0,01 to 10
Cr_2O_3	0,01 to 10
ZrO_2	0,01 to 0,5

NOTE 1 When solution (S1) is used, the SiO_2 is residual silica. When solution (S′1) is used, the SiO_2 is all of the silicon(IV) oxide.

NOTE 2 Determination of calcium oxide by this method cannot be applied to calcium oxide contents of more than 10 % by mass.

3.2.2 Reagents

Prepare the following reagents in addition to any reagents described in ISO 20565-1:2008, Clause 5, that are necessary.

3.2.2.1 Mixed standard solution 3, SiO_2 0,04 mg/ml, Al_2O_3 0,04 mg/ml, Fe_2O_3 0,04 mg/ml, TiO_2 0,005 mg/ml, MnO 0,005 mg/ml, CaO 0,04 mg/ml, Cr_2O_3 0,04 mg/ml, ZrO_2 0,005 mg/ml.

Transfer aliquot portions of standard silicon(IV) oxide (40 ml), aluminium oxide (40 ml), iron(III) oxide (40 ml), titanium(IV) oxide (5 ml), manganese(II) oxide (5 ml), calcium oxide (40 ml), chromium(III) oxide (40 ml) and zirconium oxide (5 ml) solutions to a 1 000 ml volumetric flask and dilute to the mark with water.

3.2.2.2 Matrix solution 3 or 3′.

Carry out the procedure given in ISO 20565-1:2008, 9.2.2.3 or 9.2.3.3, without the sample, but omit heating the fusion mixture or anhydrous sodium carbonate. The equivalent solution to stock solution (S1) or (S′1) is referred to as matrix solution 3 or 3′ as applicable.

3.2.2.3 Internal standard solution.

Transfer 10 ml of standard scandium oxide solution (1 mg/ml) and standard yttrium oxide solution (1 mg/ml) to a 100 ml volumetric flask and dilute to the mark with water. Prepare when necessary.

3.2.2.4 Series 3 solutions for calibration.

Transfer appropriate aliquot portions of mixed standard solution 3 to each of several 100 ml volumetric flasks. Add 20 ml of matrix solution 3 or matrix solution 3′ and 5 ml of internal standard solution, respectively, and dilute to the mark with water.

In Table 4, an example of the preparation of solutions is shown. Prepare an appropriate solution series for calibration in accordance with the composition of the sample, the type and capabilities of instrument used. When using this approach to calibration, it is essential to check for line interferences of any of these oxides on each other. If any are present, appropriate corrections should be applied.

Table 4 — Example of series 3 solution for calibration

Solution for calibration No.	Matrix solution 3 ml	Internal standard solution ml	Mixed standard solution 3 ml	Concentration of solution mg/100 ml							
				SiO_2	Al_2O_3	Fe_2O_3	TiO_2	MnO	CaO	Cr_2O_3	ZrO_2
1	10	5	0	0,00	0,00	0,00	0,000	0,000	0,00	0,00	0,000
2	10	5	2	0,08	0,08	0,08	0,010	0,010	0,08	0,08	0,010
3	10	5	5	0,20	0,20	0,20	0,025	0,025	0,20	0,20	0,025
4	10	5	10	0,40	0,40	0,40	0,050	0,050	0,40	0,40	0,050
5	10	5	20	0,80	0,80	0,80	0,100	0,100	0,80	0,80	0,100
6	10	5	30	1,20	1,20	1,20	0,150	0,150	1,20	1,20	0,150
7	10	5	40	1,60	1,60	1,60	0,200	0,200	1,60	1,60	0,200
8	10	5	50	2,00	2,00	2,00	0,250	0,250	2,00	2,00	0,250

3.2.3 Procedure

Transfer a 10 ml aliquot portion of stock solution (S1) or (S′1) as prepared in ISO 20565-1:2008, 9.2.2.3.4 or 9.2.3.3, to a 100 ml volumetric flask. Add 5 ml of internal standard solution, and dilute to the mark with water. This solution is designated as stock solution (S1dScY) 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 emission intensity of each element and internal standard elements at the appropriate wavelength given in Table 5.

Table 5 — Example of wavelength

Component	Element	Wavelength nm
SiO_2	Si	251,611
Al_2O_3	Al	396,152
Fe_2O_3	Fe	259,940
TiO_2	Ti	334,941
MnO	Mn	257,610
CaO	Ca	393,366
Cr_2O_3	Cr	267,716
ZrO_2	Zr	343,823
Internal standard element	Y	371,030
	Sc	361,383

3.2.4 Blank test

Using a 10 ml aliquot portion of blank solution (B1) (see ISO 20565-1) or (B′1) (see ISO 20565-1), carry out the procedure given in 3.2.3. The solution corresponding to stock solution (S1) or (S′1) is designated as blank solution (B1dScY) or (B′1dScY).

3.2.5 Plotting the calibration graph

Use series 3 solutions for calibration. Carry out the procedure described in 3.2.3 and plot the relationship between the emission intensity and mass of each component (SiO_2, Al_2O_3, Fe_2O_3, TiO_2, MnO, CaO, Cr_2O_3 and ZrO_2). Prepare a calibration graph for each component.

3.2.6 Calculation

Calculate the mass fraction $w_{\mathrm{M}_n\mathrm{O}_m}$ of each component $w_{\mathrm{SiO_2}}$, $w_{\mathrm{Al_2O_3}}$, $w_{\mathrm{Fe_2O_3}}$, $w_{\mathrm{TiO_2}}$, w_{MnO}, w_{CaO}, $w_{\mathrm{Cr_2O_3}}$ and $w_{\mathrm{ZrO_2}}$, expressed as a percentage, using Equation (2). Use the mass of each component derived from the emission intensity in 3.2.3 and 3.2.4 and the calibration prepared in 3.2.5.

$$w_{\mathrm{M}_n\mathrm{O}_m} = \frac{m_{\mathrm{s}} - m_{\mathrm{b}}}{m} \times \frac{100}{10} \times 100 \qquad (2)$$

where

m_{s} is the mass of each component, indicated as $\mathrm{M}_n\mathrm{O}_m$, in stock solution (S1dScY) or (S′1dScY), in grams (g);

m_{b} is the mass of each component, indicated as $\mathrm{M}_n\mathrm{O}_m$, in blank solution (B1dScY) or (B′1dScY), in grams (g);

m is the mass of the test portion (see ISO 20565-1), in grams (g).

3.3 Determination of sodium oxide and potassium oxide using stock solutions (S4) by ICP-AES

3.3.1 Principle

The emission intensity of sodium and potassium in stock solution (S4) (see ISO 29565-1) is measured by ICP-AE spectrometer.

3.3.2 Reagents

Prepare the following reagents in addition to any reagents described in ISO 20565-1:2008, Clause 5, that are necessary.

3.3.2.1 Matrix solution 4.

Transfer amounts of calcium oxide and magnesium oxide solutions, corresponding to the contents of calcium oxide and magnesium oxide in the sample, and dilute to 250 ml with water.

NOTE The added volumes of the matrix solution are approximate values ± 5 ml. For example, when the mass fractions of calcium oxide and magnesium oxide are 24 % and 71 %, respectively, the added volumes of calcium oxide and magnesium oxide solutions are 25 ml and 75 ml, respectively.

3.3.2.2 Mixed standard solution 4, Na_2O 0,05 mg/ml, K_2O 0,05 mg/ml.

Transfer 25 ml of the standard sodium oxide and potassium oxide solutions to a 500 ml volumetric flask and dilute to the mark with water.

3.3.2.3 Series 4 solution for calibration.

Transfer aliquot portions of mixed standard solution 4 to each of several 100 ml volumetric flasks. To each, add 20 ml of matrix solution 4 and 5 ml of hydrochloric acid (1+1) (see ISO 20565-1:2008, 5.1.17) and dilute to the mark with water. Typical examples of preparation are shown in Table 6.

Table 6 — Example of series 4 solution for calibration

Solution No.	Matrix solution 4	Hydrochloric acid (1+1)	Mixed standard solution 4	Concentration of solution mg/100 ml	
	ml	ml	ml	Na_2O	K_2O
1	20	5	0	0	0
2	20	5	2	0,10	0,10
3	20	5	5	0,25	0,25
4	20	5	10	0,50	0,50
5	20	5	20	1,00	1,00
6	20	5	30	1,50	1,50
7	20	5	40	2,00	2,00

3.3.3 Procedure

Spray a portion of stock solution (S4) (see ISO 20565-1) into the argon plasma flame of an ICP-AE spectrometer, and measure the emission intensity of each element at the appropriate wavelength given in Table 7.

Table 7 — Example of wavelength

Component	Element	Wavelength nm
Na_2O	Na	588,995
K_2O	K	766,491

3.3.4 Blank test

Carry out the procedure described in 3.3.3 using blank solution (B4) (see ISO 20565-1).

3.3.5 Plotting of calibration graph

Calibrate the ICP-AE spectrometer using series 4 solutions as described in 3.3.3. Plot the relationship between the emission intensity and mass of each component (Na_2O and K_2O). Prepare a calibration graph for each component.

3.3.6 Calculation

Calculate the mass fraction $w_{\mathrm{M}_n\mathrm{O}_m}$ of each component, $w_{\mathrm{Na_2O}}$ and $w_{\mathrm{K_2O}}$, expressed as a percentage, using Equation (3). Use the mass of sodium oxide and potassium oxide derived from the emission intensity obtained in 3.3.3 and 3.3.4 and the calibration prepared in 3.3.5.

$$w_{\mathrm{M}_n\mathrm{O}_m} = \frac{m_{\mathrm{s}} - m_{\mathrm{b}}}{m} \times 100 \qquad (3)$$

where

m_{s} is the mass of each component, indicated as $\mathrm{M}_n\mathrm{O}_m$, in stock solution (S4), in grams (g);

m_{b} is the mass of each component, indicated as $\mathrm{M}_n\mathrm{O}_m$, in blank solution (B4), in grams (g);

m is the mass of the test portion (see ISO 20565-1), in grams (g).

3.4 Determination of phosphorus(V) oxide by ICP-AES

3.4.1 Principle

The emission intensity of phosphorus is measured by ICP-AE spectrometer on stock solution (S1dScY) or (S′1dScY) (see 3.2.3).

3.4.2 Reagents

Prepare the following reagents in addition to any reagents described in ISO 20565-1:2008, Clause 5, that are necessary.

3.4.2.1 Matrix solution 5.

Transfer amounts of calcium oxide and magnesium oxide solutions corresponding to the contents of calcium oxide and magnesium oxide in the sample, and dilute to 250 ml with water.

NOTE The added volumes of the matrix solution are approximate values ± 5 ml. For example, when the content percentages of calcium oxide and magnesium oxide are 24 % by mass and 71 % by mass, respectively, the added volumes of calcium oxide and magnesium oxide solutions are 25 ml and 75 ml, respectively.

3.4.2.2 **Aluminium oxide**, Al_2O_3 (1 mg/ml).

Wash the surface of the aluminium (99,9 % by mass minimum) with hydrochloric acid (1+3), 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.

3.4.2.3 **Internal standard solution**.

Transfer 10 ml of standard scandium oxide solution (1 mg/ml) and standard yttrium oxide solution (1 mg/ml) to a 100 ml volumetric flask and dilute to the mark with water. Prepare when necessary.

3.4.2.4 **Series 5 solution for calibration**.

Transfer aliquot portions of diluted phosphorus(V) oxide standard solution to each of several 100 ml volumetric flasks. To each, add 20 ml of matrix solution 5, the appropriate amount of aluminium oxide solution, 5 ml of internal standard solution and 5 ml of hydrochloric acid (1+1) and dilute to the mark with water. Typical examples of preparation are shown in Table 8.

In Table 8, an example of the preparation of solutions is shown. Depending on the composition of the sample, and the type and capabilities of the instrument used, an appropriate solution series for calibration should be prepared.

In the series 5 solution for calibration, standard phosphorus(V) oxide solution may be added instead of the standard zirconium oxide solution.

Table 8 — Example of the series 5 solution for calibration

Solution No.	Matrix solution 5 ml	Aluminium oxide solution [a] (1 mg/ml) ml	Internal standard solution ml	Diluted 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	5	0,20
4	10	1,0	5	10	0,40
5	10	1,0	5	20	0,80
6	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 %.

3.4.3 Procedure

Spray a portion of solution (S1dScY) or (S1dScY), obtained in 3.2.3, into the argon plasma flame of an ICP-AE spectrometer and measure the emission intensity at, for example, a wavelength of 213,62 nm. If necessary, measure the emission intensity of an internal standard element.

3.4.4 Blank test

Carry out the procedure described in 3.4.3 using blank test solution (B1dScY) or (B’1dScY) obtained in 3.2.4.

3.4.5 Plotting of calibration graph

Carry out the procedure described in 3.4.3 using series 5 solutions and plot the relation between the emission intensity and mass of phosphorus(V) oxide as the calibration graph.

3.4.6 Calculation

Calculate the mass fraction of phosphorus(V) oxide, $w_{P_2O_5}$, as a percentage, using Equation (4). Use the mass of phosphorus(V) oxide derived from the emission intensity obtained in 3.4.3 and 3.4.4, and the calibration prepared in 3.4.5.

$$w_{P_2O_5} = \frac{m_s - m_b}{m} \times \frac{100}{V} \times 100 \quad (4)$$

where

m_s is the mass of phosphorus(V) oxide in the aliquot portion of stock solution (S1dScY) or (S′1dScY) (see 3.2.3), in grams (g);

m_b is the mass of phosphorus(V) oxide in the aliquot portion of blank solution (B1dScY) or (B′1dScY) (see 3.2.4), in grams (g);

V is the volume of the aliquot portion of stock solution (S1) or (S′1) described in 3.2.3, in millilitres (ml);

m is the mass of the test portion (see ISO 20565-1), in grams (g).

4 Instrumental methods using flame absorption spectrophotometry (FAAS)

4.1 Determination of manganese(II) oxide, calcium oxide and chromium(III) oxide by AAS

4.1.1 Principle

The absorbance of manganese(II) oxide, calcium oxide and chromium(III) oxide are measured in an aliquot portion of stock solution (S1) or (S′1) (see ISO 20565-1) by an AA spectrometer.

4.1.2 Reagents

Prepare the following reagents in addition to any reagents described in ISO 20565-1:2008, Clause 5, that are necessary.

4.1.2.1 Matrix solution 6 or 6′.

Prepare as described 3.2.2.2.

4.1.2.2 Mixed standard solution 6, MnO 0,02 mg/ml, CaO 0,10 mg/ml, Cr_2O_3 0,04 mg/ml.

Transfer aliquot portions of standard manganese(II) oxide (10 ml), calcium oxide (50 ml), and chromium(III) oxide (20 ml) solutions to a 500 ml volumetric flask and dilute to the mark with water.

4.1.2.3 Series 6 solutions for calibration.

Transfer aliquot portions of diluted chromium oxide standard solution to several 100 ml volumetric flasks. Add to each 10 ml of lanthanum solution and 20 ml of matrix solution 6 or 6′, and dilute to the mark with water. A typical example of solutions for calibration is shown in Table 9.

Table 9 — Example of series 6 solution for calibration

Solution No.	Matrix solution 6 or 6′	Lanthanum solution	Mixed standard solution 6	Concentration of solution mg/100 ml		
	ml	ml	ml	MnO	CaO	Cr_2O_3
1	20	10	0	0,00	0,00	0,00
2	20	10	1	0,02	0,10	0,04
3	20	10	2	0,04	0,20	0,08
4	20	10	3	0,06	0,30	0,12
5	20	10	4	0,08	0,40	0,16
6	20	10	5	0,10	0,50	0,20
7	20	10	10	0,20	1,00	0,40
8	20	10	15	0,30	1,50	0,60
9	20	10	20	0,40	2,00	0,80
10	20	10	40	0,80	4,00	1,60

NOTE Matrix solution 6 is used for stock solution (S1); matrix solution 6′ is used for stock solution (S′1).

4.1.3 Procedure

Transfer a 20 ml aliquot portion of stock solution (S1) or (S′1), prepared as in ISO 20565-1:2008, 9.2.2.3.4 or 9.2.3.3, 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 the absorbance of each element at the appropriate wavelength given in Table 10.

Table 10 — Example of wavelength

Component	Element	Wavelength nm
MnO	Mn	279,5
CaO	Ca	422,7
Cr_2O_3	Cr	357,9

4.1.4 Blank test

Carry out the procedure described in 4.1.3 with blank solution (B1) or (B′1) (see ISO 20565-1). The solution corresponding to stock solutions (S1dLa) or (S′1dLa) is designated as blank solution (B1dLa) or (B′1dLa).

4.1.5 Plotting of calibration graph

Carry out the procedure described in 4.1.3 using series 6 calibration solutions for calibration. Plot the relation between the absorbance and mass of each component (MnO, CaO and Cr_2O_3). Prepare a calibration graph for each component.

4.1.6 Calculation

Calculate the mass fraction $w_{\mathrm{M}_n\mathrm{O}_m}$ of each component, w_{MnO}, w_{CaO} and $w_{\mathrm{Cr_2O_3}}$, expressed as a percentage, using Equation (5). Use the mass of each component derived from the absorbance described in items 4.1.3 and 4.1.4, and the calibration prepared in 4.1.5.

$$w_{\mathrm{M}_n\mathrm{O}_m} = \frac{m_\mathrm{s} - m_\mathrm{b}}{m} \times \frac{250}{20} \times 100 \qquad (5)$$

where

m_s is the mass of each component, indicated as $\mathrm{M}_n\mathrm{O}_m$, in stock solution (S1dLa) or (S′1dLa) (see 4.1.4), in grams (g);

m_b is the mass of each component, indicated as $\mathrm{M}_n\mathrm{O}_m$, in blank solution (B1dLa) or (B′1dLa) (see 4.1.4), in grams (g);

m is the mass of the test portion (see ISO 20565-1), in grams (g).

4.2 Determination of calcium oxide, sodium oxide and potassium oxide using stock solutions (S3) by FAAS

4.2.1 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 AA spectrometer, and the absorbance of calcium, sodium and potassium is measured.

4.2.2 Reagents

Prepare the following reagents in addition to any reagents described in ISO 20565-1:2008, Clause 5, that are necessary.

4.2.2.1 Mixed standard solution 7, CaO 0,10 mg/ml, $\mathrm{Na_2O}$ 0,10 mg/ml, and $\mathrm{K_2O}$ 0,10 mg/ml.

Transfer 50 ml each of the calcium oxide, the sodium oxide and the potassium oxide into a 500 ml volumetric flask and dilute to the mark with water.

4.2.2.2 Series 7 solution for calibration.

Transfer aliquot portions of mixed standard solution 7 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 I of aluminium oxide, and dilute to the mark with water. A typical example of solutions for calibration is shown in Table 11.

Table 11 — Example of series 7 solution for calibration

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

4.2.3 Procedure

Spray a portion of stock solution (S3) (see ISO 20565-1) into the dinitrogen oxide-acetylene flame of an AA spectrometer, and measure the absorption of each element at the appropriate wavelength given in Table 12.

Table 12 — Example of wavelength

Component	Element	Wavelength nm
CaO	Ca	422,7
MgO	Mg	285,2
Na_2O	Na	589,6
K_2O	K	766,5

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

4.2.4 Blank test

Carry out the procedure described in 4.2.3 using blank solution (B3) (see ISO 20565-1).

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.

4.2.5 Plotting of calibration graph

Carry out the procedure described in 4.2.3 using series 7 calibration solutions for calibration. Plot the relation between the absorbance and mass of each component (CaO, Na_2O and K_2O). Prepare a calibration graph for each component.

4.2.6 Calculation

Calculate the mass fraction $w_{\mathrm{M}_n\mathrm{O}_m}$ of each component, w_{CaO}, $w_{\mathrm{Na_2O}}$ and $w_{\mathrm{K_2O}}$, expressed as a percentage, using Equation (6). Use the mass of each component derived from the absorbance obtained in 4.2.3 and 4.2.4, and the calibration prepared in 4.2.5.

$$w_{\mathrm{M}_n\mathrm{O}_m} = \frac{m_\mathrm{s} - m_\mathrm{b}}{m} \times \frac{100}{V} \times 100 \qquad (6)$$

where

m_s is the mass of each component, indicated as $\mathrm{M}_n\mathrm{O}_m$, in the aliquot portion of stock solution (S3) or the diluted stock solution, in grams (g);

m_b is the mass of each component, indicated as $\mathrm{M}_n\mathrm{O}_m$, in the aliquot portion of blank solution (B3) or the diluted blank solution, in grams (g);

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

NOTE If the whole solution is used, i.e. no aliquot portion, use $V = 100$.

m is the mass of the test portion (see ISO 20565-1), in grams (g).

5 Test report

Prepare a test report as described in ISO 26845.

Bibliography

[1] ISO 12677, *Chemical analysis of refractory products by XRF — Fused cast bead method*

ICS 81.080

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